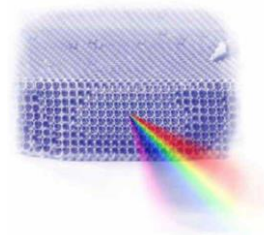


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(Конференција)

Book of Abstracts

## 12th Photonics Workshop

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## Structural analysis of perineuronal nets with high resolution microscopies

Ana Jakovljevic<sup>1</sup>, Milena Tucic<sup>1</sup>, Vera Stamenkovic<sup>1</sup>, Aleksander Kovacevic<sup>2</sup>, Tanja Pajic<sup>2</sup>, Pavle Andjus<sup>1</sup>

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**Abstract:** Perineuronal nets (PNNs) are compartments of highly organized extracellular matrix (ECM) around certain types of neurons in the central nervous system. PNNs are synthesized „on demand”, in an activity-dependent manner and participate in the stabilization of neuronal connections and in the restriction of neuronal plasticity. Their degradation can reactivate brain plasticity. Involvement of ECM glycoprotein tenascin-C (TNC) in regulation of structural organization of PNNs and neuronal plasticity during adulthood, has led us to investigate further its role in context of rearing experimental animals in enriched environment (EE). EE is an experimental paradigm that induces neuronal plasticity in a noninvasive way by cognitive, social, motor and sensory stimulation.

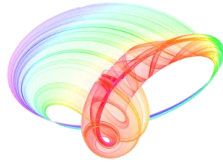
The aim of the study was to investigate how TNC deficiency affects the number and structural organization of PNNs and their reorganization upon EE. Immunohistochemistry was employed to fluorescently label PNNs with *Whisteria floribunda* agglutinin. We qualitative analysed 2D confocal images and afterwards wanted to get more precise and reliable results, so we generated and analyzed 3D confocal images. Quantification of WFA intensity was gained from z-stacks of optical slices of individual PNNs. Our results indicated subtle changes in the integrity of PNNs in the hippocampus depending on the expression of TNC and on the exposure to EE. Additional preliminary and qualitative studies were done with two-photon and second harmonic generation microscopy in search of additional high resolution information.

The results of this pilot study demonstrate that the complex and condensed structure of PNNs can only be partially resolved in its 3D organization by means of conventional microscopy. Our next goal is to investigate the topology of PNNs with super-resolution structured illumination microscopy (SR-SIM). We plan to use a novel approach developed by Dzyubenko and colleagues [1] that combines 3D SR-SIM and mathematical reconstruction in order to resolve PNNs ultra-structure.

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# Book of abstracts



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## Inducing LIPSS on multilayer thin metal films by femtosecond laser beam of different orientations

A. G. Kovačević<sup>1</sup>, S. M. Petrović<sup>2</sup>, B. Salatić<sup>1</sup>, M. Lekić<sup>1</sup>, B. Vasić<sup>1</sup>, R. Gajić<sup>1</sup>,  
D. Pantelić<sup>1</sup> and B. M. Jelenković<sup>1</sup>

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The occurrence of laser-induced periodic surface structures (LIPSS) has been known for a while [1]. Multilayer thin films, like Al/Ti, are suitable for LIPSS formation and attractive for applications – due to their wearing behavior and corrosion resistance; LIPSS generation may improve their properties as well [2, 3]. LIPSS properties depend not only on the material but also on the beam characteristics, like wavelength, polarization and scanning directions, etc. [4].

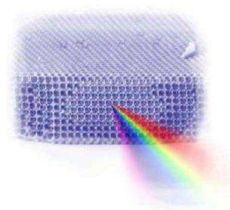
After exposing with NIR femtosecond pulses from Coherent Mira 900 laser system in several beam exposures, we have analyzed the samples of thin metal film systems with Tescan Mira3 SEM and NTegra AFM. The formation of LIPSS is most probably due to the generation of surface plasmon polariton, through the periodic distribution of energy in the interaction zone which lead to thermal processes in layers and interfaces. Two types of LIPSS were generated, which differ in shape, orientation and in ablation pronounced or not. For consecutive interactions in the same direction, LIPSS maintained its orientation, while for orthogonal passes LIPSS with mutually orthogonal orientation were generated. LIPSS period fluctuated between 320 and 380 nm and structures with pronounced ablation have significantly smaller width. Probable mechanism is that for greater accumulated energy pronounced ablation takes place giving LIPSS in the form of “trenches”, while for less accumulated energy the buildup of the material – probably due to pronounced oxidation – lead to LIPSS in the form of “hills”.

ACKNOWLEDGEMENT: The work was supported by the Ministry of Science of the Republic of Serbia under No. III45016, OI171038 and OI171005.

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## Laser-induced parallel structures on multilayer thin films of Ni, Pd, Ti, Ta and W

Aleksander G. Kovačević<sup>1</sup>, Suzana Petrović<sup>2</sup>, Jelena Potočnik<sup>2</sup>, Marina Lekić<sup>1</sup>, Branislav Salatić<sup>1</sup>, Vladimir Lazović<sup>1</sup>, Dejan Pantelić<sup>1</sup>, Branislav Jelenković<sup>1</sup>

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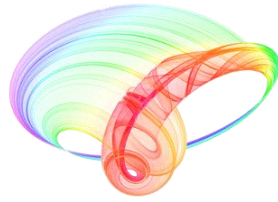
**Abstract.** The interaction of ultrashort laser beam with metal surfaces may induce the generation of periodic structures (LIPSS) with period less than the incoming wavelength, opening wide area of application [1, 2]. The presence of the underneath layer influences the quality of the LIPSS [3]. We have exposed multilayer thin films Ni/Ti, Ni/Pd, W/Ti, Ti/Ta to femtosecond beams of various wavelengths and powers. The interactions have been performed by Mira900 fs laser of Coherent. Detailed surface morphology after irradiation was examined firstly by optical microscopy, and then by scanning electron microscopy (JEOL JSM-7500F, Tokyo, Japan). Two types of structures have been noticed. Their appearance differ in the direction against the polarization direction, in pronounced ablation and in the spatial period, enabling their grouping into LIPSS of higher and lower spatial frequencies. Surface plasmon polariton is seen as the most probable cause of periodic distribution of energy at the surface and consequently to LIPSS.

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### **3. Optical materials**

## Narrowing of laser beam propagating through biological suspension

A. Kovacevic<sup>1</sup>, T. Pajic<sup>2</sup>, D. Pavlovic<sup>1</sup>, M. Stanic<sup>3</sup>, M. Lekic<sup>1</sup>, S. Nikolic<sup>1</sup>, B. Jelenkovic<sup>1</sup>

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Recent demonstration of nonlinear self-action of laser beams in suspension of biological materials, like marine bacteria and red blood cells, has been reported [1-3]. In this work, we demonstrate nonlinear optical effects of laser beam propagation through the freshwater green microalga *Chlorella sorokiniana*, cultivated in Bold basal medium with 3-fold nitrogen and vitamins (3N-BBM+V).

*Chlorella sorokiniana* is a species of single-celled freshwater green microalga in the division *Chlorophyta*. Its spherical or ellipsoidal cells (3 x 2 μm in small cells to 4.5 x 3.5 μm in large cells, sometimes >5 μm) divide rapidly to produce four new cells every 17 to 24 hours [4]. The non-pathogenic species has been chosen as a model organism due to its small cell dimension, rapid growth, non-mobility and non-toxicity. The algae were kept in the light chamber and the temperature was maintained at 22°C. Mid-exponential growth phase of algal culture was used for the experiments.

In the experiments, the 532 nm CW laser beam is directed to the glass cuvette that is filled either with the medium or with algae suspended in the medium. We have monitored the laser beam diameter at the entrance and exit of the cuvette, and its axial profile through entire cell length. The concentration has been determined by optical microscopy and optical density and has been varied between 10<sup>6</sup> and 10<sup>8</sup> cm<sup>-3</sup>.

The concentration of the algae and the laser beam power affect the beam radius. Our preliminary results have shown the effect of light self-trapping, i.e., the decrease of laser diameter when the algae concentration exceeds 10<sup>6</sup> cm<sup>-3</sup> while laser power is above 1 W. The difference of the refractive indexes of the algae and the medium can induce optical trapping of algae, which subsequently changes the concentration of the algae within the laser beam. This in turn can explain different behavior of the beam in the medium with and without algae.

We discuss the mechanisms which led to narrowing of the beam including nonlinear effects as well as potential applications in waveguiding, medical imaging and optimal propagation of laser beam in biological suspensions.

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# Kvantni generator sa solarnom pumpom sa strane teorije, eksperimenta i stvarnosti



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## Apstrakt

Kvantni generatori sa solarnom pumpom i drugim svojevremeno egzotičnim načinima eksitacije ka sistem koji su svoj razvoj počeli još u doba latne kvantne elektronike, i dalje su u žiži istraživanja u nekoliko područja, počevši od energetike, do svakodnevnih potencijalnih primena. Zavisno od aktivnog materijala, koji je ocenjen kao potencijalno pogodan, do drugih razvijenih metoda pumpanja (nesolarne prirode) neki od materijala su se već dokazali kao dobri aktivni materijali, a drugi su još u procenama i analizama mogućih zahteva i daljih poboljšanja. U radu je dat nekoliko formalizama, s obzirom da se radi o raznim mehanizmima pobude, postojeće ili procenjene inženjerske karakteristike i postojeće ili potencijalne primene sa budućim trendom.

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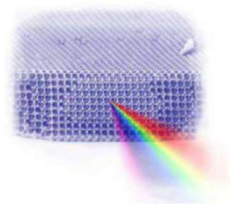
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## Laser beam waveguiding capabilities of the suspension of *Chlorella sorokiniana* in water

Aleksander G. Kovačević<sup>1</sup>, Tanja Pajić<sup>2</sup>, Danica Pavlović<sup>1</sup>, Marina Stanić<sup>3</sup>, Marina Lekić<sup>1</sup>, Olga Fedotova<sup>4</sup>, Stanko N. Nikolić<sup>1</sup>, Oleg Khasanov<sup>4</sup>, Ryhor Rusetski<sup>4</sup>, Najdan Aleksić<sup>5</sup>, Branislav M. Jelenković<sup>1</sup>

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**Abstract.** Controlled light guiding to target regions in biological and biomedical systems is important for applications like sensing and diagnosis. The penetration depth in tissues, limited due to scattering, is increased by using conventional optical waveguides, built on materials like silica glass and hard plastics. More potential for formation of biophotonic waveguides having higher biocompatibility and biodegradability have natural biomaterials, like living cells.

Strong scattering and absorption loss in cells is overcome by nonlinear effects arising during laser light propagation through suspensions of living cells, like marine bacteria [1]. Microalga *Chlorella* shows more attractiveness due to robustness, simple structure, high growth rate and ability to grow in various conditions, and its species *Chlorella sorokiniana* is most robust and most resistive to heat and intense light [2, 3].

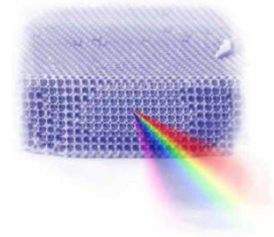
We examined the propagation of the 532 nm CW laser beam of various powers through the suspension of freshwater green microalga *C. sorokiniana* of various concentrations. Due to nonlinear effects, like thermo-optical, scattering, optical gradient forces, the beam modified. Self-guiding and the changing of cross-section occurred for chosen parameters of power and concentration. Some of the outcomes might be of interest for applications in biophotonics and biomedicine: waveguiding, medical imaging and optimal propagation of laser beam in biological suspensions.

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## Beam modification during propagation through aqueous microalgae suspension of interest to waveguiding

Aleksander Kovačević<sup>1</sup>, Tanja Pajić<sup>2</sup>, Djordje Jovanović<sup>1</sup>, Marina Stanić<sup>3</sup>, Danica Pavlović<sup>1</sup>,  
Olga Fedotova<sup>4</sup>, Oleg Khasanov<sup>4</sup>, Rygor Rusetski<sup>4</sup>, Marina Lekić<sup>1</sup>, Branislav Salatić<sup>1</sup>,  
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**Abstract.** *Chlorella sorokiniana* Shih. et Krauss [1], due to its highest resistivity to heat and high light intensity among all *Chlorella* species [2], is a good candidate in the applications of light generation, waveguiding and modulation. Relative refractive index with respect to water is makes the cells the positive polarizability particles and lowest absorption in the green region of the visible spectrum [3] reduces the thermal effects generated from the propagating high power laser beam.

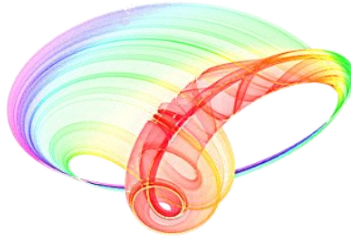
During laser beam propagation through aqueous suspensions of metal nanoparticles or microscopic marine bacteria, nonlinear effects, like thermo-optical, scattering, optical gradient forces take place in shaping the beam [4, 5]. However, strong thermal absorption of metal and sensitivity to strong light of cells limit the range of beam power. We examined the propagation of the 532 nm CW laser beam of various powers through the suspension of freshwater green microalga *C. sorokiniana* of various concentrations, placed in a glass vessel. For two concentrations of algae ( $0.5 \times 10^7 \text{ cm}^{-3}$  and  $1 \times 10^7 \text{ cm}^{-3}$ ) and several selected values of beam power (2-4 W) the beam experiences self-guiding and changes in exit cross section [6]. In this work, we pay attention to broader range of powers (0.1-5 W) and concentrations and investigate the diameter change during propagation and the cross-section change at exit wall of the vessel due to nonlinear effects, which might be interesting for waveguiding and optimal laser propagation in biological suspensions.

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### **3. Optical materials**



## Real-time fabrication of microstructures on the modified chitosan

B. Murić, S. Savić-Šević, A. Kovačević, D. Pantelić and B. Jelenković  
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Chitosan is a natural polymer, partially deacetylated derivative of chitin extracted from crustaceous shells, insoluble in pure water and organic solvents, but dissolves in diluted acidic solutions. Biocompatible, biodegradable and nontoxic nature of chitosan makes it a suitable polymer for various applications (biomedical, engineering, food, pharmacy, etc.) [1,2].

A simple and cheap method for preparing modified chitosan (MC) layer as ecofriendly, optically transparent, elastic, and durable material, for microstructures fabrication is presented. The anthocyanin food dye (E163) was used for MC layer sensitization makes it responsive to experimentally used blue laser light. The MC has no toxic effect, that confirmed by the biocompatibility test. The various microstructures were produced on the MC layer by direct focused laser radiation at 488 nm, with maximum output power of 100 mW, using homemade laser writing system. The fast produced concave or convex, aspheric microlenses (individual or closely packed arrays), microchannels, diffraction gratings, various complex structures etc. can be used directly, without any additional chemical processing and any waste, for a variety of applications such as: lab on a chip, medical laser, optical sensors, light-field cameras, security, biological structure, etc [3-5].

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## **Laser Additive Manufacturing Techniques**

**Milesa Srećković<sup>1</sup>, Aleksandar Bugarinović<sup>2</sup>, Nada Ratković Kovačević<sup>3</sup>, Željka Tomić<sup>4</sup>, Stanko Ostojčić<sup>5</sup>, Aleksander Kovačević<sup>6</sup>**

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Lasers used in versatile industries and aspects of life [1]-[10], in manufacturing are exploited for decades in various ways: for material characterization, for measurement and control, “subtractive” or classical processing\ machining, post processing and for LAM. LAM can be elaborated and classified with respect to: materials used, design, technologies, or applications. Variations in terminology exist, since the term Laser-based additive manufacturing (LBAM) can be found also, referring to a versatile manufacturing technique, extensively adopted to fabricate metallic components of enhanced properties [11].

Laser techniques, being one of ELION techniques, could be compared in many ways with respect to the specified role. An operational range of the processing methods grouped within ELION techniques should be expanded with respect to their emerging point. Comparison can or should be made, especially for large endeavours, where potentially positive and negative aspects can be easily found without consequences.

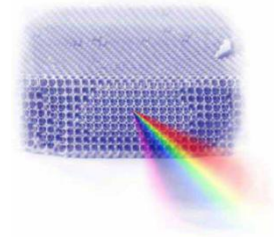
The comparison having more generalized or more sophisticated nature is performed for the chosen cases and for at least two techniques in paper presented. Special attention is paid to metrological aspects of more or less destructive laser applications and techniques, i.e. the list of some kind is presented in generalised manner in *descending order of provoked damage*. There are also many of laser-material interactions which are of destructive nature, however these are either potentially used or serve only as a replacement for incoherent methods, which gained in accuracy. A selection of results, obtained from several experimental testing of few processes, is presented.

In particular the paper considered chosen applications of LAM technologies in Biomedical Sciences and Medicine. Attention is paid to the analytical methods as well and some results with the laser interaction will be given.

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Institute of Physics Belgrade  
Kopaonik, March 10-14, 2024



Book of Abstracts  
**17<sup>th</sup> Photonics Workshop**  
(Conference)



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## Single-pulse and scanning multiple-pulse ultrafast laser beam interaction with Ti/Zr multilayer thin films

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**Abstract.** Ultrafast laser surface structuring gains more popularity due to the ability to improve material characteristics, such as reduction of friction and wear, improving solar cell performances, activate biomaterials, increase wettability etc. [1]. Compared to longer-pulsed beams, femtosecond beam interaction with materials has small heat-affected zone (HAZ) leading to fine modification with precise ablation [3]. Linearly polarized ultrashort laser beam (multiple pulses) can generate periodic surface structures (LIPSS) of sub-wavelength spatial periods on the surface of materials among which metals are of interest [4-6].

Multilayer nano-scaled thin metal film materials are attractive for applications in biomedicine as implants or tools, as protective coatings, optical devices, catalytic components... [7]. Having similar characteristics, Ti and Zr are metals interesting to combine in specific geometries, like multilayer thin films. The interaction of ultrafast laser beams with multilayer Ti/Zr thin films can lead to the formation of LIPSS and to selective ablation (layer-by-layer) of the material from the surface [8].

After static single-pulse irradiation for specific range of pulse energies, ablation region in the form of concentric circles appeared. For both lower and higher energies, the number of circles decreased. During multipulse scanning irradiation, where scan velocities varied, LIPSS of different spatial periodicity were formed.

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## Contemporary laser techniques, general application in heritology and case of building in 7 Balkanska street, Belgrade

### ABSTRACT

*It seems that the role of lasers in heritology and art has grown more and more since the end of the 20<sup>th</sup> century; the early attempts of cleaning artistic objects, old monuments / artifacts during the times of Hedy Lamarr and the first unsuccessful nuclear tests as well as of thinking of holographic records. After the first series of circumstances linked to laser applications in restoration and conservation, it seems the coupling of words serendipity-zemblanity-bahramdipity has been activated. A long time has passed since the first works linked to the Porta della carte of the Palazzo Ducale (Doge's Palace) in Venice (marble relief and ruby laser). Nowadays, this type of work can be treated as standardized and it is implemented in great number of countries [1, 2]. In the case of Florence Cathedral, the conservation of artworks was proposed by J. F. Fonatello, panels of the Giotto's tower of the Florence Cathedral by Andrea Pisano.*

*The unique roles of quantum generators – lasers – exist both in restoration and diagnostics. Besides that, the question of source existence – a source that provides completely new artistic impression with respect to its ideal characteristic of coherency – introduced new tools and techniques and could be (and was) implemented in many new processes and effects. New artistic directions were performed, where the source of the coherence became a part of a new artistic object, a hologram slide provokes the impression of the train entering the crowd, etc. [1–5] The laser role by using LIBS method in diagnostics in the case of the building in 7 Balkanska street, Belgrade, was presented in this paper.*

**Keywords:** laser methods, LIBS, heritology, spectroscopy.

### 1. INTRODUCTION

#### 1.1. Laser methods

Considering of large meaning of cultural heritage, generalization of material study could be monitored through: mechanical, corrosion, optical, dielectric, acoustic or other performances and appropriate methods. Special role in monitoring is paid to lasers scattering either in static or dynamic regimes. Some of methods cover quantum generators in the region of ultrafast regimes (zeptos) up to CW regimes. They are of interest for diagnosis of material quantifications including the shape of treated artifacts of cultural heritage. The most usual are quasi elastics Rayleigh, Brillouin, Raman scattering and their variants.

Laser spectroscopies with complementary techniques (infrared – IR), could be applied as a tool for the investigation of microstructure up to residual stresses. With inclusion of holographic methods many answers could be obtained (seismic data of environment). Note that first mentioned laser scattering is employed also for control of ejected particles (material) by laser cleaning of artifacts [1–9]. Material performances are expressed in various formalisms that include complex expression of all response functions and higher orders of magnitudes in the case of nonlinear optics. Depending on input laser parameters and demanded responses, respective techniques should be chosen. Contemporary theories and optical/acoustic methods give new possibilities and links with mechanical elastic moduli. Values for sound speed in materials implemented in calculations of characteristic frequency shifts could be used in corresponding laser techniques (stimulated Brillouin scattering). It is obtained by monitoring basic laser lines, scattered line shapes, their shifts and provoked other nonlinear effects, when the intensity of incident laser beam exceeds threshold values for

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stimulated processes (second harmonic generation – SHG – up to the disintegration level-threshold for *hardness* of material to radiation). Note that the obtaining the prediction of the disintegration threshold from measured linear Rayleigh components [10,11] is important.

The answer on fundamental relationships which link the interactions of electron, X, gamma and other photon (quasi-photon) beams of electromagnetic (EM) radiation with relations between moment and energy, is sought during making the decision which technique will be used for measuring the chosen processes [6]. The key parameter in selection of measuring techniques is energy transfer vs. moment transfer during scattering of various beams of radiation and particles.

Details could be followed through abundance of literature on ELION techniques, ELION being one-word abbreviation which denotes electrons, lasers, ions and neutrons [6,12]. Interesting conclusion could be retrieved on the choice of measuring techniques/spectroscopy (visible photons, X-rays, neutrons) comparing flux of monoenergetic quanta, associated wavelength,  $\Delta\lambda/\lambda$ , coherent volume, degeneration parameter and quantum counting efficiency. Being new sources with frequency tuning, numerous laser types open possibilities for obtaining various relaxation times and monitoring of solid state dynamics. Abundance of literature provides some characteristics of sources of optical photons, neutrons and X-rays, which are used for purposes of scattering or of material interaction. During examining the interaction of EM radiation with material, is necessary to use classical tools for considering light scattering as like Maxwell theory, Hertz's potentials and Poynting vector.

Many techniques, including micro-interferometry, can be used for the estimation of practical technological questions in the research of metals, surface roughness/relief and the effects caused by various processing regimes. Of particular importance is to study the effects of processing finesse to surface reflectance. Surface physics and the propagation of ELION beams through two or more different milieux are particularly intricate areas both from experimental and from theoretical approaches [12].

### 1.2. Categories of newer spectroscopies

Spectroscopies have intruded in various areas of life: science and engineering and new categories with nano-particles have their place [12–18]. They master the same techniques which had been developed for sophisticated purposes of military or civil applications, and swiftly transfer to everyday

use. In such way, they helped each other in the frame of physics-chemistry-technology of materials, and measurement techniques. Instruments for reflection measurements in non-linear range are complex, and the moments of phase transitions could be monitored, rate of crystallization with laser-assisted processes, plasma phenomena, etc. In measurement hierarchy, precise measurements of reflection coefficient of various materials, leads to lower and higher-quality definition of material's reflective characteristics (color, gloss, haze, etc.). Results of radiation-matter interaction (melting, evaporation and ejection processes, micro-hardness changes etc.) are not in accordance with data obtained by the spectroscopic analyzes of ejected material [19].

Surface physics could be treated as a branch of science for the processes on the phase borders [14–16]. Basic surface parameters (composition, atomic distribution, electron states, morphology and texture), have a direct influence on functional characteristics: optical, mechanical, chemical, electrical and magnetic. This is important for material synthesis in contemporary technologies: nanoelectronics, optoelectronics, heterogeneous catalysis, protective layers with high micro-hardness and with long chemical *durability*, magnetic memories. For most of them, a task for quantum generators and stimulated processes could be defined. Many developed experimental techniques differ by the object of measurement: sensitivity, information depth, lateral resolution and destruction. There is no technique which encloses only positive aspects, not even for a unique object of measurement. Complementary techniques have become *the rule*. Many of them are connected to laser-material interactions. Among them are: direct recoiled spectroscopy (DRS) and low-energy ion scattering spectroscopy (LEIS), used for compound analysis and the distribution of atoms. The list of techniques is broadened with mass analysis, secondary ion mass spectroscopy (SIMS) and mass spectroscopy of direct recoiled ions (MSRI). They provide deep profiles to analysis. LEIS and DRS are superior to SIMS and MSRI in surface sensitivity. The implementation of laser spectroscopies can give: electron structure, surface symmetry, surface morphology by laser speckle analysis, dynamic processes on surfaces. They are non-destructive and are used for analysis of both transparent and non-transparent materials. Obtained information on optical characteristics enables research of phase boundaries inside sample. Detailed examinations *in situ* of epioptics implemented to good characterization of system under ultrahigh vacuum (UHV) still are not numerous.

At least 17 new methods for characterization of dielectric susceptibilities of various orders in formalisms of nonlinear and epioptics could be found as a support. The same holds for optical absorption measurements where are calorimetry, photothermal, photoacoustical, photoconductive and photoluminescence excitation spectroscopies. Some of new techniques as LIBS (Laser Induced Breakdown Spectroscopy), LAMS (Laser and Molecular Spectroscopy), LAMMA (Laser Microprobe Mass Analysis), SHG (Second Harmonic Generation), and other non-linear techniques are linked to microscopic destructive processes. Some of our results are linked to computer approaches that may lead to transformations for melting or other phase changes. With respect to materials (dielectric, metal and semiconductor) and dynamical regimes of operation, new theories could be included for ultrashort interaction (some of them already penetrated into nucleus). Specially, fields that include up-to-now separated processes are developed thank to non-linear phenomena.

## 2. THE CASE STUDY OF BUILDING IN 7 BALKANSKA STREET AND LIBS SPECTROSCOPY

In case of the building of the Central Institute for Conservation (CIK) in Belgrade, various methods should be applied for obtaining an objective comparison between old and contemporary conditions, always in accordance with the regulations of heritage preservation and construction requirements. Diagnostics could be performed by laser and conventional techniques and in this paper weather stripping (mortar) is analyzed by LIBS. More informations could be obtained after the implementation of several techniques, which can be used for the estimation of the age and quality of old or recent materials, used since the construction times to contemporary times.

Besides conventional methods, new methods for historical houses with walls of pressed soil and weather stripping – mortar – have also been analyzed and developed nowadays [20–22]. Characterization usually begins from old methods, but among the new ones many are based on quantum generators in various working regimes. (Old methods for better understanding weather stripping/mortar include many of next characterizations: proportioning – water, consistency, water/cement ratio, water/binder ratio, compressive strength, flexural strength, elasticity modulus, density of hardened mortar, capillary water absorption; diagnosis of main parameters affecting contact area between mortar/weather stripping rendering and substrate – 3D laser

scanning). There are many publications about historical engineering materials of the object implemented. A number of test groups have been set. These are the characterizations of chemical and mineralogical characteristics, physical and mechanical characteristics, particle distributions and trembling. The determinations can be grouped into two instrumental techniques: XRF/XD and SEM/XRD, as well as the adaptations of standard UNE-EN methods for investigations and qualifications of these materials. The example of the ramps in Seville and Malaga may serve as a case study. It is a masonic construction of modular type, where weather stripping has been put and it has been filled with dirt and other components and tamping down in batches.

Rammed earth is in the focus of research of old buildings: in the context of sustainable building, contemporary interest for the earth as an engineering material is widely developed. Heritage of rammed earth in Europe and world is significant and scientific approach is being held. Durability and sensitivity to water, thermal characteristics, life comfort and mechanical compressive strength should have been taken into account.

### 2.1. Short Historical background of building

Here, some historical details of analyzed object will be presented. The object was erected for business purposes, built in around 1925. It is shaped in the spirit of late academism with elements of secession in decorative solution in the gable from the central part of the roof, and fences of the balconies of floor etages. Façades are flat and simply shaped with shallow side ryalites with encapsulated windows. The name of the architect who conducted the project is unknown, because the original documentation disappeared during the bombing of Belgrade in the 2<sup>nd</sup> World War. From the aspect of protection agency, it is valorized as an architectural-urban value harmoniously incorporated into the historical ambient of the Terazije area which enjoys the status of the heritage under previous protection.

It is in the same lot with Anker Palace, which was built by “Anker” Vienna Insurance Company in 1899 under the project of architect Milan Antonović. This area incorporates “Atina” palace, hotel “Moskva”, the house of Aleksa Krsmanović and the building of the Bank of Smederevo as a part of a representative civil engineering ensemble of Terazije formed in the end of the 19<sup>th</sup> and the beginning of 20<sup>th</sup> century [23].

In Figs. 1 and 2. the pictures of the object are presented, including details of deteriorated surfaces.

Layers of mortar (weather stripping) are clearly visible on many places.



*Figure 1. Front view of the main front façade of the object*

*Slika 1. Pogled sa prednje strane na glavnu fasadu objekta*



*Figure 2. Another view of the main façade of the object*

*Slika 2. Bočni pogled na glavnu fasadu objekta*

## *2.2. Some performed experiments with LIBS for further restoration*

Part of the study of buildings of interest for cultural heritage could be and should include the state of the building's material. There are many standard techniques for the testing of the civil engineering materials [21, 22, 24–27]. Having in mind that here it started with laser techniques suitable for buildings, their parts, interiors and exteriors, first usable techniques could be and are LIDAR, scanner, range finders, and other developed systems that are based on some of the quantum generators [28-30]. Here is chosen the

state of weather stripping from some special places that are selected from by experts from the field of restoration [23]. In theory, material for restoration should be, if possible, similar to the initial used material.

LIBS (or LIPS) is chosen parallel with other non-laser systems and methods to reveal and specify components of selected samples of mortar (weather stripping) from the façades. For the beginning, here is started with LIBS experiments. It means that there is a possibility to analyze the interaction between laser beam and mortar (weather stripping) or to see the contents of the material. Further analysis with complementary

techniques is expected to reveal whether the material is from the time when building has been erected or some other details about influence of weather stripping on mortar up to day. LIBS techniques could be defined through ~18 techniques among which are based on the interaction of various laser types: micro-destructive 2, destructive 10, non-destructive 6 (among which some of them are both destructive and non-destructive). LIBS system is micro-destructive was non-portable in the beginning [22]. LAMMA is excluded in the table from this reference. LIBS is chosen as a relatively new technique for material analysis; it is linked to the development of lasers, spectrometers and detectors.

LIBS techniques show lower precision compared to other analytical methods, but in turn are contactless and suitable as a preliminary investigation. The methods are characteristic for their complexity of interaction, but some depend on chosen laser and material. In general, the methods are based on the analysis of plasma radiation. The emission spectrum of provoked plasma consists of atomic, ionic and molecular states, etc. The inhomogeneity and time dependent changes of the shape of LIBS plasma are followed by the change of plasma parameters, specific distribution, spectral lines and the emission in plasma plume. Therefore, the spectra interpretation might be hindered [28–38].



Figure 3. Mortar sample ( $\approx 5$ cm long) subjected to analysis with microdamages provoked by LIBS

Slika 3. Uzorak maltera ( $\approx 5$ cm dužine) za analizu; mikroštećenja su nastala tokom LIBS analize

One of the samples, presented in Fig. 3, has subjected to the analysis of weather stripping composition by monitoring the most intensive recorded lines. Those samples will be the object for further investigations, respecting to the interaction with one or more laser beams. Here, the damage obtained by LIBS is presented as macroscopic view and further by spectral diagrams, with markings on curves offered by LIBS. After those recordings, the same samples will undergo to XRD

analysis, and provoked microdamages need applying using techniques as SEM or TEM, may be other complementary spectroscopies.

### 2.3. LIBS spectra of mortar samples

Here, three samples were selected for the analysis with notation as follows:

1. Sample 1 - small grey mortar;
2. Sample 2 - large grey mortar;
3. Sample 3 - pink mortar.

The plasma was generated by a Nd<sup>3+</sup>:YAG laser (Q-smart 450) emitting 5 ns pulses at 1064 nm. The beam was guided by mirror (45° angle) to focusing lens of 10 cm focal length placed perpendicularly to the target. The mortar target was positioned 1 mm in front of the focal point, so the 0.5 mm spot was obtained. The incident laser energy on the sample was 500 mJ. In such conditions, the laser fluence was 2.5 J/mm<sup>2</sup>.

In Fig 4, the macroscopic image of a crater after LIBS action is presented as a detail from Fig. 3. The damages could be the object of further analyses in the aim of finding the relation between laser parameter and provoked damages at the materials. Here, in the first plane, are only the results of the analysis of ejected materials via plasma phenomena.

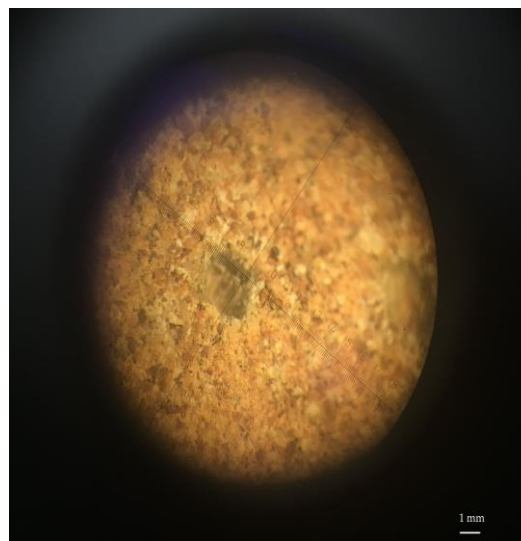


Figure 4. Macrograph of the area marked as B 9955, recorded by optical camera

Slika 4. Makrosnimak površine označene kao B 9955, snimljeno optičkom kamerom

Generally, there is not much difference in the shape and qualitative parameters of the created damages. Plasma radiation was through the entrance slit (50  $\mu$ m wide) of the Shamrock 303 (Andor) imaging spectrometer using mirror and quartz achromatic lens (focal length  $f = 33$  cm). The change of the diffraction grating (300, 1200 or 2400

grooves/mm), slit width and wavelength position were performed using commercial Andor Solis software. The instrumental width with 1200 grooves/mm grating and 50  $\mu\text{m}$  slit width, determined using Oriel penlight calibration lamps was 0.35 nm.

The acquisition gate width,  $t_G$ , and delay,  $t_D$ , are determined with digital delay generator, DDG (Stanford Research Systems SRS, Model DG535). The spectra were recorded using gate delay of 0.6  $\mu\text{s}$  and gate width of 100  $\mu\text{s}$ . Such gating is a standard LIBS procedure which prevents the recording of pronounced continuum radiation in the

first 0.6  $\mu\text{s}$ . Much faster decay of the background emission versus line emission opened the possibility for improvement of S/N ratio of lines of interest by selecting proper time for spectra recordings. The accumulation of the signal was necessary due to the very weak signal of lines. For that reason every spectra were accumulated over 10 laser shots.

In Figs. 5–10, some characteristic LIBS spectra of samples are presented by using common denotation which are commonly used in describing of LIBS results, according to the appropriate literature [32-34].

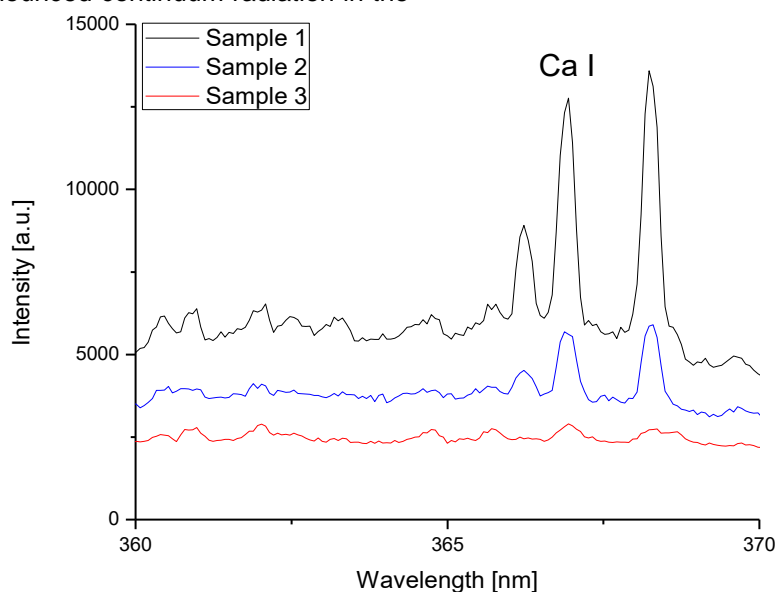


Figure 5. LIBS of the samples in the range 360–370 nm

Slika 5. LIBS uzoraka u opsegu frekvencija 360–370 nm

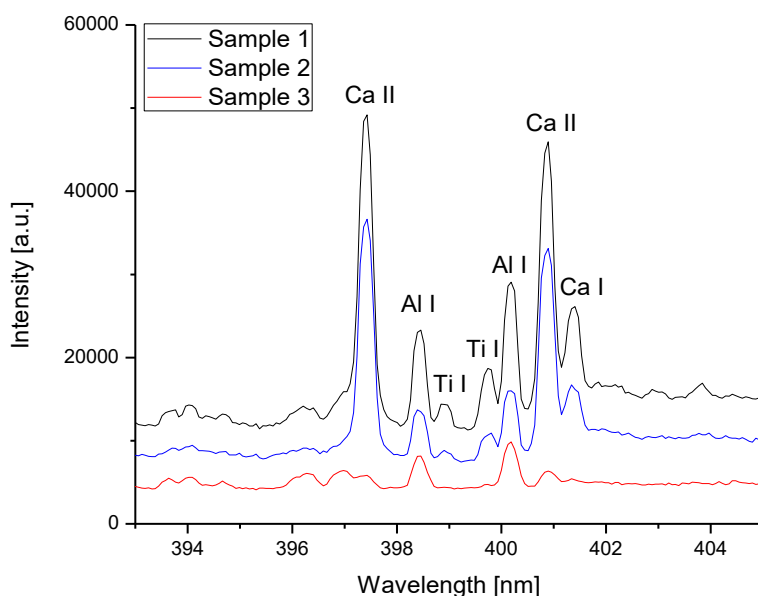


Figure 6. LIBS of the samples in the range 394–404 nm

Slika 6. LIBS uzoraka u opsegu frekvencija 394–404 nm

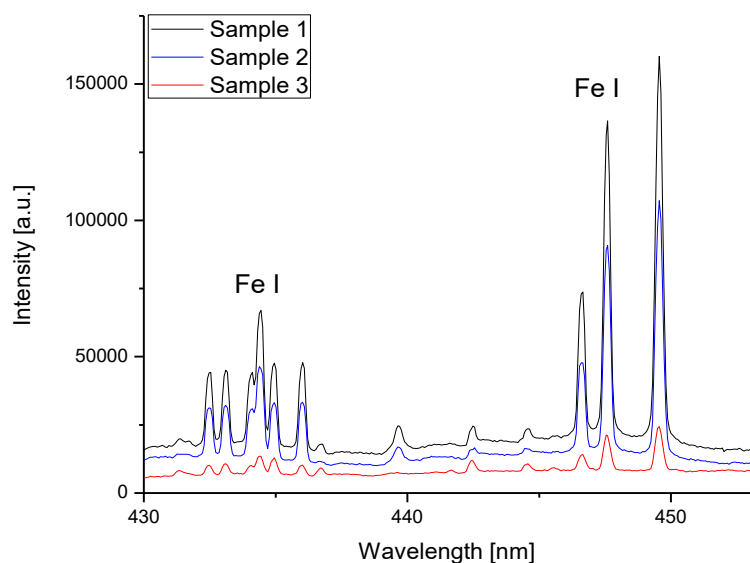


Figure 7. LIBS of the samples in the range 430–450 nm

Slika 7. LIBS uzoraka u opsegu frekvencija 430–450 nm

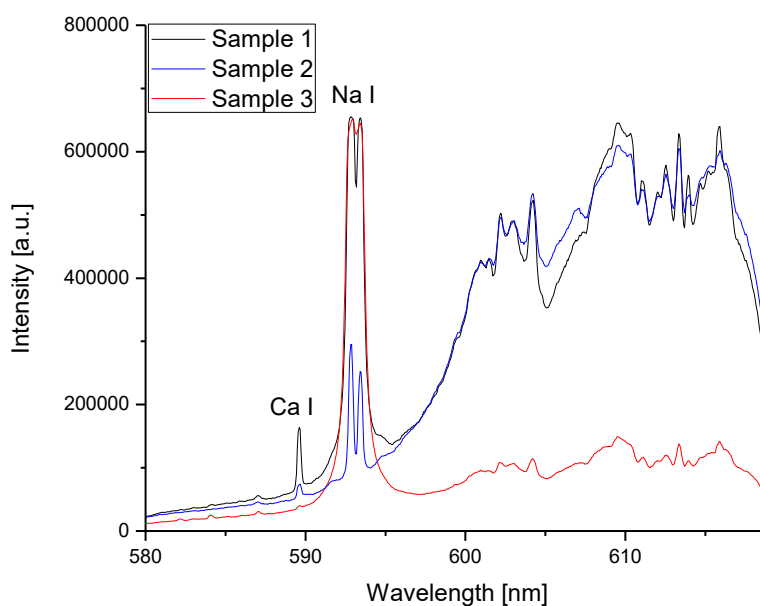


Figure 8. LIBS of the samples in the range 580–618 nm

Slika 8. LIBS uzoraka u opsegu frekvencija 580–618 nm

### 3. DISCUSSION

For this preliminary investigation of chosen samples, LIBS was used as *contactless* method which provokes minor material damage. Detailed investigation of material and the interpretation of obtained LIBS data, however, are in correlations in complex theoretical and experimental facts, as a consequence of searching the correlations between the found element position at the Periodic

table, their characteristic constants linked to ionisation, thermodynamics data, surface physics also with depth of the laser provoked craters, etc.

LIBS spectra from Figs. 5–10 represent the identification of several elements found in samples 1–3. At those wavelengths and recording parameters, here are registered peaks from resonant transitions of those elements (in form of excited ions), when excitation is performed during

interaction of laser beam with target surface, assuming that multi-photon transitions did not take place. The aim in this moment is to find out preliminary results, and after that to use various other methods based on other systems as non-destructive spectroscopies but more conservative

methods, in which the sample will be crushed to powder state. Other techniques might be used for defining specimen's color, before and after laser treatments in solid state, by using quantitative formalisms, etc.

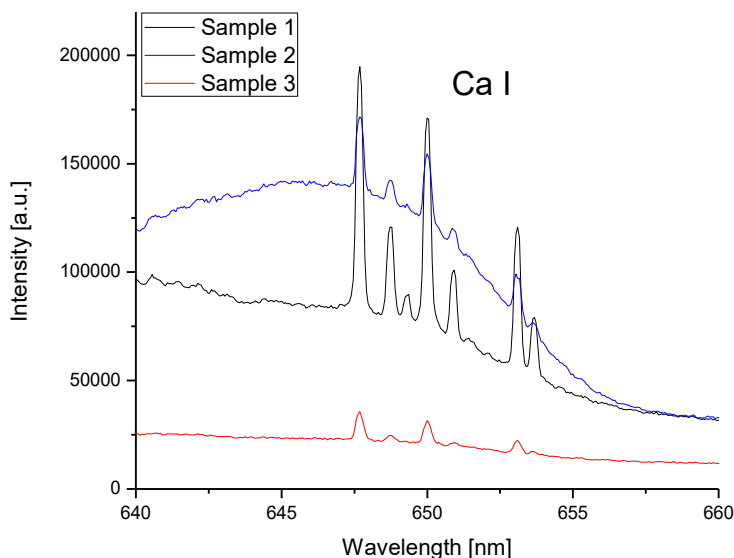


Figure 9. LIBS of the samples in the range 640–660 nm

Slika 9. LIBS uzoraka u rasponu frekvencija 640–660 nm

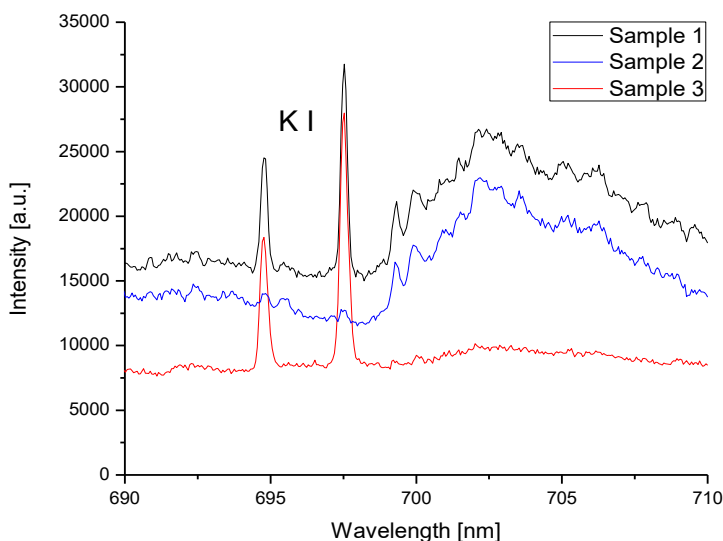


Figure 10. LIBS of the samples in the range 690–710 nm

Slika 10. LIBS uzoraka u rasponu frekvencija 690–710 nm

The explanation of provoked dynamic plasma state is complex task that presume to use complementary techniques and theoretical models. Our approach was to perform experiments and to analyze the most intensive detected processes. By

analyzing the spectroscopic view in short range of wavelengths, only the most intensive peaks were found and identified as clear representatives of existing transitions. Each laser spectroscopy as well as spectroscopy with non-coherent sources is

characterized with their own spectroscopy formalisms. Note that in some of them parallel exist two formalisms. Here are used common notations for LIBS, and its database in which are recognized the state of some constituent of samples. The identification of Al or Ti could be found in the literature [37,38]. More details for composition of used samples should be expected in further investigation, for example by using infrared (IR), Raman and other techniques as well as XRD, or chemical analysis. For Raman and IR spectra there is a direct correspondence among the two spectra which enables the interpretation of the connection between the materials, their bonds etc.

For materials with unknown content, as is in the case of cultural heritage artefacts, it is necessary to damage the specimen as little as possible. Sample preparation for electron microscopy or some other technique needs the transformations of sample into powder. In this way, the samples will be completely destroyed and lost for further evaluations.

Spectra of two grey mortar samples are similar, with all analyzed lines in small grey mortar sample spectra being more intensive. In grey mortar sample spectra it was possible to detect Ti I which is not present in pink mortar sample. Nevertheless, those given lines are detected also in pink mortar sample, in which their intensities are significantly greater, together with the concentration of given elements: Fe I, Ca I, Al I. The only difference between two grey mortar samples is that the small grey mortar sample has K I, while large grey mortar sample has not. In the pink mortar sample spectrum K dominates. There is more Na than in grey mortar samples. In the spectrum, the lines of Fe I, Ca I, Al I are also detected, but they are of significantly lower intensity than in grey mortar samples. These initial data could be used for further refinement of the LIBS measurement possibility.

#### 4. CONCLUSION

Chosen examples from the cultural heritage here are investigated by LIBS, which was developed on the basis of long-term research and couplings with laser-plasma-diagnostics-production methods. Comparing the damages of three samples, it could be noticed that there are no much differences in the shape and quantitative parameters of the performed damages.

Since laser induced breakdowns cause complex atmospheric compositions, identification is performed among ion transition of the elements as like Ca, Na, Al, Fe, Ti, when characteristic notations were used as valuable for this spectroscopic method. At wavelengths applied here it was not possible to register elements as like silicon or oxygen, for which other laser or non-laser

techniques should be used in further investigations, due to the complexity of the problem.

For further investigation, the answers according to the question on the relation between SEM and EDX to the LIBS method, their advantages also disadvantages of these parallel techniques, are expected. Discussions on various contemporary and classical types of mortar and other applications of laser related with cleaning of façades will be provided.

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## IZVOD

### SAVREMENE LASERSKE TEHNIKE I OPŠTA PRIMENA U HERITOLOGIJI I SLUČAJ ZGRADE U BALKANSKOJ ULICI 7, BEOGRAD

Čini se da je uloga lasera u heritologiji i umetnosti u stalnom porastu od kraja 20. veka; rani su pokušaji čišćenja umetničkih objekata, starih spomenika ili artefakata od vremena Hedi Lamar (Hedy Lamarr) i prvih neuspešnih nuklearnih testova, kao i razmišljanja o holografskim zapisima. Posle prve serije okolnosti vezanih za primene laserskih tehnika u restauraciji i konzervaciji, izgleda da je sprezanje reči serendipity, zemblanity i bahramdipity aktivirano. Prošlo je mnogo vremena otkad su obavljani prvi radovi vezani za Porta della Carta Duždeve palate (Palazzo Ducale) u Veneciji - mermerni reljef i rubinski laser. Danas ovaj tip radova može da se tretira kao konvencionalan/standardan i primeni u nizu zemalja [1, 2]. U slučaju Katedrale u Firenci Fonatelo (J. F. Fonatello) je predložio konzervaciju umetničkih objekata, Pizanove (Andrea Pisano) panele na Đotovoj (Giotto) kuli Katedrale.

Jedinstvene uloge kvantnih generatora – lasera – postoje i u restauraciji i u dijagnostici. Osim toga, pitanje postojanja izvora – izvora koji nudi potpuno novi umetnički utisak u odnosu na idealne osobine koherencije – uvelo je nove alate i tehnike i moglo je da (i jeste) primeni mnogo novih procesa i efekata. Novi umetnički pravci su izvedeni, gde je izvor koherencije postao deo novog umetničkog objekta, hologram je izazvao utisak da voz ulazi u masu ljudi, itd. [1–5] Uloga lasera u dijagnostici i u svrhu konzervacije i neke primene na slučaj zgrade u Balkanskoj 7 u Beogradu je prikazana u ovom radu.

**Ključne reči:** laserske metode, LIBS, heritologija, spektroskopija.

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Златко Јарневић, Друштво за ЕТРАН



SS-FO1.8 DIGITALNA FORENZIKA - PRAVCI RAZVOJA NASUPROT  
AKTUELNOJ PRAKSI

*Milica Janković, Radovan Radovanović, Nikola Mitrović and Milan Jovanović*

Дигитална форензика представља најактуелнију грану форензике. Како се развија информатичка наука и технологија у целини, тако се и дигитална форензика мора развијати да би пратила трендове и дала решење за све проблеме у свом научном домену. У овом раду се приказују планиран напредак у оквиру ове науке, као и тренутни поступци у пракси, и даје се њихова упоредна анализа.

**SPECIAL THEMATIC SESSION: MULTIDISCIPLINARY  
SESSION/  
SPECIJALNA SESIJA: MULTIDISCIPLINARNA**

**SS-MD1**

Utorak/Tuesday, Jun/June, 06<sup>th</sup>, 15:00 – 18:00, Sala 5/Hall 5

**Chair/Predsedavajući:**

**Mileša Srećković, Univerzitet u Beogradu, Elektrotehnički fakultet, Beograd, Srbija**

SS-MD1.1 LASER INDUCED SURFACE NANOSTRUCTURES AND POTENTIAL  
CONTEMPORARY AND FUTURE APPLICATIONS  
**INVITED PAPER**

*Aleksander Kovačević*

The interaction of laser beam with materials surface induces various effects and features, commonly leading to the modification of the material – both the surface and the bulk. Ultrafast laser beams have special properties which their interaction with the materials separate from the interaction with other types of laser beams. Pulse length is shorter than the response time of the material which enables material modification under specific conditions. The occurrence of plasmon polariton, together with the self-organization of the material, generates parallel surface nanostructures with period shorter than the beam wavelength. The potential applications are interesting and can be implemented in many areas.

SS-MD1.2 MIXED SPEARMAN'S CORRELATION SUITABILITY FOR SINE-  
SHAPED INDEPENDENT VARIABLES IN NATURAL SCIENCES

*Miodrag Malović, Vera Vukanić, Darko Jevremović and Ljiljana Brajović*

Pearson's and Spearman's methods are most often used in popular software packages to calculate the correlation coefficient between two variables in natural sciences. The difference between them is that the Pearson's method takes actual numerical values into account whereas the Spearman's operates with their ranks. Ranking is often efficient in dealing with nonlinearities and outliers in the data. However, not both independent (x) and outcome (y) variable have to be suitable for this. We propose not to rank the independent variable in case it comes sine-shaped. Most notable example of such a variable in nature is the temperature. It is sine-shaped due to day-night and summer-winter transitions. It does not contain many outliers, not only due to the shape, but also



Друштво за електронику, телекомуникације,  
рачунарство, аутоматику и нуклеарну технику

## ПОТВРДА

Друштво за ЕТРАН, Београд, Србија, исказује своје дивљење и захвалност

**АЛЕКСАНДЕРУ КОВАЧЕВИЋУ**

за презентацију РАДА ПО ПОЗИВУ

**“Laser induced surface nanostructures and potential  
contemporary and future applications“**

на Специјалној тематској седници Мултидисциплинарна  
на LXVII конференцији ЕТРАН – ЕТРАН 2023,  
у Источном Сарајеву, Босна и Херцеговина.

Источно Сарајево, Босна и Херцеговина, 6.6.2023.

Председник Друштва за ЕТРАН  
Проф. др Владимир Катић



## ЛАСЕРСКЕ ТЕХНИКЕ И СПРЕГЕ СА ДРУГИМ ТЕХНИКАМА У ПРОБЛЕМАТИЦИ ДАНАШЊИЦЕ У ТЕОРИЈИ И ПРАКСИ

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**Апстракт:** Проблеми данашњице, *pro et contra* нових продора у високе фреквенције, али и пандемија, као да су позвали на још сложеније корелације и спрезања области: наука, пракса, мас-медији у савладавању што боље дескрипције одређених процеса у заједничким напорима да се изађе из ситуације у којој се наш Глобус нашао. Више него икад, потребно је много мултидисциплинарног рада, али и уско специјалистичких софистицираних познавања области у којима се нешто истражује.

У широком и генерализованом приступу интеракције кохерентног зрачења са материјалом као да се губи граница између класичнијих прилаза расејању / интеракцији, јер је појам стимулираних процеса и врло кратких импулса отворио блиска подручја у спрезању са материјалом (у широком смислу укључујући и био-феномене) и тиме замаглио тренутак кад су живе ћелије почеле да учествују у оптичким записима, а познато је да статистика из биолошких процеса може да решава проблеме саобраћаја и сл.

У овом раду ће се размотрити интеракција материјала са стимулираним електромагнетним зрачењем, њени резултати и што тачнија дескрипција могућих процеса. Стављањем акцента на оптичку видљиву, инфрацрвену и ултраљубичасту област, скицираће се и неке примене осталог дела електромагнетног спектра и модулације материјала или њихове мерне могућности.

За изабране примере материјала разних класа, посматраће се њихов могући опис карактеристика пре и после интеракције са гледишта ласерских, термовизијских, и других техника, где долазе до изражаја за-

писи проузрокованих акустичких или низа других спрегнутих ефеката са терминологијом магнетно-оптички, електро-оптички, акусто-оптички, оптомеханички и сл. Размотриће се и изабрани теоретски сложенији модели са рачунарском подршком, али и једноставне крајње формуле, које воде до релативно корисних процена у исходу интеракције приликом праћења појединог канала излаза. У спектроскопском прилазу дискутоваће се делови спектроскопија који у себи имају ласерске или стимулисане изворе и тиме отворене нове или само побољшане спектроскопије.

Таквим прилазом, интерпретације постају веома осетљива подручја. Приступ примене ласерских и неласерских техника на материјале уопште (на биоматеријале, на објекте херитологије или за специјалне намене) и дефинисање узорака и мерења на одабраним објектима или материјалима, и пракса, теорија, реалност у областима културне баштине, медицине или специфичних области које укључују оштећења, корисне деструктивне процесе или експлозивне процесе при примени ласерских техника, опет отвара питања ласерских оштећења и њихових дефиниција. Улазак у области заштите са изазивањем пробоја ласером или иницијацијама разних процеса, мора да располаже са детаљним познавањем главних оптичких показатеља / дескриптора материјала, укључујући и појмове коефицијената рефлексије, расејања, апсорпције, термодинамичких параметара, тачку паљења, величине честица, ако је материјал у стању праха, итд.

**Кључне речи:** ласерска техника, интеракција, расејање, материјали, дескрипција, моделовање.

## 1. УВОД

### Рефлексија светлости на граници две средине

Као стари проблем са богатом литературом, рефлексија светлости на граници две средине, везана је за класичне резултате са *понашањем* електромагнетних таласа на граници и односи се на доласерски период. Тада су посматране само транспарентне и апсорпционе средине, као и средине су посматране линеарно (Оптички параметри нису зависили од густине снаге зрачења и од интензитета упадне светлости). За хомогене раванске таласе, никад се не прелази 1 и увек постоји пропорционалност са упадним зрачењем. Појавом услова и средина које појачавају, стављен је поново акценат на рефлекси она својства материјала. Сада се више није радило само о прецизнијим, већ о квалитативно новим резултатима, ефектима који траже нове интерпретације и формализме. Радило се о чињеницама: коефицијент рефлексије за оптички активну средину  $R > 1$ , могуће је и појачање, а у вези са тоталном рефлексијом на граници са нелинеарном средином, долази до појава оптичке бистабилности. Проблематика је привукла пажњу много истраживачких група и процветале су: интегрална оптика, резонатори, пасивни и

активни). Најважнији су проблеми: рефлексија од појачавачких линеарних средина и оптичких нелинеарних средина са Керр-овом или термалном нелинеарношћу. Махвелл-ове једначине су добиле нов значај и нове резултате, када је рефлексиона средина активна или нелинеарна. Показана је и могућност тоталне рефлексије од инверзних средина [1] и апроксимације у 2 случаја [2]. За класичну електродинамику допушта се у случају стационарних средина само рефлексија и  $R < 1$ , независно од карактера рефлексионе средине. Тако су реализовани нови квантни генератори и појачавачи [3] перспективни за интегралну оптику, активну средину у виду танких филмова и полупроводника. Разни модели укључују појачавачку средину као: хомогену полубесконачну средину са планпаралелним слојевима. Разматрана је и нехомогена инверзна средина са експоненцијалним и линеарним профилима  $\epsilon$  и реализовани технички детаљи. Шема ласера са тоталном рефлексијом на граници прозачне изотропне средине са активном појачавачком средином: самоинтеракције везане за снажне интензитете, а проблеми су управљање интензитетом, поларизацијом и правцем деловања. Ту су и нове законитости: феноменолошка теорија за Керров тип нелинеарности, угао Brewster-a, позитивна нелинеарност, интерференциони слој, оптички хистерзиси, нови типови око 1975 г [4]. Оптички хистерзис доводи до бистабилности и рефлексије и не само у „теорији“, него и у примени: управљања зрачењем, рс импулси, (оптички прекидачи и застори). Уз танке филмове, везују се Керров и топлотни ефекти. Нарушавање тоталне рефлексије и интерференције, може да се употреби за модулацију.  $R$  близу граничног угла тоталне рефлексије -  $TP$  показује да може да служи за управљање зрачењем и нелинеарним елементима; добијају се идеје за оптичке филтре интензитета и енергије, рефлекторе, модулаторе, прекидаче.

Рефлексија светлости на граници са хомогеном појачавачком средином

Разматрање проблема ове врсте, почело је 70-тих година прошлог века, када је експериментално показано [5] да при рефлексији од активне појачавачке средине, коефицијент рефлексије близу граничног угла тоталне рефлексије  $R$ , може да буде мањи од 1. При одређеним условима је могуће појачање ЕМ таласа. Из теорије, отишло се у примену у вези са реализацијом нових појачавача. У ласерској техници има своја преимућства. Просторно раздвајање у резонатору и активне средине и нехомогено грејање при генерацији погоршава спектралне и енергетске карактеристике. То снижава техничке захтеве за радне средине ласера, њихову хомогеност, прозачност. За добијање појачања процесом тоталне унутрашње рефлексије [6] је рађено и експериментално и потврђено појачање (и генерација), са танким стакленим влакном диметра  $8 \mu\text{m}$  окруженог љуском  $\text{Nd}^{3+}$  стакла диметра  $26 \mu\text{m}$ ; затим је следио слој дебелог и прозачног стакла ( $1,5 \text{ mm}$ ; дужина таласовода  $1 \text{ m}$ ). Индекси преламања су: језгро  $\text{Nd}$  стакла  $n_1=1,529$ ,  $n_2=1,509$  и спољног  $n_3=1,510$ . На границама пасивног језгра и  $\text{Nd}^{3+}$ :стакла су услови и за тоталну рефлексију.

Генерисано зрачење 1,06  $\mu\text{m}$  у том систему је добијано из пасив ног дела, по представама феноменолошке оптике; R на граници са инверзијом био  $R > 1$ , Таб.1.

Следује [7] да за углове упада  $\alpha$ ,  $\alpha < \alpha_0$ , далеко од ( $\alpha > 0$ ,  $\alpha \gg |\Delta(\omega)|$ ,  $\tau(\omega)$ ), величина помака посебно за танке слојеве (N мало), мора да се смањи,  $(|\delta|) \rightarrow 0$ ; највећи коефицијенти рефлексије од појачавачког слоја, треба да се очекују за фреквенције  $\omega$ ,  $\omega \approx \omega_p$ . Аналитичко разматрање зависности величине  $\delta$  помака од односа параметара слоја и околне средине, при разним угловима упада  $\alpha$  и *дебљина* слојева  $h/\lambda$  је сложено због облика зависности  $R_1(\omega)$  и  $\Delta_1(\omega)$ . Нумеричко решавање је разматрано са зависношћу  $\epsilon_2(\omega)$ [7]. За задате вредности параметара слоја и околне средине ( $\Delta\epsilon = \epsilon_1 - \epsilon_2$ ) и  $\tau_0$ ) израчунава се интервал могућих углова генерације  $\Delta\alpha = \alpha - \alpha_0$  и помаци од резонантне фреквенције,  $\delta = (\omega - \omega_p) / \gamma$  ( $|\delta| \leq 1$ ) и  $h/\lambda$ , Таб. 1,2.  $\epsilon_1 = 2,5$  ( $n_1 = 1,5$ ). Ту је фиксирана је вредност за  $\tau_0$  и мењано је  $\Delta\epsilon$ , а у Таб.2, фиксирано је  $\Delta\epsilon$ , а различите су  $\tau_0$ . Анализа података показује да у свим случајевима генерације за танке инверзне слојеве постоји у малој околини углова  $\alpha \sim \alpha_0$ . Порастом  $\tau_0$  (појачање у максимуму), или смањењем  $\Delta\epsilon$  тј. порастом параметра  $p = \tau_0 / \Delta\epsilon$ , интервал могућих углова генерације  $\Delta\alpha$  се шири. При великој разлици  $\Delta\epsilon$  диелектричних пропустљивости граничних средина и мањих  $\tau_0$  (параметер  $p$  мали), могући су углови генерације  $\alpha_{gen} < \alpha_0$ , смањењем  $\Delta\epsilon$  или порастом  $\tau_0$  (параметар  $p > 1$ ), могући су углови  $\alpha_{gen} > \alpha_0$ . Увек важи и зависност [7]

$$\epsilon_0 - \epsilon_1 \sin^2 \alpha + \Delta(\omega) > 0 \quad (1)$$

За углове  $\alpha_{gen} \geq \alpha_0$ , помак  $\delta$  је увек позитиван, генерација постоји за фреквенције  $\omega > \omega_p$  ( $\delta > 0$ ). У неким случајевима, могуће је  $\delta > 0$  и за углове  $\alpha_{gen} \leq \alpha_0$ . За углове  $\alpha_{gen} \leq \alpha_0$  или  $\alpha_{gen} \square \alpha_0$ , помак фреквенције  $\delta$  је увек негативан ( $\omega < \omega_p$ ). Прелазом генерације из области углова  $\alpha < \alpha_0$  у области:  $\alpha_{gen} \geq \alpha_0$ ;  $\alpha \geq \alpha_0$ ;  $\alpha \square \alpha_0$  фреквенција генерације се помера из резонансне ка вишим фреквенцијама. То се користи у експерименту, у тој области углова генерација постоји за  $\omega > \omega_p$  ( $\delta > 0$ ). Могуће је и ( $\delta > 0$ ) и за углове  $\alpha_{gen} \leq \alpha_0$  или бар приближно). По правилу за  $\alpha_{gen} < \alpha_0$  помак фреквенције  $\delta$  је негативан ( $\omega < \omega_p$ ). Тако прелазом генерације из углова  $\alpha_{gen} < \alpha_0$  у област  $\alpha_{gen} > \alpha_0$ , фреквенција генерације се помера од резонансне области у област виших фреквенција. То се користи, а о знаку помака  $\delta$  се суди о области углова генерације. Важна је зависност  $\delta$  при разним угловима генерације  $\alpha \approx \alpha_0$  и задатом  $\tau_0$  од односа диелектричних пропустљивости слоја и околне средине, од  $\Delta\epsilon$ , Таб. 3, за углове  $\alpha_{gen} = \alpha_0 \pm \Delta\alpha$ , где је  $0 \leq \Delta\alpha \leq 05^\circ$ ),  $\min.$ , за узете  $\tau_0$  и  $\Delta\epsilon$ , величина  $\delta$  је позитивна, расте, кад се  $p$  се смањује (тако се смањују и могуће

минималне дебљине  $h/\lambda$ ). Пораст је потврдио и експеримент [8], што важи и за смањење  $\tau_0$  при фиксираном  $\Delta\varepsilon$ , ако су углови генерације:

$$\alpha_{gen} \approx \alpha_0 \pm 0. \quad (2)$$

За углове кад одступања  $\Delta\alpha$  по апсолутној вредности расту, Таб.3,  $\delta$  је негативна и са порастом параметра  $p$  расте по модулу, што потврђује и експеримент [8]. Генерација постоји за  $\alpha_{gen} < \alpha_0$ . За веће коефицијената апсорпције  $R \gg 1$ , интересантни су и други оптички системи.

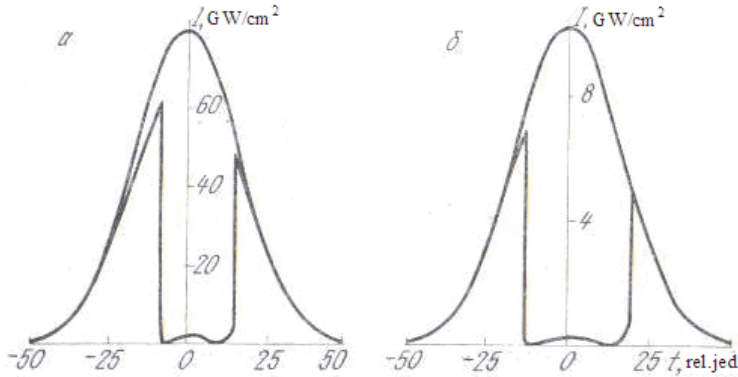
*Таб.1 Подаци за процену интервала могућих углова генерације  $\Delta\alpha = \alpha - \alpha_0$ . Дебљина појачавачког слоја  $h/\lambda$  и величина помака фреквенције од резонансне  $\delta = (\omega - \omega_p)/\gamma$  при задатом параметру појачања слоја  $\tau_0 = 3 \cdot 10^4$ . Део Таблице из [7]*

$\Delta\varepsilon(p)$ ,	$\Delta\alpha_{0,min}$	$\delta$	$h/\lambda$
$\Delta\varepsilon=10^{-2}$ ( $p=0,03$ ) $\Delta\alpha_0=86^{011}'$	-9	-0,52	18,4
		-0,06	17,0
	-5	0,75	20
	-4	0,99	21,6
$\Delta\varepsilon=10^{-3}$ ( $p=0,3$ ) $\Delta\alpha_0=88^{047}'$	-13	-0,79	26,6
		-0,40	23,1
	-10	-0,005	21,6
	-5	0,44	23,6
$\Delta\varepsilon=10^{-3}$ ( $p=0,3$ ) $\Delta\alpha_0=88^{047}'$	-14	-1.08	31,4
		-0,40	23.2
	-10	-0,06	21,1
	-5	0,24	21,3

#### Карактеристике рефлексије/транспаренције од оптички нелинеарних система

Ова област обухвата: појаве хистерезиса при постојању границе са нелинеарном средином, хистерезис фазе у условима тоталне рефлексије, интерференције са нелинеарном границом, рефлексију са оптички нелинеарним прозачним слојем, нелинеарни диелектрични слој као **филтар** интензитета ласерског снопа. Хистерезис фазе у условима рефлексије од интерференционог слоја са нелинеарном границом има карактеристичне измене форме Gauss-овог импулса при рефлексији, сл.1, [7] о нелинеарном прозачном слоју, уз интерференциона својства слојева. Тако су применом нелинеарне рефлексије од танких диелектричних слојева са Керовим механизмом нелинеарности, могуће реализације оптичког уређаја са прагом, који се аутоматски под дејством упадне светлости пребацује из стања тоталне рефлексије у стање потпуне транспаренције.

Према [7], следи вредност интензитета на прагу, зависно услова, за дебљину слоја  $h/\lambda$ , смањује се. Поставља се питање како се при заданим индексима преламања слоја  $n_0$  и околне средине  $n_1$  и



Слика.1 Форме Гаусовог импулса при рефлексији од нелинеарног прозачног слоја за  $\alpha = \alpha_0 + 0,5^\circ$ , [7].

параметра нелинеарности  $\epsilon_2$ , изабрати дебљину нелинеарног слоја тако да тотално пропушта интензитет  $I_0$  при хистерезисном скоку из тоталне у парцијалну рефлексију. У пракси не постоји идеалан ра ванско-монохроматски талас; сноп је коначне дивергенције, па  $R \neq 0$  у минимуму. Дебљина слоја  $h/\lambda$  мора да се узима са дивергенцијом  $a$ , за упад под  $\alpha > \alpha_0$ , дивергенција мора бити мања или једнака полуширини  $\Delta\delta$  процена у  $R_{\min}$ . Нису добијена тачна решења граничног задатка за снопове у случају нелинеарног слоја. За приближне оцене, узимањем у обзир дивергенције упадног зрачења,

$$\alpha_0 \Delta\delta_N = \frac{\sqrt{gN^2}}{(n_1 h / \lambda) \cos \alpha_0 \sin 2\alpha_0} \quad (3)$$

$g = R_{sl} / (1 - R_{sl})$ . Ако је сноп доста широк, интензитети по пресеку top hat типа, а оса снопа се поклапа са једним од минимум коефицијента рефлексије  $R_{sl}$ , средњи коефицијент рефлексије снопа је

$$\langle R_{sl} \rangle \sim (1 - \arctan \beta) / \beta, \quad (4)$$

$\beta = g^{1/2} \gamma_0 / \Delta\delta_N$  и  $\gamma_0$  дивергенције. За  $\gamma_0$  на нивоу  $R_{sl} = 0,5$ ,  $g = 1$ , нумеричка вредност је полуширина,  $R_{sl} = 0,21$ . Реално ће бити 0 са  $\gamma_0$  са  $h/\lambda$  по [7], када ће бити тотална транспаренција. Угао упада  $\alpha \leq \alpha_1$  одређен хистерезисним скоком,



Таб. 2. Вредности за  $\Delta\alpha$ ,  $h/\lambda$  и  $\delta$  при фиксираним  $\Delta\varepsilon=5 \cdot 10^{-4}$  ( $\alpha_0=89^{\circ}9'$ ) (део таблице из [7]).

$\tau_0$	$\Delta\alpha, \text{min}$	$\delta$	$h/\lambda$
$1,5 \cdot 10^{-4}$	-9	-0,89	39,1
$1,5 \cdot 10^{-3}$	-44	-1,03	13,5

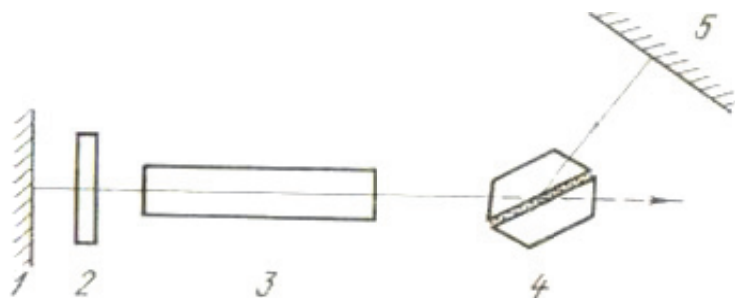
Таб. 3 Вредности  $I_0$  и  $h/\lambda$  при разним  $\gamma_0$ 

$\gamma_0, \text{min}$	2	5	7	10	12	15
$h/\lambda$	5,8	4,2	3,7	3,3	3,1	2,0
$h/\lambda$	8,7	6,4	5,1	5,1	4,8	4,5
$I_0, \text{GW/cm}^2$	1,9	6,5	10,3	16,5	21,1	28,3
$I_0, \text{GW/cm}^2$	1,2	4	6,3	10,1	12,8	17,3

$I_0(q)$  [11] за овај интензитет мора да буде истовремено интензитет прекида нелинеарног ПВО режима трансмисије. У Таб. 3 су оцене за разне угловне дивергенције  $\gamma_0$  упадног снопа,  $h/\lambda$  и  $I_0$  за  $N=1$  за случај  $\gamma_0=0,5$   $\Delta\delta_N(g=1)$  и  $n_0=1,600$ ,  $\varepsilon_2'=3 \cdot 10^{-8} \text{ cm}^2/\text{MW}$  ( $\text{CS}_2$ ) уз 2 различите вредности  $n_1$ , тј.  $\alpha_0$ . (Горње вредности  $h/\lambda$  и  $I_0$  су  $n_1=1,74$  ( $\alpha_0=66^{\circ}51'$ ), доње  $n_1=1,64$  ( $\alpha_0=77^{\circ}19'$ )).

Повећањем дивергенције  $\gamma_0$ , мора и  $h/\lambda$  да се смањује, а расте интензитет светлосних флукса, који скоро потпуно пропушта нелинеаран слој (кофицијенти рефлексије слоја у минимуму онда не прелазе 10%). Смањењем  $\varepsilon_1-\varepsilon_0$ , тј., са порастом  $\alpha_0$ , дефинишу се интензитети прага, (смањују се при заданој дивергенцији зрачења- $\gamma_0$  ако расте дебљина  $h/\lambda$ ).

Следи да танки прозачни слојеви са нелинеарношћу Керовог типа могу да обезбеде близу граничног угла  $\alpha_0$  тоталну рефлексију слабих интензитета зрачења и потпуно пропуштање снажних светлосних интензитета (преко стотине  $\text{MW/cm}^2$ ). Тако врше улогу филтра интензитета ласерског снопа. Аналоган је резултат при простирању снажног ласерског снопа у срединама са просторно периодичном структуром [9,7]; рефлексија танког слоја резонансне нелинеарне средине  $h/\lambda \ll 1$ . То се може користити за реализацију управљања ласерским снопом типа :нелинеарног оптичког прекидача, нелинеарног рефлектора, модулятора доброте ласерских резонатора, ограничавање снаге светлосног флукса и др. у ласерској техници. Користи се сличан уређај и као нелинеарни рефлектор - оптичког прекидача на путу простирања снопа. Код ласера са синхронизованим модовима може се добити генерација појединачних  $ps$  импулса, сл.2. Подешавање резонатора треба да буде такво, да формиран сноп пада на елемент 4 (тачније на нелинеаран слој између призама) под углом  $\alpha$ ,  $\alpha > \alpha_0$ . < При доласку било ког кратког импулса, нека критична густина зрачења се тотално рефлектује и може да наруши и сноп без губитака пролази кроз резонатор.



Слика 2 Оптичка шема ласера са нелинеарним рефлектором у резонатору 1, 5-глува огледала, 2- пасивни затвор, 3-активан елемент, 4-нелинеарни рефлектор.

Овом диспозицијом се може добити генерација хиперкратких импулса без пасивних модулатора добротом. (Огледало 5 треба да замени полупрозрачним, а на путу излазног снопа из резонатора поставља се глуво огледало. Улогу резонатора преузима филтар интензитета 4). Ако се обезбеди довољно висока густина зрачења у резонатору када се нарушава тотална рефлексија у нелинеарном филтру 4, формира се снажни врло кратки оптички импулс на излазу. Шема представља пасивну варијанту шеме (Weilstecke-a) за добијање хиперкратких импулса. Практична реализација на основу нелинеарног прозračног слоја уређаја типа филтра интензитета ласерског снопа је потребна, чак и за врло мале дивергенције ласерског снопа (реда неколико угл. мин.) концентрација у светлосном флуксу гигантских снага (реда десетине  $\text{GW}/\text{cm}^2$ ). Ти снопови су одавно достижни.

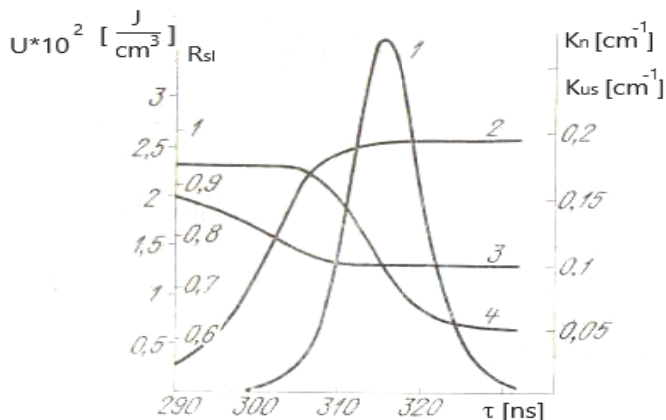
Појачање светлосних губитака је на сл. 3. и  $R_{sl}$ . Моноимпулсно зрачење, карактерише се густим зрачењем у максимуму  $U_{\max}$  енергије  $E$  и дужином  $\tau$ , на нивоу  $U_{\max}/2$ . Енергија на излазу из резонатора по [11a] се дефинише са (5)

$$\mathcal{E} = \frac{VU_{\max}}{2l} (v\tau) \ln \left( \frac{1}{R_1} \right) \quad (5)$$

$V$  запремина активног елемента,  $\tau \leq 10$  ns,  $E=4,3$  J (дијаметар активног материјала. До тренутка на којима је коефицијент губитака практично добијен – коефицијент појачања радног материјала се мало мења и остаје близак почетној вредности. Количина енергије, складиштена у  $V$ , може да се појави и као индуковано зрачење, пропорционално почетном коефицијенту појачања. У тренутку добијања максимума густине енергије снопа, коефицијент рефлексије апсорпционог слоја је  $\approx 1$  и у отвореном стању, модулатор овде практично не уноси допунске губитке. Ове битне околности приближавају се ласеру са модулатором на основу апсорпционог слоја као ласеру са тренутним

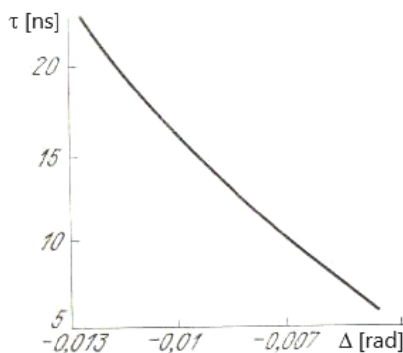
укључењем доброте. Реалне оцене  $U_{\max}$  и дужине single импулса  $\tau$  су [11b] за ласере са тренутним укључењем губитака блиске вредности ових параметара.

Важну је околност анализираних модулација и да се променом угла упада зрачења на слој може континуално променити коефицијент рефлексије у широким границама и варирати величина почетних губитака резонатора. Тако се управља енергијом и дужином импулса генерације, сл.4.



Сл. 3. *Измена густине зрачења  $U(1)$ , коефицијенти рефлексије апсорционог слоја  $R_{sl}'$  (2) коефицијента губитака  $K_n(3)$  и коефицијента појачања  $K_{us}$  (4) у резонатору ласера у времену ( $\tau \Delta = \alpha_{N-1} - \alpha$ .)*

Резултат повећања реалне вредности  $R$ , тј. повећање модула  $\Delta$  је веће вредности  $\tau$  и мање вредности енергије моноимпулса  $E$ . При измени  $R$  од 0,29 до 0,76  $E$ , смањује се 3 пута.



Сл. 4. *Дужина импулса  $\tau$  генерације ласера са апсорционим слојем у резонатора и величина отступања  $\Delta = \alpha_{N-1} - \alpha$  угла упада  $\alpha$  на слој.*

При избору могућих радних средина за модулатор на бази термалног слоја, важна је „дебљина” слоја  $h/\lambda$  и коефицијент рефлексије  $K$  на параметер

генерисаног импулса. Улога  $h/\lambda$  је разматрана у вези са применама као филтар. Повећање  $h/\lambda$  доводи до скраћења уз измене брзине  $R_{sl}$  са изменом индекса преламања слоја [7]. Пораст  $h/\lambda$  теоријски због постојања апсорпције расте и вредност  $R_{min\ sl}$ . Повећање се врши истовремено са смањењем угловних минимума  $R_{sl}$  и за вредности  $h/\lambda$  када ширина минимума постаје истог реда величине као  $\gamma_0$  дивергенцијом формирањем у резона тору и упадном слоју зрачења минимално  $R_{sl}$  одређено величином  $\gamma_0$ . Ова два фактора доводе до смањења могућег препада губитака у резонатору и ка порасту дужине моноимпулса  $\tau$ . За мале дебљине слоја, реализоване  $\Delta n$  су недовољне да се преведе слој из стања са минималном рефлексијом у стање тоталне рефлексије  $R_{sl} \approx 1$ , што опет води до пораст дужине импулса

О ширини спектра зрачења оптичког квантног генератора, квантно-механички историјски прилаз и сабирање линија

При анализирању рада квантних генератора, ширина линија је била једна од најважнијих величина, која је у спектроскопији на разне начине играла улогу у обради података из астрономије или физике материјала. После првих радова на масеру и експерименталне потврде сужавања спектра при генерацији су били предмет истраживања почев од друге половине 20-ог века. Извођење ширине линија неklasичним путем је полазило од Voigt-ове линије, која је у граничним случајевима постајала Lorentz-ијан или Gauss-ијан. Формализми Fourier-ове анализе укључени у ширине линија су доводили до разних апроксимација, што је даље водило до закључка да је ширина више различитих емитера једнака директној адицији ширина линија појединог емитера. Ови резултати могу да буду дискутабилни, али могу да служе као оцена за разликовање процеса у којима су чланови ансамбла у хаотичној или некој дефинисаној динамици. Квантно механички третман и примена једначине динамике зрачења са двонивоским системом су коришћени за израчунавање ширине спектра, а затим је следило аналогно извођење у генератору на рубину [10-11,12,13,14]. Показано је да се спектар зрачења сужава до границе, која зависи од величине рупре и ширине спектра спонтаног зрачења. Почетак извођења је био задатак о интеракцији система од  $N_0$  двонивооских атома са пољем зрачења унутар резонатора са идеално рефлексивним зидовима. Хамилтонијан је (2,3) из штампе од пре више од пола века [12]

$$H = \sum_{j=1}^{N_0} \frac{\hbar\omega_0}{2} \sigma_z^j + \sum_{k\lambda} \hbar\omega_k a_{k\lambda}^+ a_{k\lambda} + \sum_{k\lambda j} \hbar [B_{k\lambda j} a_{k\lambda} \sigma_+^j + B_{k\lambda j}^* a_{k\lambda}^+ \sigma_-^j],$$

$$B_{k\lambda j} = \sqrt{2\pi/\hbar\omega_k V} (M^j e_k^\lambda) e^{ikx_j}. \quad (6)$$

где је садржан сопствени оператор енергије атома, слободног квантног поља зрачења и интеракција атома са пољем зрачења, величине  $a_{k\lambda}$  и  $a_{k\lambda}^+$ , су оператори апсорпције и рађања фотона импулса  $k$  и поларизације  $e_k^\lambda$ , а  $M^j$ ,  $M^{j*}$  – матрични елементи прелаза за атоме координата  $x_j$ . Оператори  $\sigma_z^j$  и  $\sigma_{+,-}^j$  одговарају односима комулације. Затим следи разлагање поља по раванским таласима, резонатор се претпоставља у форми паралелопипеда запремине  $V$ . Затим следе формализам диференцирања оператора и једначине за средње квантомеханичке вредности. У обзир се узима  $n_{k\lambda}$  средњи број фотона са импулсом  $\hbar k$  и поларизацијом  $e_k^\lambda$  у запремини  $V$ . Претпоставком да постоје слабе везе међу атомима уз посредно поља зрачења, посматрају се само реални прелази међу стањима слободних честица у пољу у току целог процеса рада генератора. Уводи се релаксациони параметри  $\gamma_1$ ,  $\gamma_2$ ,  $W_0$ , који описују губитке фотона у резонатору, осветљавање кристала спољњим зрачењем / пумпом, која проузрокује позитивну разлику насељености  $N$  и проширење горњег нивоа под утицајем вибрација кристалне решетке. Користи се вероватноћа за спонтане и стимулисане прелазе:  $\tau_0^{-2} = 2\pi c^3 W / \omega_0^2 V$  –  $W$ -вероватноћа спонтаног зрачења. У првим извођењима, појављивале су се грешке и замењивале су се величине, а мењала се и терминологија. Ако је на почетку процеса са  $N$  атома у горњем нивоу  $N_2^0$  и ако је у  $t=0$ , за случај,  $\gamma_2 t \gg 1$ , следи

$$\dot{n}_{k\lambda} \approx \frac{\gamma_2 N_2^0}{2} \frac{1}{2\tau_0^2 (\gamma_2^2 / 4) + (\omega_0 - \omega_k)^2} \quad (7)$$

Следи да се ради о Lorentzovoj форми ширине,  $\gamma_2$ . За случај генератора на рубину,  $\gamma_1 \sim 10^9 s^{-1}$ ,  $W_a \sim 10^3 - 10^4 s^{-1}$ ,  $\gamma_2 \sim 10^{11} s^{-1}$ . Како се у геометрији са планпаралелним огледалима поставља рубински кристал, ради се о појави са просторним углом  $\Omega$ , суме се замењују операцијама интеграла, што води до режима слободне генерације, релаксационих осцилација. За потребне услове добија се  $\eta_{stac}$

$$\eta_{stac}(\omega) = \frac{\Theta_{stac} \Omega}{2\pi\gamma_1\gamma_2\tau_0^2(\xi^2 + a_0)} \quad (8)$$

што се уз одређене апроксимације своди на

$$\eta_{stac}(\omega) = \frac{A_0}{(\gamma_2^2 / 4) + (\omega - \omega_0)^2} \quad (9)$$

$$A_0 = \frac{(1 + \gamma)\Omega\gamma_2}{16\pi\gamma_1\tau_0^2} \quad (10)$$

$$\gamma_0 = \frac{\gamma_2 \Omega}{4\pi(W_0/W_n - 1)} \quad (11)$$

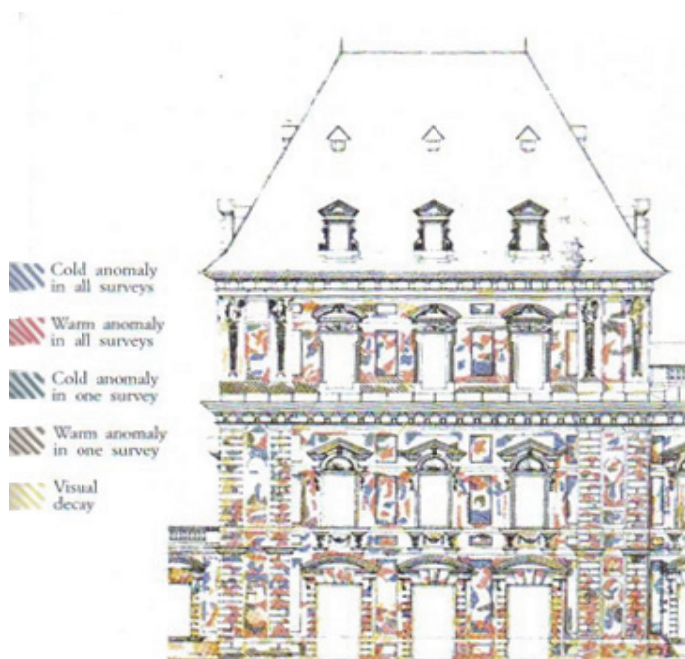
$\gamma_0$  представља ширину спектра зрачења у стационарном режиму. Опада са сужавањем простор ног угла и повећањем снаге пумпе. У резонаторима са сферним огледалима  $\Omega=10^{-3}$  rad. При  $\omega_0=2\omega_+$  постиже се  $\gamma_p=10^7$  s<sup>-1</sup>. Тако се на излазу у стационарном режиму фотонски спектар сужава, што је условљено нелинеарним карактером развитка фотонске лавине. Зато се релативно брзо умножавају фотони, са фреквенцијама близу центра линије  $\omega_0$ . Као закључак у раду [12] је описивано зрачење са прогресивним таласима; претпоставља се да су димензије резонатора врло велике. Тоталан број фотона у резонатору је дат зависношћу

$$\eta_{stac}(\omega) = \frac{Q_{0stac} \Omega}{2\pi^2 \gamma_1 \tau_0} \int_0^\infty \frac{d\xi}{\xi^2 + \gamma_1^2 \gamma_2^{-2}} = \frac{1+\gamma}{2\gamma_1 \tau} \left( \frac{W_0}{W_p} - 1 \right) = \eta_0 \quad (12)$$

То се поклапа са величином  $\eta_{stac}$  [12] уз помоћ кинетичких једначина. Поклапање важи и за прелазни режим. При вредностима пумпе блиских прагу генерације, јављају се неамортизоване вибрације интензитета зрачења, који се не описују са (4). За то је потребан строжији геометријски прилаз резонатору и активној средини [1-5]. У многобројној литератури ширина линије је третирана: а) као дефиниција облика, односно представника одређене динамике, б) као део процедуре у сређи вању експерименталних података, ц) као квантитативна интерпретација за много физичких величина: коефицијенти дифузије, коефицијенти топлотне проводности, апсорпциони коефицијенти средина, дијаметар центра расејања, компресибилност, или чак низ акустичких и механичких параметара, укључујући заостале напоне, ако се везује за помаке и Brillouin-ову и Raman-ову линију. У радовима смо се подсетили пакета програма, којима се може оценити димензија расејавача (постоје развијене теорије за разне класе и димензије у односу на извор квантни генератор који осветљава одређени ансамбл). Уз класичну ангуларну расподелу и корелације фотона из прошлога века, развијен је сна жан теоретски апарат уз професионалну техничку подршку, којима се разним шемама професи онално дефинишу ансамбли расејавача разних облика и димензија од макроскопских молекула, вируса до бактерија и других микро-организама. Старије примене за дефиницију полимера, мицела, колоида, итд. су биле добро развијене и пре корелације кроз избијање фотона, а класика добијања динамике у систему разних димензија почињале су од везивања са Brown-ијевим кретањем, углавном расподелом процеса расејања, укључивања поларизација, дифузије, полидисперзности, хомогеност и низотропију средине. Развој полупроводничких и фибер ласера је смањио габарите извора, ако се не мисли одмах на напајања нуклеарним и електронским сноповима [12,17-22].

## Примена инфрацрвене термографије за услове конзервације замка Валентино.

У рестаурацији замка Валентино, у првим фазама, вршена су осматрања са ИС камером, и оштећења су констатована термалним аномалијама. Квантитативна и квалитативна анализа је показана кроз неколико чињеница: одвајање гипсаних и других грађевинских материјала у конструкцији. Како се радило о вредној историјској грађевини, пажљиво је приступано испитивању. За квалитативно испитивање, примењена је Agema 488 LNJ термална камера са сочивом и слике су рађене са резолуцијом 140x140 pixel (пиксела), сл. 5. [13а].



Слика 5. Квалитативна анализа DIPRA графичка представа термалних аномалија [12]

Рађено је у разним временским условима: лети и зими, уз визуелну инспекцију уз интерпретацију кључних аномалија, Таб.4, сл.1, 2.

Таб. 4. Параметри за приказ кључних аномалија

Зона	Експозиција	Датум	Час	Темп.ваздуха, °С	Ур %	Време
DIPRA	Југозапад	28/5/97	9:33-11:24	17,4	НА	Јасно небо
Централни део	Северозапад	28/5/97	13:45-14:47	27,1	52	Јасно небо
DICAS	Северисток	28/5/97	15:23- 16:56	26,8	НА	Јасно небо

### Квантитативна анализа

Квалитативна анализа је давала целокупну слику термалних аномалија на фасади, али, није давала индикације о типу дефеката, који су је проузроковали. Зато је одлучено да се изврши квантитативан прилаз за следећи корак испитивања. 2 тест зоне су изабране у сагласности са низом специфичних критеријума-контрасна експозиција у односу на сунце, добро дефинисана површина. Познат одзив на *тос king тест* и бар за једну површину лаки прилаз - да би се извршило поређење квалитативно, квантитативно и посматраног стања.

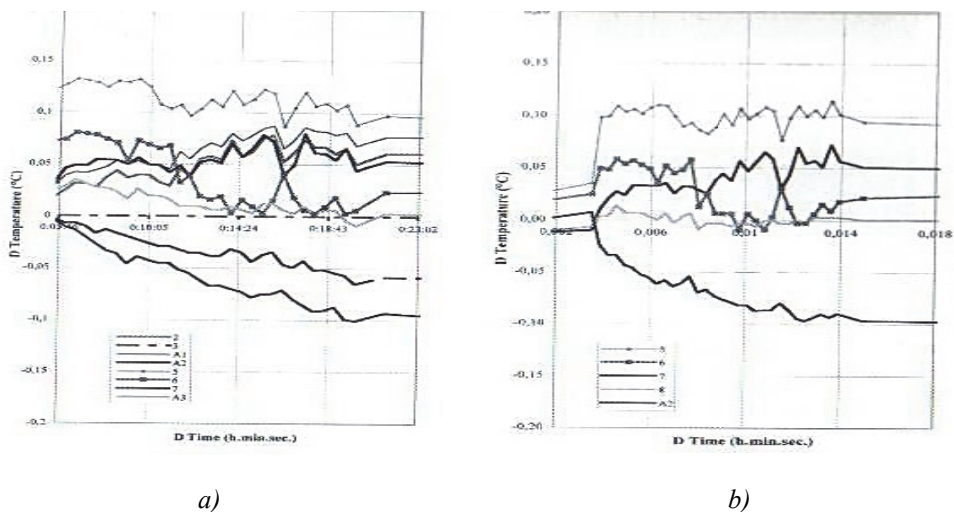
Док су један од иницијалних предмета били, тј. квантификација површине, која треба да се рестаурира, преглед је индицирао да је неопходна рестаурација целе површине, не само локализовано. Суперпозиција аномалија је учинила могућим да се елиминише шум сметња као ефект сенки у лето или грејање зими. Али није довело до значајних знакова. Зато је одлучено да се поново испитају све термалне слике да не би било без важних индиција – успешан резултат примене ове врсте методологије зависи од искуства оператора и оставља велику маргину непознатости.



Слика 6. Квалитативна анализа DICAS графичка представа термалних аномалија [12]

Квантитативно гледање је могуће да да детаљнију информацију о индивидуалним аномалијама / верификација закључка ове врсте анализе директна визуелна посматрање структуре. Треба пажљиво анализирати податке и стварање/користити банке података.





Слика 7. а) Тест 2. Температурно понашање у времену нормализовано са површином 3, б) Температурно понашање нормализовано са спотом 9[12].

### Новије спектроскопије

Не тако нова, али ипак међу новијим техникама је ласерски индукована спектроскопија пробоја (laser-induced breakdown spectroscopy, LIBS), метод атомске емисионе спектроскопије (atomic emission spectroscopy, AES) који користи ласерски генерисану плазму као *вруће испаравање*, атомизацију и извор екситације. Плазма се формира фокусираним оптичким зрачењем, па метод има многа преимућства у односу на AES конвенционалну технику са електродама и калемовима, за формирање извора. Важно је да се узорак може на лицу места испитивати и даљински без припреме. У свом најосновнијем облику, LIBS мерења се врше формирањем ласерске плазме на или у узорку и сакупљањем и спектралном анализом светлости у плазми. Квалитативне и квантитативне анализе се врше монито рингом емисионих линија њиховог положаја и интензитета. Иако је LIBS стар више од 4 деценије, од 1980, интерес је концентрисан на формацију плазме. Од тада су аналитичке могућности знатно по расле, а развијено је неколико типова инструмената на његовој бази. Метод је стално привлачио интерес за широке примене, као резултат знатног технолошког развоја компонената. (ласера, спектрографа, детектора) у LIBS инструментима и одатле потреба да се ураде мерења под условима који нису могућа конвенционалним аналитичким техникама. Литература о LIBСу доказује да је овај метод са детекционом осетљивошћу за више елемената упоредив или прелази карактеристике других технологија и он је примењен за материјале из некадашње оплате ЦИК-а [13, 14].

### *ИЦ спектроскопија*

Инфрацрвени спектри настају у процесу емисије или апсорпције. Ако се кроз испитивану супстанцу пропушта електромагнетно зрачење у ИЦ опсегу у његовом спектру ће због апсорпције недостајати фотони оних енергија које одговарају енергијама прелаза у молекулима и резултат ће бити апсорпциони спектри. У овом подручју молекули треба да поседују стални диполни моменат (наелектрисање асиметрично распоређено. Неполарни молекули ( $X_2$ ,  $H_2$ ,  $O_2$  итд) немају апсорпционе спектре. Код вишеатомских молекула дешава се да нека од њихових основних вибрација изазива апсорпцију (активна је), а нека не (неактивна је), [15].

ИЦ имају одавно вишеструку примену у многим областима. У физици и хемији дају податке о структури молекула, структури течности и чврстих тела. Омогућавају да сазнамо састав супстанце кроз коју је прошло ИЦ зрачење. Различите групе атома у молекулима показују у одређеним областима таласних бројева у спектрима апсорпционе траке, без обзира на састав осталог дела молекула. На основу тога закључујемо да нису позната два различита једињења са потпуно идентичним ИЦ спектрима. Када се зна, од којих је функционалних група састављено дато једињење, поређење са познатим спектрима једињења те класе даје тражени одговор. За поређење спектра могу се користити веома опсежне картотеке спектра, базе спектра сопствене лабораторије у сопственој лабораторији или бази података на њеб мрежама. Класичнији приступ је постојао и без рачунара, али данас је уз директну мерну тезнику омогућено директно или касније интерпретација спектра. Приступ је коришћење РС рачунара у чијим се меморијама налазе подаци о разним једињењима или базе података различитих класа једињења.

## 2. ЕКСПЕРИМЕНТ И РЕЗУЛТАТИ

Рестаурација Централног института за конзервацију (ЦИК) у Београду је трајала неколико година [13-16]. Зграда ЦИК-а, из чије фасаде потичу узорци материјала типа малтера, има два излаза – на улицу и на двориште. Изглед фасада је приказан на сл. 1 а и б. Дворишна фасада је са пропалом оплатором. Улична фасада зграде, за Балканску улицу је често одредиште за овај споменик културе (ЦИК, Балканска 7). Формална адреса је везана за Теразије 26. Детаљи на сликама су из доба пре рестаурације на основу чијег стања је и потекао читав пројекат [13b-16]. Делатност Института готово све време током рестаурације није прекидана и обављане су све врсте делатности. У овом раду је у првом плану дато неколико резултата из предвиђене методологије везане за праћење материјала из кога је грађевина састављена.



a)

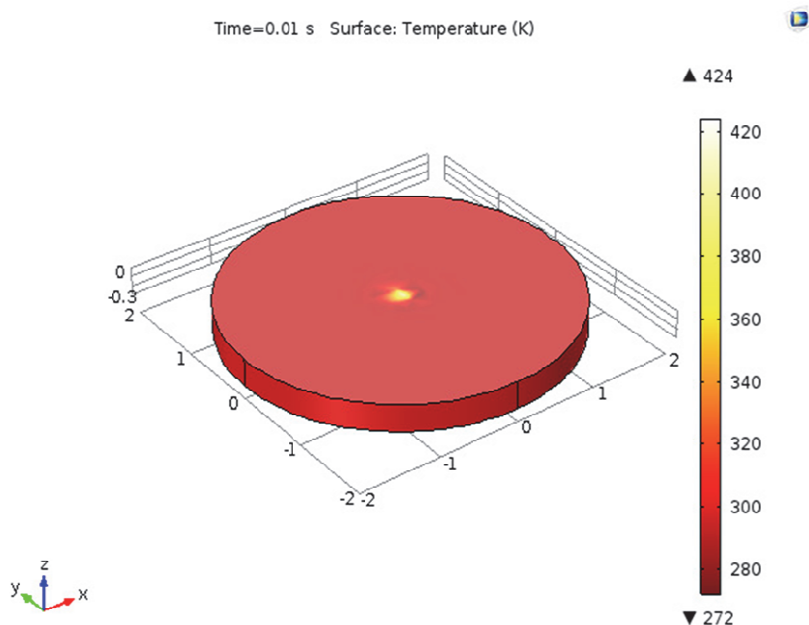


б)

*Слика 8. Фасада зграде ЦИКа – Централни институт за конзервацију а) Балканска улица, б) Дворишна фасада зграде [13,14,15,16]*

## Симулације

Симулација за интеракцију ласерских снопова за изабрани режим рада ласера и узете узорке оплате са зграде ЦИК-а (сл. 8 а, б) је урађена по термалном моделу, и то само у границама, где се оцењује температура на површини узорка при изабраним параметрима. То је део проблематике у којој је касније дато другачије, микроскопско стање добијених повреда, са изабраним режимима рада ласера. Претпоставка је да се ради о материјалу типа малтера; за почетак симулације, узети су параметри за један тип малтера по литератури доступној на Интернету и базама софтвера Comsol, према једној од верзија [13б, 14, 16, 30].



Слика 9. Симулација температурног поља за претпостављеног режима рада ласера са параметрима, од којих се овде сато даје снага – 10 W, од интереса за оплате ЦИК-а.

Параметри за оцену дистрибуције температуре су:  $\text{Nd}^{3+}$ : YAG, репетиција 30 Hz, снага 10 W са Гаусовом (Gauss) расподелом и спотом димензије 0,3 mm. За густину је усвојено  $1000 \text{ kg/m}^3$ , термодинамички подаци специфичних топлота и проводности су, према софтверској бази Comsol верзија 5.2,  $C_p=1800 \text{ J/kgK}$  и  $1,4\text{-}3,6 \text{ W/(m}\cdot\text{K)}$ , редом. Искоришћена је зависност промене проводности са променом температуре. На сл. 2 су усвојени ти параметри и оцењена је температурна дистрибуција у 0,01 s при интеракцији ласера са малтером. Симулација је спроведена и представљена на сл. 9.

### *Методологија анализе материјала оплате зграде ЦИК-а*

Из оплате зидова узето је неколико узорака неправилног облика, који су били изложени атмосферским утицајима дуги низ година. Димензије наших узорака су били реда величине 1-неколико cm. Са намером да се узорци не униште методом за испитивање садржаја, прво је коришћено неколико недеструктивних техника, какве су оптичке технике са ласером или оптичким извором. Овде су дате анализе везане за инфрацрвене спектре који се мере у рефлексивној или трансмисионој варијанти / модификацији инструмента. Друге алтернативе би биле LIBS, електронска микроскопија, хемијске анализе. Дати су спектри узети са узорака, који су излагани сноповима ласера произвођача Quanta systems са више прелаза:  $\text{Nd}^{3+}$ : YAG и полупроводничким ласерима о чему је дато више детаља у [26] раду и са кратким освртом на могућности одређивања.

Снимањем спектра узорака је добијена ИЦ природе везана за рефлексивне или трансмисионе спектре узорака, који одражавају вибрациона стања материјала. У истој серији су мерени и узорци спинела, ферита које смо претходно излагали ласерским сноповима и пратили морфологију и састав повреда. За овај рад важно је било имати прве потврде о молекуларним *сликата* израженим преко разних доприноса ИЦ спектрима. Спектри су дати за узорке малтера разних боја са зграде ЦИК-а, у ознаци С1 и К1, што има своје експерименталне параметре. У експерименталној фази су снимани парови спектра у кратеру (нова излагања) и неповређене околине, али је илустративно приказано само неколико представника разних материјала. Следећи корак је поређење резултата ове анализе и других типова спектра, затим отклањање *лажних* или *коинцидентних* стања.

Снимци микроповреда узорака са својим параметрима су дати у скупини микроповреда са истим ознакама која одговара местима на узорку. Овде се исто третира сав материјал биолошке / протетске / неорганске природе у погледу примењених техника мерења, а у погледу симулација са интеракцијом, избор модела (термални или други) зависно од типа примењеног ласера и сл. Постоје разни прилази, у зависности од постављених задатака за анализу и лабораторијску опремљеност и време *заузећа* хардверских ресурса, а зависи и избор третмана.

### *Микроскопска и макроскопска анализа материјала*

А) Узорци оплате типа малтера, који су узети за предмете дескрипције/дефиниције су, понавља се, узимани из оплате зграде ЦИК-а, сл. 1 (а,б). Узорци су неправилног облика и извршена је серија излагања узорака једном или два снопа истовремено (у исту тачку). Коришћени су ласери из лабораторија ЦИК- а. Узорци су неправилног облика (слика 10).

На сл. 8 су приказани оплата и оштећења ласерским сноповима; неке повреде су и резултат истовременог дејства, по габариту, малог полупровод-

ничког ласера (366 mW,  $\lambda=552$  nm,  $\Delta\lambda=0,1$  nm, неполаризовани сноп, MGL-S-532) и неког од снопова ласера на чврстом телу, професионалног уређаја за чишћење наслага на предметима културне баштине. Ласер лабораторије ЦИК-а је импулсног типа и има неколико прелаза / виших хармоника 1,06; 0,505 и 1,300  $\mu\text{m}$ . На макроскопским сликама узорака оплате или других материјала који су излагани ласерима (spinel), могу да се уоче повреде направљене ласерским сноповима. Дате повреде су даље анализирани оптичким микроскопом и типа су кратера ,слика 13. (Димензије свих узорака имају габарите реда по неколико cm, што је једна од претпоставки за симулацију).

Б) Узорци типа Б су били лабораторијски производ друге лабораторије, и проблематике, сл. 11 [23- 25].



Слика 10. Оштећења проузрокована сноповима полупроводничког или  $\text{Nd}^{3+}$ : YAG ласера са хармоницима; само по један сноп од сваког ласера одвојено, или симултано у исту тачку



Слика 11. Оштећења на макроскопским узорцима делови cm по габариту и дебљини реда 2-3 mm, типича/ / лабораторија / /...

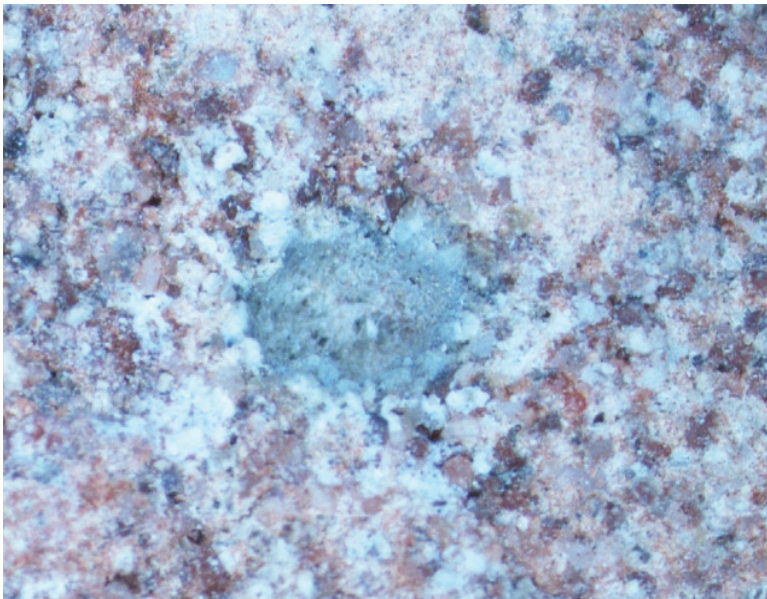
*Трећа група узорака*

Ц) Трећа група узорака (сл.12) су били *филтри* који се користе у телевизијске сврхе, чије је стање пропуштања/одбијања ИЦ зрачења контролисано пре много година у лабораторији (мр Владимир Красњук и мр Драган Попадић) која се бавила камером на телевизији. Контрола, у којој је као сарадник учествовао и Зоран Ристовски, студент Електротехничког факултета, је показала да не постоји свуда слагање са произвођачким спецификацијама.



*Слика 12. Оштећења на палети филтара за ТВ сврхе и контролу рада камере*

*Анализа оптичким микроскопом*



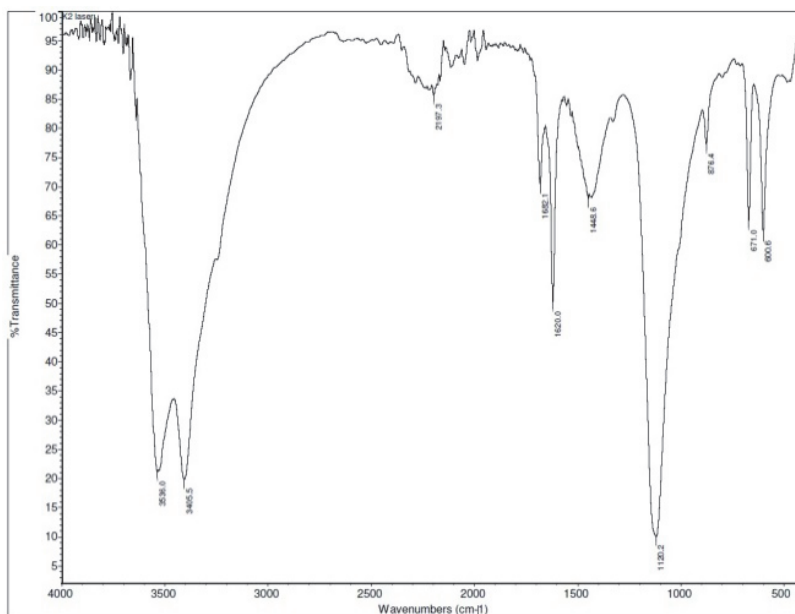
*Слика 13а. Микрограф анализе материјала узорака ЦИК-а, типа малтера*



Слика 13б) Микрограф анализе материјала узорака [13]

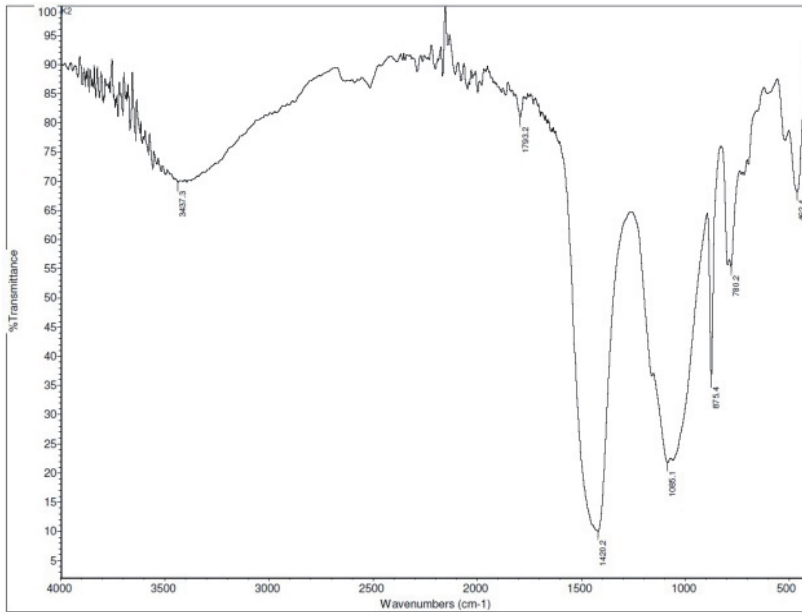
## ИНФРАЦРВЕНИ СПЕКТРИ

Снимање ИЦ спектра изабране категорије узорака је вршено на узорцима оплате и приказано на слици 14 и за материјале типа спинела на слици 15.



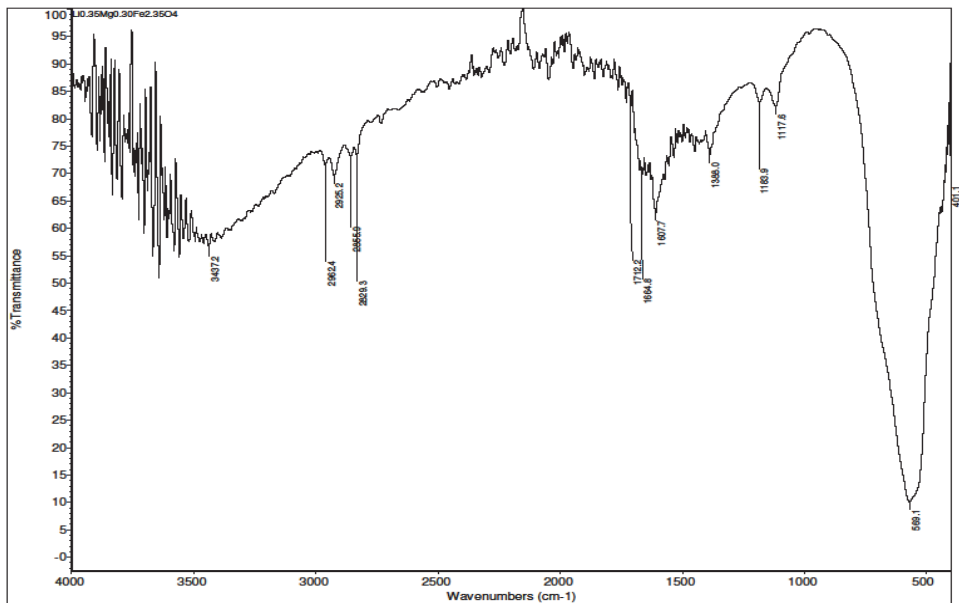
a)





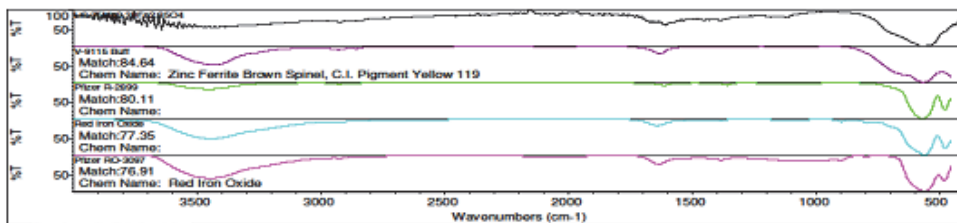
b)

Слика 14. a) ласер, у повреди (K2) ласером на оплати материјала узорка ЦИК [13],  
b) K2 без повреде материјал узорка. K2-ознака за радне услове



a)

Search results for: L10.35M0.30Fe2.35O4  
 Date: Thu Aug 20 11:49:41 2020 (GMT+02:00)  
 Search algorithm: Correlation  
 Regions searched: 3222.52-2466.55, 1834.00-551.55



Search results list of matches

Index	Match	Compound Name	Library Name	
1	1360	84.64	V-9115 Buff	Coatings Technology
2	1352	80.11	Pzizer R-2699	Coatings Technology
3	1280	77.35	Red Iron Oxide	Coatings Technology
4	1353	76.91	Pzizer RC-3097	Coatings Technology
5	1351	76.21	Pzizer R-1299	Coatings Technology
6	1362	75.00	V-9119 Tan	Coatings Technology
7	1361	75.77	V-9117 Deep Tan	Coatings Technology
8	1303	71.91	Versamag Magnesium Hydroxide	Coatings Technology
9	1354	70.41	Pzizer RC-5097	Coatings Technology
10	1350	69.77	Pzizer B-2093F	Coatings Technology

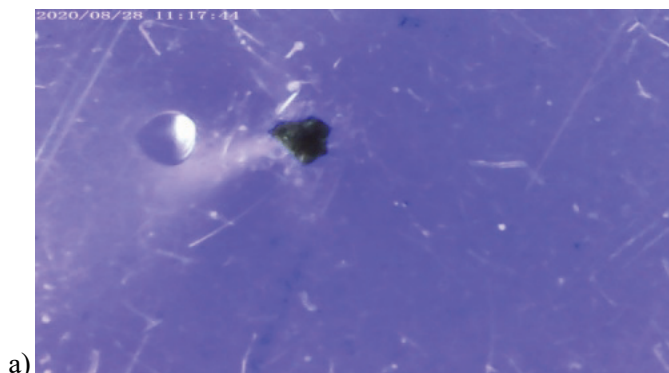
Слика 15 Инфрацрвени спектри материјала од интереса и за активне материјале ласера типа спинела некомерцијалног типа који су снимани и на другим системима ИЦ, а и карактерисани другим адекватним техникама

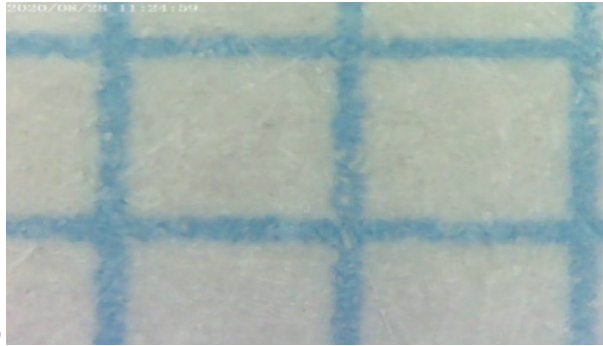
### Термографске анализе

Телевизијски зелени филтер из палете сл. 12 је излаган снопу полупроводничког ласера (0,4 W зелени прелаз ) у трајању 1 и 2 min. Зелени филтер PP1 (No. 78) је за време озрачавања праћен тер мокамером CIE Lab и белажене су температуре на месту спота на филтру предвиђеном за ТВ камере. Минималне температуре су зависиле од енергије /снаге ласера за повреде, сл.16 и представљене су на сл.17.

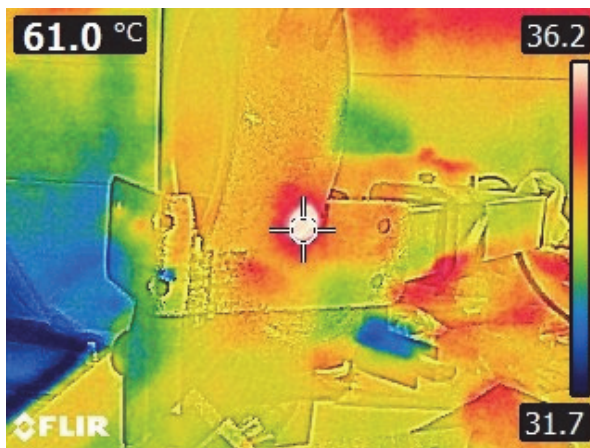
Стање површине места на филтеру изложеним снопу су анализирана системом микроскоп-рачунар. Стање повреде филтера је дато на сл.16 F1 и 16F2, развијене температуре у центру су биле 42,3 и 12,8 °C за излагање у трајању од 2 min, односно са FLIR 189 камером: filter plan Sp 1= 64,9 °C и 31,7 а за два пута мању снагу 52,9 и 32,0 °C, сл.17.

### Термографија





Слика 16 а) Филтар излаган полупроводничком ласеру (зелени прелаз један) б) Детаљ скале за анализе микроскопот сл.б

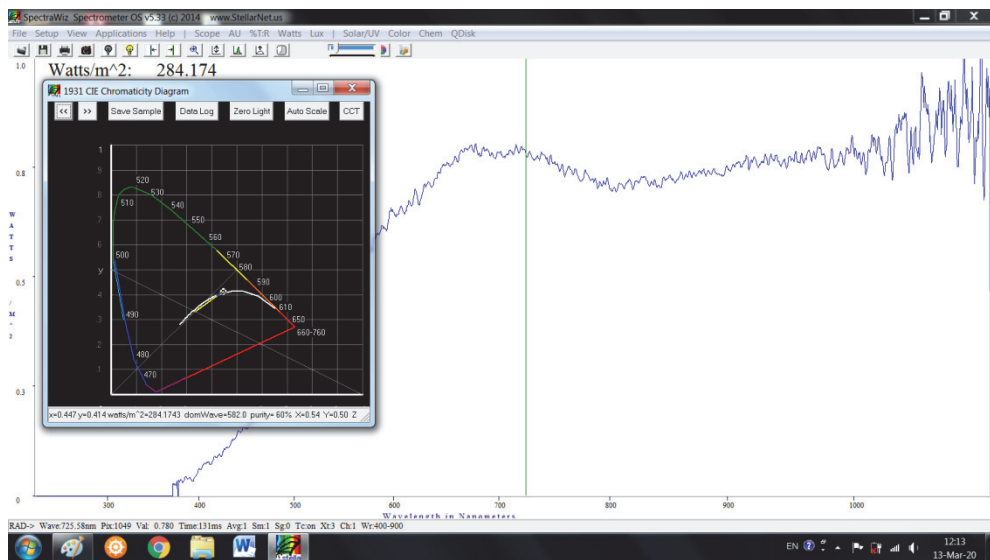


Слика 17. Тертографија неких излагања ласерским сноповима са временима реда минута.

### *Одређивање боје узорака*

При методологији тражења параметара за дескрипцију ласерских повреда, од интереса је промена боје узорака, дефинисана неким системом. У поступку калибрације полази се од дефиниције белог еталона и црног еталона и у односу на њих се одређују коефицијенти рефлексije и параметри према формализму инструмента спектрофотометра са Кг-лампом.

На сл. 18 В1 и В2 су детаљи одређивања спектара еталона који играју улогу апсолутно белог и апсолутно црног. У литератури постоји доста разних еталона за дефиницију црног и белог, па су подаци подложни анализи. За то су потешни спектри са другим еталонирањима и разлике параметара/спектара, јер немамо у целом мерном опсегу равну карактеристику, већ се мора вршити нормирање. При снимању је често коришћено растојање 4 mm од површине узорка при CIE Lab експериментима.



Слика 18. Одређивање боје узорка. Процедура за одређивање боје узорка преко координата и графичке представе података о спектру у датот подручју који представља рефлексију/ апсорпцију

У раду [26] се налази део података (слике спектра) помоћу којих се одређује боја узорка.

Сви приказани делови експеримената су од интереса и за културну баштину и за узорке материјала без обзира да ли су органског или неорганског типа, и могу се користити за стање површина, биофактора, са најразличитијим наменама, али припадају категорији квантитативних показатеља који се даље могу подвргнути симулацијама, пошто је овде у суштини увек потребан извор (овде је акценат на кохерентном зрачењу и ту би у свакој од метода могао да се укључи извор са кохерентним- ласерски, место извора са хаотичним зрачењем, што би потенцијално требало да допринесе нивоу прецизности [27-30].

### 3. ЗАКЉУЧАК

За закључак може да се узме у обзир неколико теза:

- разматрање формализма рефлексије на границама средина са интерференционим процесима;
- подсећање на прва извођења рада ласера, уз оцене за рубински ласер и једно извођење лоренцијана којим се често моделују ласерски снопови;
- разматрање неких корака у добијању филтера за велике интензитете и транспаренцију;

- у експерименталном делу рада, показивање оштећења на филтерима добијена много слабијим сноповима;
- извођење методологије за разне интеракције са ласерским снопом;
- за случај објекта културне баштине, односно њене оплате, снимање ИЦ спектра после LIBS који не уништава узорак. Приказани су микрографи микроскопских анализа који могу да послуже за детаљнију анализу и моделовање.

Микроскопске слике објеката као и део експеримената са термалном сликом интеракције је приказан, али стоји да би идеално било да се у сваком експерименту интеракције истовремено добије и запише податак о некој презентацији (брзе камере, ИЦ камере) а да се повреде свакако третирају електронском микроскопијом и EDX техником. За претпостављени материјал оплате, претпостављена је температурна расподела. Свим узорцима је одређивана и боја у једном систему везаном за ИЦ спекталну рефлексију (апсорпцију).

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## LASER TECHNIQUES AND COUPLINGS WITH OTHER TECHNIQUES IN CONTEMPORARY PROBLEM SOLVING IN THEORY AND PRACTICE

**Abstract:** Problems of today, *pro et contra* of new breakthroughs in high frequencies and pandemics, seem to have called for even more complex correlations and couplings of the fields: science, empiria (practice), mass-media in mastering the best possible description of certain processes in joint efforts to get out of a situation where our Globe had come to. More than ever, a lot of multidisciplinary work is needed, but also narrowly specialized sophisticated knowledge of the fields in which research is conducted.

In broad and generalized approach of coherent radiation interaction with material, the boundary of more classical scattering / interaction approaches seems to be lost, because the notion of stimulated processes and short-term pulses opened close areas in interaction with material (in a broad sense including bio-phenomena) and smeared the moment when living cells began to participate in optical recordings, and it is known that statistics from biological processes may solve traffic problems, etc.

In this paper, the interaction of materials with stimulated electromagnetic radiation, its results and the most accurate description of possible processes will be considered. By emphasising the optically visible, infrared and ultraviolet areas, some implementations of the rest of the electromagnetic spectrum and modulation of materials or their measurement possibilities will be sketched.

For selected examples of materials of various classes, their possible description of characteristics before and after interaction from the point of view of laser, thermal imaging, and other techniques will be considered, where records of induced acoustic or series of other coupled effects, like magneto-optical, electro-optical, etc. come to the fore. Selected theoretically more complex models with computer support will also be considered, as well as simple final formulas, which lead to relatively useful estimates in the outcome of the interaction during monitoring a particular output channel. In the spectroscopic approach, parts of spectroscopies that have laser or stimulated sources included and in that way new or only improved spectroscopies opened, will be discussed.

In this regard, interpretations become very sensitive areas. The approach of the application of laser and non-laser techniques to materials in general (e.g. to bio-materials, or to heritage objects) and defining the samples and measurements on selected objects or materials, as well as practice, theory, reality in the fields of cultural heritage, medicine or specific areas which include damages, useful destructive processes or explosive processes in the use of laser techniques again raises the issue of laser damages and their definitions. Entering into the field of protection by the induction of laser breakdown or by the initiation of various processes, must have detailed knowledge of the main optical indicators / descriptors of the material, including the concepts of reflection coefficients, scattering, absorption, thermodynamic parameters, ignition point, particle size if the material is in the powder state and similar.

**Key words:** laser techniques, interaction, scattering, materials, description, visualization.



# PRIMENA LASERA U AUTOMOBILSKOJ INDUSTRIJI

## APPLICATION OF LASERS IN AUTOMOTIVE INDUSTRY

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*Uloga lasera i sprežanja sa solarnom energijom i problemima „tematike“ vozila je vrlo široka. U ovom radu su analizirani principi, savremeni razvoj specifičnih disciplina i posmatranja sa tehničko/ naučno/ inženjersko/ metroloških tačaka gledišta. Ova oblast je u širem smislu vezana za „istoriju“ naše planete ili, u užem, sredinu prošlog veka i zlatno doba kvantne elektronike. Neki od generalnih problema su rešavani sporo, ali sa druge strane, posmatranjem sadašnjeg stanja nekonvencionalnih napajanja vozila/ automobila i realizovanih komponenti, neke karakteristike su menjale konvencionalne pristupe realizovanju i kapacitansama u sistemima napajanja. Glavni cilj je da se analiziraju problemi koji bi rešavanjem povećali gradijente budućeg razvoja solarne energije i njene primene u automobilskoj industriji, putem multidisciplinarnih pristupa. Uz podsećanja na potrebe, razmotrena su sprežanja kroz teoretsko inženjerske pristupe u polju tehnologije, metrologije i generisanja snage/ energije, i transformacije u području razvoja, primene i rada lasera, automobila i solarne energije.*

**Ključne reči:** laser; Sunce; obnovljivi izvori energije; vozilo; tehnologija; metrologija; solarno pumpanje lasera; holografija; plazma

*Laser role and couplings with vehicles and solar power are numerous and in this paper we analyzed the principles, contemporary development of special areas and observation from the scientific/ engineering, or from the metrological point of view. This area is, in broader sense, connected to history of our planet, or since the midst of the previous century and golden age of quantum electronics. Some of the general problems have rather slow dynamics of solving, but on the other hand, considering contemporary state-of-the-art of unconventionally powered vehicles and realized components, some characteristics change conservative opinions on the realizable capacities. The main goal of this consideration is to point to the unity of problems, which might speed up the gradients of the developments in solar technology of automotive technology, by multi-disciplinary approach. Overall, we have considered both theoretical approaches and currently developed systems in the fields of technology, metrology and power production and transformation.*

**Key words:** laser; Sun; renewable energy sources; vehicle; technology; metrology; solar laser pumping; holography; plasma

### 1 Introduction

Main parts of electrical power system bring to mind the importance of electrical power, production, distribution, consumption and production capacities of Serbia and neighboring countries

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(BiH - Republic of Srpska, Montenegro, Romania), as well as Europe and other continents. There are issues of small/ large hydro-, thermo-, wind and geothermal power plants and systems (and others) concerning planned and realized capacities. Wind energy transformation, properties of energy, transmission/ distribution/ development of networks, distribution facilities, photographs, questions of consumers and areas, generation of local area diagrams and averaging of capacities are still contemporary topics. The question and the stance of education of necessary theoretical and practical knowledge, as well as the skills for education systems are of special importance. Detailed data regarding climate and weather, like wind charts, etc. should be constantly recorded, due to (debatable) changes of climate.

### 1.1 Sun and wind

Almost entire renewable energy and also fossil fuel energy originate from the Sun's energy. Sun as a star rotates with the speed of 30 km/s around its axis with period of 27 days at Sun's equator and of around 31 day at Sun's poles. In the core, temperatures are estimated at  $10^7$  K, pressure  $10^{14}$  Pa and density  $10^5$  kg/cm<sup>3</sup>. Here the thermonuclear fusion of hydrogen to helium takes place, generating vast quantity of energy which is transferred to the surface and from there transmitted into Space in the form of electromagnetic radiation.

Photosphere, chromosphere, corona (seen during eclipse) and heliosphere (reaching out towards Space) are areas which parameters are expressed with temperature -  $T$ , pressure -  $p$ , radiation/ solar constants, state of ionized particles and gases. The temperature at the photosphere is  $T = 5769$  K and radiating power 64MW/m. Sun attributes, like the change of irradiation power with the distance from the Sun to the Earth is decreasing and solar constant drops to  $\approx 2353$  W/m. The causes of wind formation could be found. One parallel is that laser motor was known although it did not work on laser with solar drive. Other parallels are lasers pumped by solar power.

For a long time, Germany was leading in wind power production capacity. In 2008 Germany had dropped to the second place and in 2010 to third, with the China taking the precedence. The development of wind energy in the first two decades of the 21<sup>st</sup> century and the factsheet on the wind power in Germany from various references are shown, Figs. 1-3. Consumption management in the smart power grids with variable production puts on line issues: electrical power, distributed sources, smart network, response, consumption, load, prognosis, management, peak power, variable production. Nowadays, economic and ecologic interests are often opposed and are the topics of everyday consideration and debate [1-22].

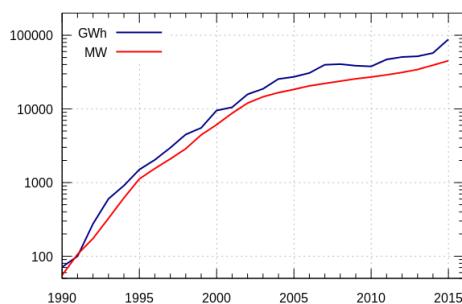


Figure 1. Wind power in Germany from 1990 to 2015 [2]

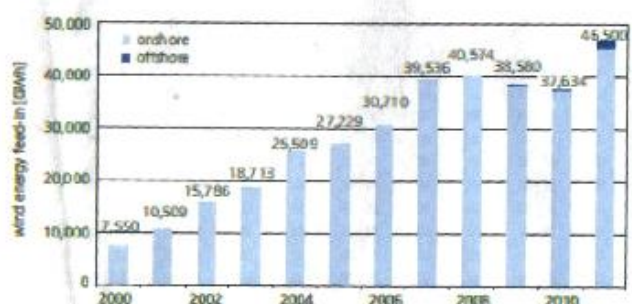


Figure 2. Development of wind energy supply in Germany

## 2 Technological and metrological application

The use of lasers in automotive/ vehicle industry, aircrafts and shipbuilding - maritime industry [23-27], for civil and military purposes is increasing. Historically, a series of references describing application of lasers in certain area of everyday life contain it as a topic. As devices, lasers were developed in scientific disciplines worldwide and used in advances to Space, medicine, in art and heritology as well [23-27]. Here, several examples are presented existing in generally all categories

covering material processing [27-34], a role in vehicle sensors, state of tires and similar, concluding with laser motors (engines) and laser rocket engines (motors) [35-54].

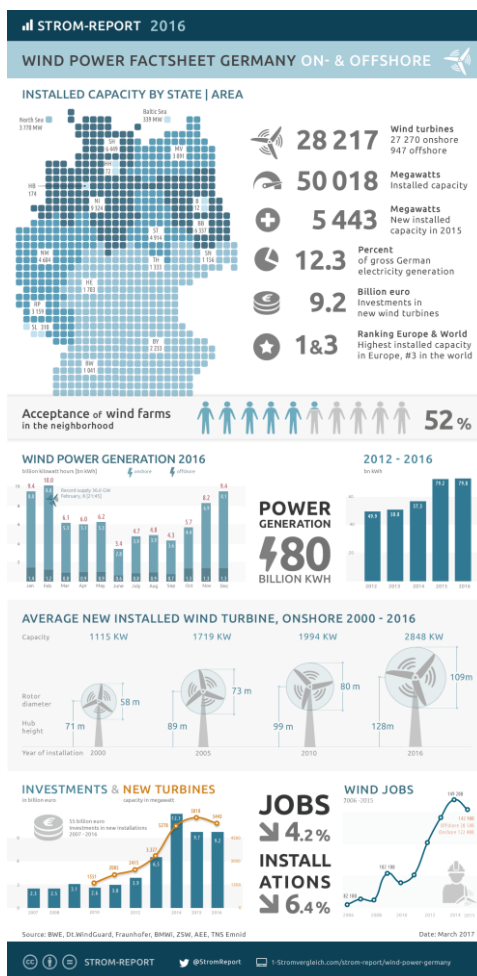


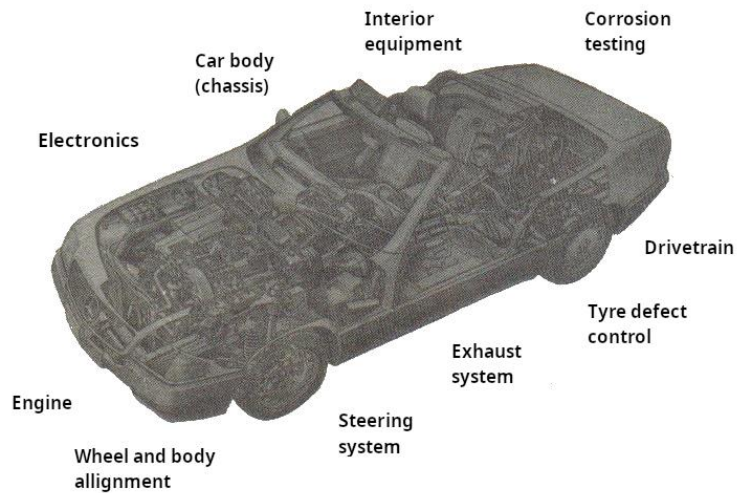
Figure 3. The factsheet on wind power in Germany in 2016 [1]

The application of lasers in automated visual inspection systems, quality management and in the measurement technique of vehicle production and control are among the measurements developed on the basis of coherent sources for non-destructive testing (NDT) of: fatigue, vibration, deformation, flow and combustion processes. The attributes are: simplicity, easy handling, ergonomic design of the car interior, methods of geometric and physical optics, profiles and surface positions, detection of defects and optical vibration measurement and monitoring.

To control oil purity and combustion processes, a number of spectroscopic methods are used. For other features linked to automotive industry and vehicle operating, laser based methods of frequent use are anemometric methods, interferometry, holography, tomography, however speckle based methods are also implemented. In many methods, quantum generators are widely used as sources. In the field of precision mechanics and analogous problems, Moiré methods were known as more classic. Some developments that are linked to all the methods mentioned above are: CCD batteries and the development of small computers including more *conservative classical methods*.

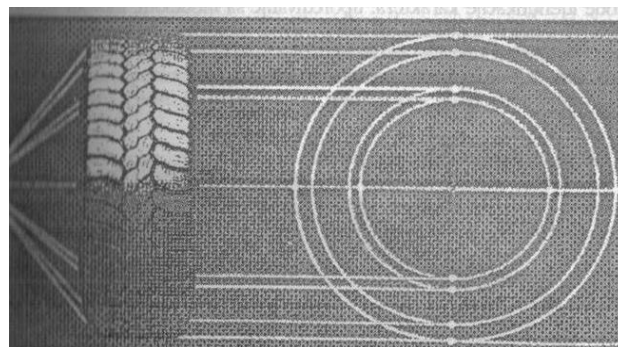
In *pure* processing of materials in automotive industry, laser role is included through the tasks of engraving, printing on metal parts, drilling, cutting, welding (machining/ processing of objects of various geometries), printing through glass or layers of other material, after the system has been assembled. Printing techniques are known in the methods of masks (in photolithography) and matrix or scaffolding in 3D printing and are connected to branching procedures and artificial intelligence. Monitoring by camera is also performed in the vehicle industry, especially in the automotive industry. The trend in general is to replace the human work with robotic or digitalized solutions. Once upon a time, acquiring an image of a work piece in good illumination was sufficient for further analysis, which excluded the necessity to observe or to have access to the work piece. Pro-

cessing speeds and solutions depend on the complexity of the part to be produced and the current technology. In perspective, the quality of acquired images improves with illumination by coherent sources. As an example, lasers were included in bar code technology much later after it was introduced. Much of the theoretical work has been invested and many technological operations were developed until laser centering of automotive parts became common in many car repair centers in *almost every town*. A large number of possible, potential or current laser applications are sketched in Fig. 4.



*Figure 4. Typical points of inspection and quality control in vehicles. Emphasis is on the measuring role of the quantum generator, but it was not taken into account that the laser methods also participated in the construction of a particular component or assembly*

Characteristic places of inspection and quality control in vehicles are steering wheel and measuring elements, engine, drive unit, electronics, exhaust gases, interior equipment, car body, centering of gears and wheels, control of tires, corrosion testing, measuring of vehicle speed and wheel rotation speed, topographic types of control, performed at normal speed of movement. For instance, poorly centered pneumatics cause vehicle vibrations and have serious impact on the car suspension and support system. The implementation of such systems, Figs. 4-8, has been developed by car and tires manufacturers, Michelin among the others.



*Figure 5. Control (monitoring) of tires with a laser beam*

Profile and surface quality examination, and the surface position measurement are also performed by laser. In contour measurements, a sharpened probe is used maintaining the mechanical contact with the surface of interest. Changing the position causes the probe to move, which further evaluates the contours of the surface profile. Mechanical devices in various areas verify the thickness of details and determine the surface parameters in the process of automatic control by using the servo systems and feedback by adequate comparison. For the exact reproduction of the 3D representation, the method of profilometry of the template is used.

Optical solutions in profilometry have the following advantages: no damage to the work surface, no wear of instruments, measurements can be performed with templates made of various mate-

rials. Developed models range from polished materials of various degrees of metal surface quality to plastic and matt surfaces. Accessories with wedge-shaped components and topography recording are increasing in numbers. Measurements provide a profile, horizontal map, object thickness, as in Fig. 9a.

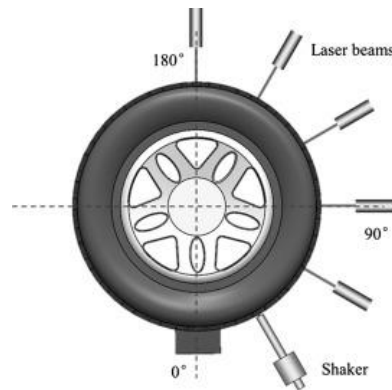


Figure 6. Control of a tyre with a laser beam [24]

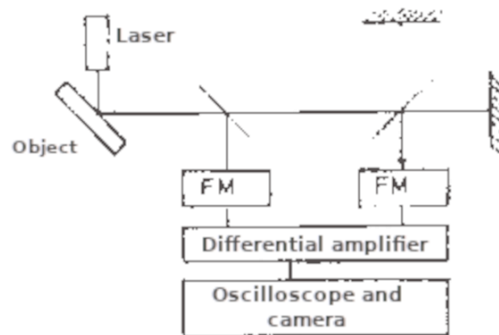


Figure 7. Displacement measurement scheme

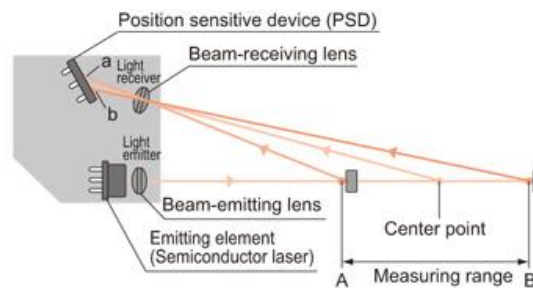


Figure 8. Laser displacement measurement [25]

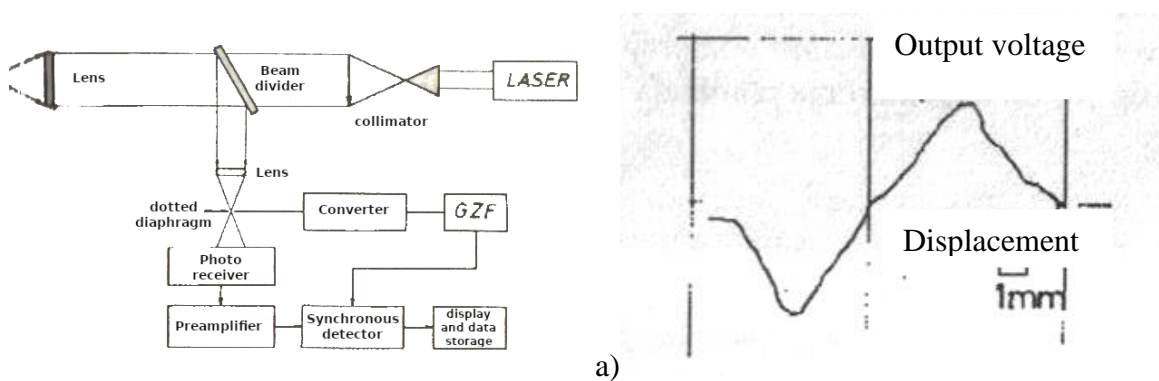


Figure 9. a) Block diagram for measuring the surface profile; b) synchronous detector output

In the surface profile measurement system, Fig. 9, the laser beam is focused at a small spot on the surface of the examined object. Scattered radiation is collected by lenses and focused through

the diaphragm vibrating at sound frequencies onto the photodetector. The variable component is amplified and detected by synchronous detection in relation to the frequencies of the generator. Diaphragm dimension is 250  $\mu\text{m}$ , and vibration frequency is 525 Hz. The sign of the output signal depends on the direction of movement in relation to the focus. This is how maps are recorded (2D analysis).

### 2.1 Systems for defect search and diagnostics

The advantages of laser quality inspection and control methods come to the fore when it is difficult to provide automatic control. There are various cases: surface quality, mass production, rapid control of multilayer materials, small details in the automotive industry. The system works on the principle of light scattering (laser beam). For cylindrical geometry, the He Ne laser beam is transformed into a line, which follows the workpiece with a mechanical manipulator of the auto-revolver type. Various optical signals are collected from the surface of the working part, transmitted through a system of fibers and other optical elements, and detected by a photomultiplier. The frequency spectrum of the obtained signals is compared with/ to the spectra obtained from the surfaces without defects.

### 2.2 Optical vibration measurements

The character of vibrations of one structural part during harmonic excitation is measured. Complex processes in engines, brakes, body parts and testing areas at several points can be analyzed. For instance, Volkswagen used the contactless methods for obtaining the data for analysis, holographic and laser Doppler measurement techniques. Among them, there are two-pulse holography, LDA measurements of vibrations in small volume (SOVAS technique); Tabs.1 and 2 give characteristics of laser methods during time.

Table 1. General methods of vibration measurement

	Real time holography	Pulse holography	SOVAS
Object	Single construction elements	Construction blocks, aggregates, chassis	Construction blocks, aggregates, movable parts of chassis
Measuring quantity	Amplitude	Analysis regarding laser pulse	Speed
Measuring interval	0.2 $\mu\text{m}$ - 0.5 $\mu\text{m}$	0.2 $\mu\text{m}$ - 200 $\mu\text{m}$ (extrapolated)	0.001 m/s - 1 m/s
Excitation	Harmonic	Harmonic or auto-excitation	Harmonic or auto-excitation and excitation of noises
	Frequency variation	Dominant frequency time filtering	Spectral analysis
State of vibrations	Forced	Transitional regime	Stationary regime
Mode of operation	Surface	Surface	30 dots $\times$ 30 dots
Duration of measurement	Several seconds	Instantly	10 s - 300 s

Table 2. Methods of holographic interferometry

	Real time holography	Pulse holography	Holographic interferometry with averaging in time
Operations	Hologram exposure, loading the object	Hologram exposure, loading the object, secondary exposition	Hologram exposure during object moving and development
Advantages	Total information on object changes	Easy realization, one-for-all single setup, constant emulsion	Easy realization and easy interpretation of results
Drawbacks	Difficulties in interpretation, necessity of repeating the setup, emulsion, distortions	Pure information, difficulties in interpretation	Not suitable on immobile surfaces

Applications	Analysis of mechanical stresses, defects	Analysis of mechanical stresses, defects, Analysis of transitional processes	Analysis of vibrations
--------------	------------------------------------------	------------------------------------------------------------------------------	------------------------

### 2.3 Active holography - real-time holography

With the help of a real-time holography, the vibrations caused by the construction elements are visualized. The vibration distributions are observed on a monitor with a video camera. Resonances are established by varying the excitation frequency. The measuring area relates to the area of the interference fringes  $0.1 \mu\text{m} - 5 \mu\text{m}$ . The interferometer must be on a special stand with insulation from vibrations of a mechanical nature. There is also the holographic working camera, the control of the illumination of the object, excitation and the stroboscopic device. For structural vibrations of the engine block, the range above 1 kHz is reached.

Another application with laser technique is related to the technique of optimization of brakes from the stress-strain point of view, Fig. 10. The problems of real-time holography are related to: a) placing the hologram in the exposure position, b) distorting the photoemulsions and c) detailed interpretation of the interference image in relation to the theory of local zones.

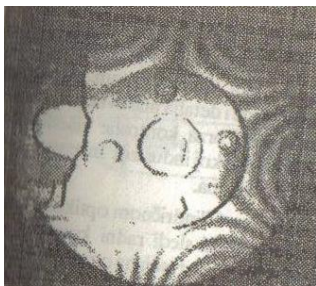


Figure 10. Vibration: technical optimization by holographic interferometry

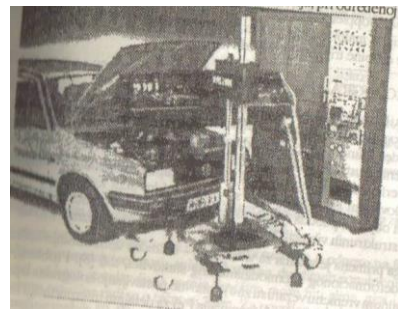


Figure 11. Holocamera

### 2.4 Two-pulse holography

Pulse holography has long been used outside of optical laboratories. Illumination with two pulses is used for the analysis of transient modes with a holocamera and adequate electronics, Figs. 7, 10, 11, which evaluate the vibrations (displacement/ dynamic) of the car body and the sub-systems. Free and forced vibrations are monitored. The resulting interference distribution showed the exact position and time (moment) of the vibration at a certain frequency. In addition, structural material weaknesses, noise sources and material strength are also examined. One of the first applications of holography in general, including this method, is based on the examination of pneumatics and defects in production and exploitation, Figs. 12, 13. Compared to the methods of real-time HRV holography, the method is less flexible and cannot monitor continuous deformation. The problems are in the interpretation, localization of zones, etc., Figs. 13, 14. The existence of both defects and places of different loads causes the occurrence of thickening and deformation.

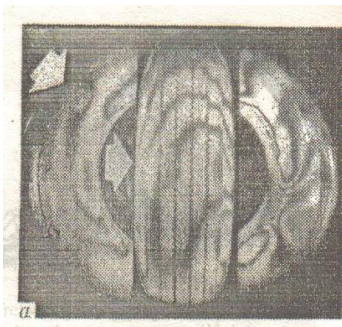


Figure 12. Holographic tests of tires; interference fringes



Figure 13. Detail of hologram for defect analysis

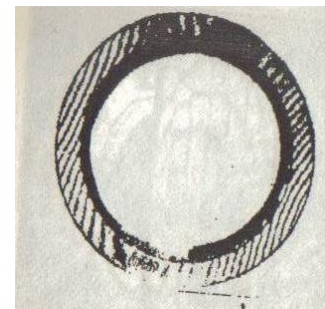


Figure 14. Changes on disk couplings examined by holographic techniques (mechanical and thermal stresses)

## 2.5 Laser Doppler methods: LDA and LDV

Holographic interferometry, HI, with time averaging does not demand that the detail, the construction ensemble and the scene are without vibrations of the environment (“without moving”), and that mechanical displacements are less than  $\lambda/4$ , where  $\lambda$  is the wavelength of the applied quantum generator. The characteristic of the beam intensity dependence on  $x$ , taken by one of the holographic schemes, is connected to  $D(x)$  – surface displacement from the equilibrium state (or undeformed state) – and to  $\lambda$ . The surface analysis and estimation of its dynamics is possible if the condition  $4\pi D/\lambda \gg 1$  is fulfilled.

In general, if the Doppler effect is present, the methods inherit its name. It is often used with the systems that follow both the development of techniques/ technology and the widening of our experience from acoustics to area where relativistic effects had to be taken into account for the case of EM radiation. HI methods are based on the optical path manipulation and on obtained visualization of hologram. The image is closely connected to vibrations, resonant frequencies, provoked deformations, and stresses of the objects or system. The vibrometer uses the direct and referent beams. Laser beam reflected from the object is recorded in the measuring system. The laser beam operates with defined number of measuring. In the case of vibrations, vibration processes are imprinted in signals. The obtained spectrum is the principal task for analysis and from the depth of modulation characteristic (resonant) frequencies and the local distribution of the vibrations can be obtained, Figs.15. Besides the first known application in violin resonator (or other musical instruments), and in turbine blades, HI methods with time averaging are applied in automotive, aircraft and other vehicle industries.

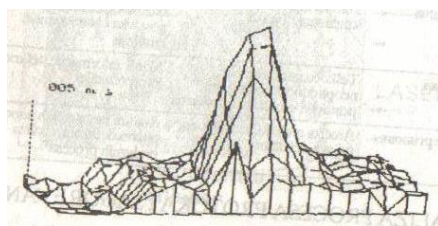


Figure 15a. SOVAS techniques define resonance frequencies of component, part of the system or in general recorded area (valve dynamics)

The analyses of the combustion processes have been performed by the methods of laser beam scattering and have been used for monitoring of engine operation, and for monitoring of turbulent processes. The Schlieren method is commonly used in this case, Fig. 15b.

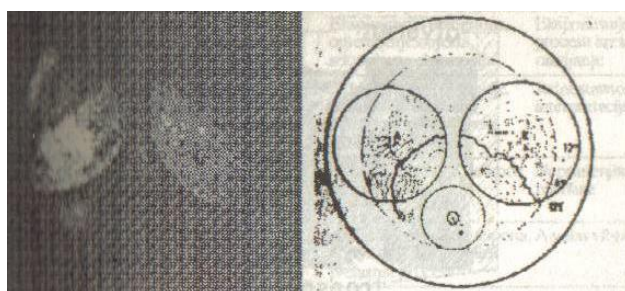


Figure 15b. Schlieren method applied in motor dynamic control/ monitoring

## 2.6 Laser Rocket Motors

New trends in the development of rocket techniques have opened new possibilities for lasers to be implemented and for solving problems related to classical motors used in interplanetary flights. In this paper, some aspects of the design and construction of laser rocket motors as well as the implementation of exchange of laser power are presented. A lot of debate concerning the types of optimal design of the systems of energy transformation in cosmic investigation still exists, and new technologies in recent years have opened up many new possibilities.

Laser rocket motors run on various energy types: electrical (electrostatic, electromagnetic and electrothermal), nuclear, solar, magneto-hydrodynamic and numerous types of chemical fuel, and



more. They all have many advantages and disadvantages, with different degrees of efficiency. Solar-powered laser motors also exist. Both solar into thermal energy transformation and motors (ionic or plasma) are considered for the cosmic travel. However, it seems that the ideas concerning classical – thermal – motors are still dominant in the field of contemporary solutions in the Earth’s atmosphere. The link between reactive motors and laser as a power source is still a topic of discussion as also is the grouping of laser power transmission into several groups based on the principle of operation, thermal, electric and hybrid.

One possible solution of laser implementation is where a directed, focused laser beam is absorbed in some solid, liquid or gas fuel, or for laser-ignited combustion, Fig. 16. The basic mechanisms of absorption in heat exchange devices are: Bremsstrahlung radiation, CW absorption, molecular absorption and particle-level absorption.

The implementation of pulsed, periodical laser beams, or CW laser beams depends on the combustion material type. The essential part of the design is linked to the current generator in the motor nozzle. In our terminology, the motor nozzle means establishing plasma in the working material, and combustion is the main process in sustaining the plasma.

The aerodynamic windows are especially important when working with high-power lasers. There exist non-linear optic solutions in surmounting window-related problems; these solutions are based on multi-layered materials and inverse-population processes. Optoelectronic systems are used for energy transport from Earth to orbital stations; the purpose of these systems is the transmission of CO<sub>2</sub> and free electron laser beams, Fig. 17.

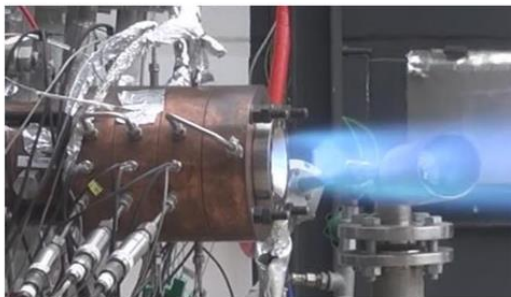


Figure 16. Laser-ignited combustion [50]

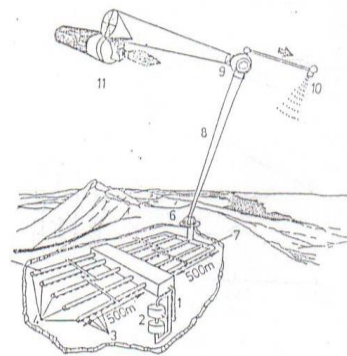


Figure 17. One of the first schemes of transmission of energy from free electron lasers on Earth to cosmic satellites. This scheme relies on the application of reflection and refraction processes

The station is built on high altitudes to minimize loss of power/ energy in the atmosphere. The principal parts of the system are: the free electron accelerator, powering sources, vacuum pumps, the transmitting mirror, the optical resonator, the collector/ concentrator of radiation; beam trajectory, geostationary orbital retranslator, etc. The feedback with the power station and the interorbital rocket with laser motor are also important aspects of the complex.

Laser rocket motors produce pressure based on the transformation of the beam energy into kinetic energy of fuel molecules via heating and expansion processes in the nozzle. The gases involved are of low molecular mass, and are therefore heated to very high temperatures. The principal tasks for the practical realization of motors of this type are: fulfilling the conditions for optimal absorption mechanisms of the laser beam, selection of the wavelength, the minimization of energy losses in all phases of motor’s functioning, the design of the motor, and even the choice of materials or fuel, Figs. 18, 19.

Characteristic values are linked to simple approach to 1D modeling of flow, cylindrical geometry of the nozzle of constant cross section. These are the first approximations related to the real absorption of the laser beam, when effects of detonation, laser plasma wave transmission, supersonic processes and deflagration are possible. For CO<sub>2</sub> laser beam wavelength and hydrogen, at 0.1 MPa, power threshold is 10<sup>10</sup> W/cm<sup>2</sup>.

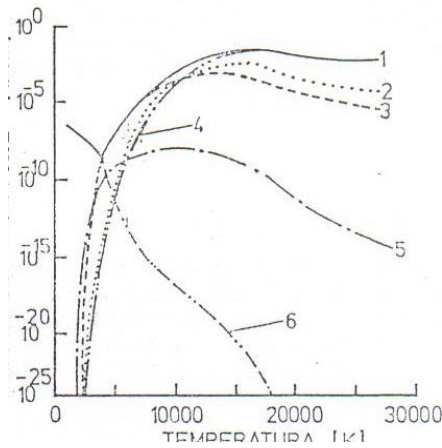


Figure 18. Absorption coefficient [1/cm] for radiation of  $2.2 \mu\text{m}$  wavelength versus temperature [K] for pure hydrogen at  $p = 0.1 \text{ Pa}$  and the contribution of various causes to absorption; 1 - additive coefficient, 2 - photo-ionization, 3 - electrons in atom, 4 - electrons in ion field, 5 - electrons in molecule field, vibration excitation of molecules

Considering laser plasma in 2D modeling of rocket engine, operation characteristics of laser thermal engine are presented in Fig. 19, and the influence of its losses is presented in Fig. 20.

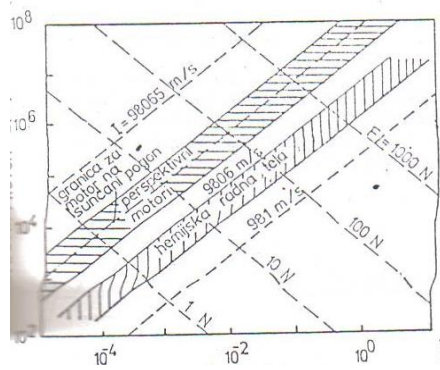


Figure 19. Operational characteristics of various ideal motors – fuel consumption [kg/s]

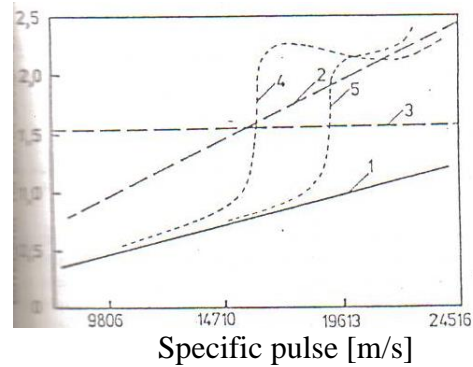


Figure 20. Influence of various losses to specific pulse: 1 - ideal characteristics, 2 - with atmospheric losses and partially influenced by operating body, 3 - losses by focusing/insufficient aligning, 4 - freezing for  $M = 1.5$ ,  $M = 2$

In Figs. 21-22, some characteristic results and facts from literature are presented.

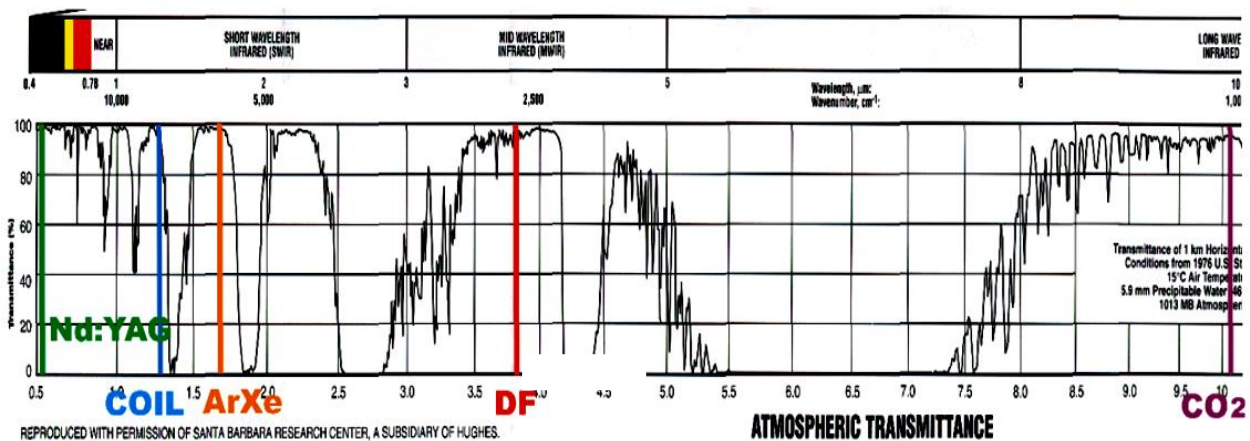


Figure 21a. Places of some high power lasers and atmospheric transmittance [54]

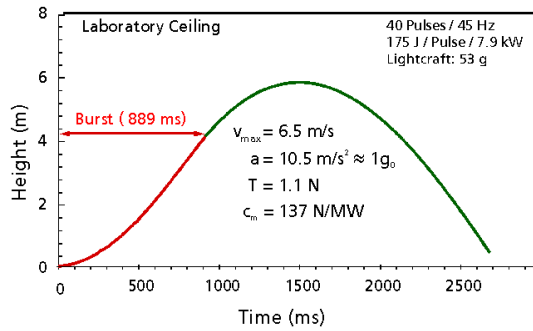


Figure 21b.



Figure 22. Gabarites in contemporary systems.

### 3 Laser Cladding on Exhaust Motor Valves

#### 3.1 Plasma and Laser Cladding on Stainless Steel for Exhaust Motor Valves

The exhaust motor valves are exposed to high temperature of hot combustion gasses, followed by the pressure at the contact surface with the valve seat. The valves for fossil fuel motors are manufactured from stainless steel materials, but many of which do not have satisfactory wear properties at high temperatures. The thermal stability can be improved by cladding. For that purposes, hard and corrosion resistant material should be used, such as Co- or Ni-based materials. Due to high boiling and melting points, the plasma spraying and laser cladding must be used for fast melting. Laser cladding is distinguished from plasma cladding in a smaller fusion area at the parent metal and in producing a well metallurgical bond of the hardfaced material. Furthermore, the combustion of vaporized liquid fuel produces specific kind of high-temperature corrosion. Many attempts have been done in choosing the material with adequate properties. The Ni alloys for motor engine valves must contain strengthening elements, commonly Co, Al, and Ti. Due to lower price, the Co coating alloys are superior to stainless steel as a surface material. The industrial acceptance of laser cladding is still slow.

**Powders.** Typical stainless steel for exhaust motor valve has contributions of five elements (percent): C (0.50), Cr (21.0), Mn (9.0), Ni (4) and N (0.4). After quenching from 1140 °C- 1180 °C in aqua, steel retains the austenitic structure; it means that hardness values are not increased. As corrosion resistant and hardfaced materials for exhaust motor valves, available are Ni-Cr-B-Si and Co-Cr-W alloys (Stellite 6 or similar) in powder form [27]. For melting of those alloys, the high energy welding methods should be used.

**Characteristics of cladding methods for engine valves.** The contact surface between the engine valve and its seat, assigned by an arrow in Fig. 23a, subjected to the pressure in the atmosphere of combustion gases, achieves a remarkable wear. In order to decrease the wear, surfaces should be cladded/ hardfaced, Fig. 23b. This surface is rough, Fig. 23c, and has to be grinded.

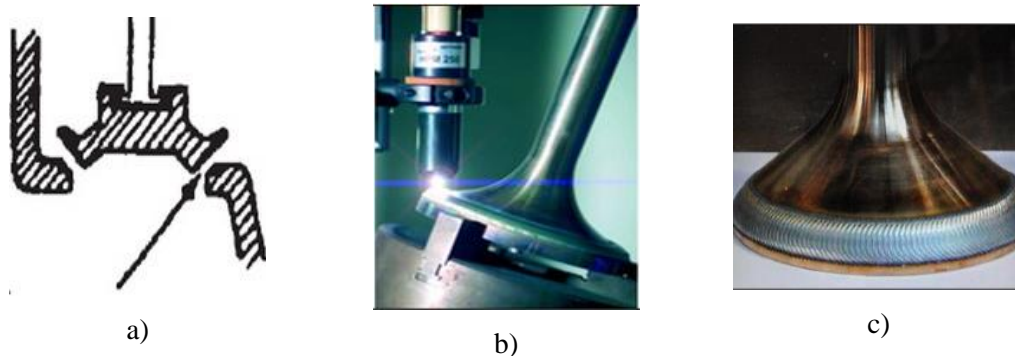


Figure 23. a) Contact surfaces between valve and seat, b) Surface during cladding, c) Cladded surface at the edge of motor engine valve

It is possible to achieve high energy torch in the plasma gun (*plasmatron*), Fig. 24a, where the stream of high velocity gases does not carry the molten particles. The plasma gun enables achieving high temperatures ( $> 4000$  K), fast and strong stream of formed plasma gas, so that different powders could be melted at the valve edge.

The laser beam produces high temperatures in interaction, even more than plasma torch. Powder for laser cladding could be fed from one or both sides, Fig. 24b. It is possible to produce a thinner layer by laser beam than by plasma torch. One of the main advantages of laser beam is in lower dissipation of an expensive powder material to be melted and clad. Depending on used powders, the hardness values of clad material range from  $\approx 390$  HV to  $\approx 550$  HV. It is reasonable to consider hardfacing. In plasma or laser cladding, different technological parameters are applicable in a wide range: speed of beam travel, powder mass flow rate, used plasma or shielded gas, pulse frequency, energy of beam, etc.

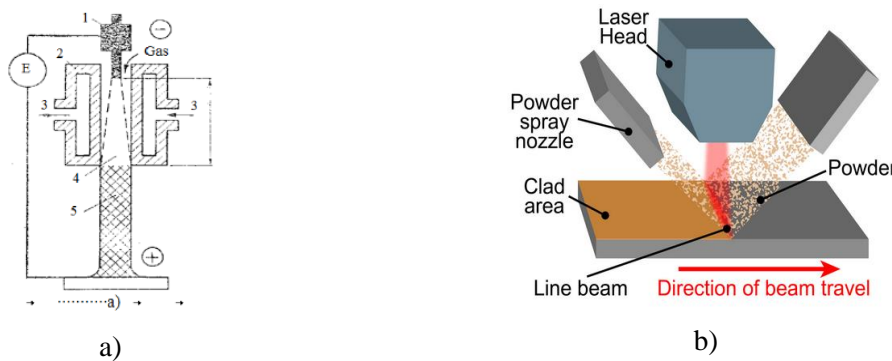


Figure 24. Cross section through: a) plasma gun and b) laser head, for cladding; 1-W electrode, 2-Cu nozzle, 3-cooling water, 4- created plasma, 5-plasma stream, E-current source

For laser cladding of Co alloys, when sufficient energy of the beam reaches levels of  $> 18$  J/pulse, CO<sub>2</sub> or pulsed Nd<sup>3+</sup>:YAG lasers are used [27]. Heat affected zone, HAZ, and especially interface layers are very small after laser cladding, Fig. 25. The pores occurring in microstructure after laser cladding are rare and small, Fig. 25a.



Figure 25. Microstructures of clad layers: a) stellite-6 layer and small interface on the parent metal; b) parent metal (left), HAZ and Ni-Cr-C-S alloy (right), magnification 300 $\times$  [27]

The dilution of clad layer with parent material is higher with plasma method than with laser cladding method. Surface roughness,  $R_a$ , after grinding of clad layer on motor engine valves [27] has to be on level  $< 1.8$   $\mu\text{m}$ .

Following statements are for further analysis. Plasma and laser claddings are cost effective methods for improving the wear- and corrosion- resistance characteristics of motor engine valves. Therefore, for surfaces, a material with better properties than the parent metal must be used. After cladding, the surface is rough. Industrial lasers are controllable power sources, meaning that the control of the heat input into the workpiece is better with the laser cladding than with the plasma gun cladding. The plasma cladding gives significantly higher power input into the workpiece, which is usually disadvantage in the sense of higher distortion of treated machine component. Lower distortion means smaller grinding thickness, which is of importance for manufacturing schedule. In both processes, it is possible to employ high-hardness alloys, even mixtures containing WC, SiC or similar intermetallic compounds, for a variety of applications [27].

## 4 Conclusion

Characteristics of plasma and laser cladding methods could be emphasized as: plasma and laser methods for cladding of motor engine valves dominate in automotive industry. In laser cladding the higher power density is achieved at the small target area than in plasma method. Hard and corrosion resistant materials may be easily melted by both methods. Obtained hardness values are in closer relation to chosen chemical composition of clad material rather than the used method for deposition of a layer. The negative role of strong plasma stream is the blowing of powder material to be clad. The smaller dissipation of powder material is achieved by using laser beam instead of plasma torch. The dilution of melted layer is lower in laser than plasma cladding. Both methods are available for producing new parts, as well as in additive manufacturing and for rebuilding of partially worn surface(s).

The main branches of the field of laser processing are: the study of technical characteristics of laser systems and laser types, as well as the study of other optical components, mechanical systems and the specificity of the designed devices. Since the era of MILK and the first PhD theses in former Yugoslavia about the industrial applications of lasers, there have been numerous other studies about basic functioning and practical applications: namely, about laser pumping, beam shaping, transportation, reflection and refraction processes. There have also been studies about systems based on fluid mechanics, system cooling, thermostating for increasing laser stability. It is important not to forget about the social and health aspects of research, i.e. the education in the application of the developed systems and demand to provide the necessary protective measures. Note that some of the old terminology for the deviation of the actual results from the nominated/projected/ fabricated results (for example, drilling diameter, surface roughness, precise contour cutting) needs to be updated – for example, the term “error” should be replaced by “uncertainty”; this not just a matter of terminology. Considering the variable nature of energy transformation, a multidisciplinary approach is inevitable.

From the economic perspective, there is no doubt about the fact that quality is the best investment, and this is true for the vehicle industry as well. In order to establish a system of quality in the automotive industry, one must have: technical documentation depending on the type of the vehicle, production equipment, personnel, realization monitoring, environmental conditions and ecological trends.

Several highly trained people are required to study on large and complex array of machines. One engineer today with a laptop can do more sophisticated studies of much larger systems. Steady-state flows are simulated as the positive sequence equivalent, presented by several  $\pi$  sections connected in series. Each of them represents a length of line as shunt capacitors and series reactive elements. The system was replaced on a large panel, the face of which was a maze of physical connection links. Unbalanced faults required an interconnection of three networks, each one for positive, negative and zero sequence. Dynamics were simulated by a step-by-step process with some representation.

Lasers are used in various ways for technical verification purposes. New measuring methods for displacement and sizes are constantly being developed, but they are all based on geometrical and physical optics, diffraction, interference, holography, holographic interferometry, etc. Methods for the measuring of vibrations in general as well as stresses and stress distribution are also based on the aforementioned physical disciplines and processes. Many of these methods are in essence classical, where the laser is only a substitute for a natural source of energy; however, many others can only be realized with the use of lasers/coherent beams. The term “coherent” should be used both in the spatial and temporal sense, but in practice, it usually refers to only one of these two. Because of the development of lasers with very short pulse (ns-ps-fs), certain measurements, which could previously only be done in a laboratory, can now be performed in the field.

Laser measuring methods are often cheaper, simpler and faster than their more classical counterparts. Lasers are also used in systems which measure displacements caused by static and dynamical loadings, which are a very important aspect of monitoring in the automotive industry. One such example is the tuning and positioning of car suspension. Also, corrosion is nowadays monitored by

small portable devices. All of these processes are carried out from a distance, without interrupting the production processes. Laser-based microanalysis of materials (like LIBS, ...) has numerous advantages over other microanalysis methods (chemical, ion and electron beams, SEM and TEM, ...). Many practical applications rely on laser plasma. Laser-based microanalysis methods are very precise even for very small samples ( $< 1\mu\text{g}$ ). There are numerous illustrations for the state of *know-how technology* and potential laser implementations.

Although they have their own histories, the topics of laser propulsion and rocket motors have connected several contemporary disciplines in recent years, initiating much theoretical research, as well as large international projects on the applied side of the areas.

A detailed examination of the comparison and current state of contemporary laser motors, which would consider both the theoretical and practical point of view, would necessarily be a very long/demanding project. Therefore, in this paper, only rough sketches of the subject matter have been given. From an educational point of view, one should neither dismiss the historical dynamics of the development of laser motors, nor the current state of today's most promising designs. The lasers have been given a special place in this paper, because many commercial and non-commercial quantum generators exist (laser transitions). One of the most important areas of research concerning lasers is the transfer of mechanical magnitudes by optical systems: buoyancy force or the transformation of electromagnetic energy to other ranges. In order to improve the functioning of laser motors, the most important aspect is increasing the efficiency of absorption (multi-layer material, coating, concentrator, etc.).

The theoretical foundations have existed for almost 30 years. The realization depends on both the needs and currently available technology. When minimizing losses in the application of lasers in motors, one must pay special attention to the processes in: plasma, transmission of energy to the working fluid, flow regulation, the mixing of hot and cold materials, the transfer of heat into the motor walls by convection and radiation, and in the protection of walls. Other important points of view include the gasdynamics of the flow through the plasma and the nozzle design. It is important to select the optimal laser wavelength for laser motors (engines), due to very large number of quantum generators. In particular, aerodynamical windows require many new solutions on their own. To reach the goals, the activities of today implement more and more, but efficient models, and also increase the level of approximation of the thermodynamic and other real processes. This can be achieved by developing better numerical models and more mathematical complex representation or mathematical symbolism/ formalism. The technical side consists of decreasing the project costs, increasing reliability, safety, "user-friendliness", simplification of design and ease of maintenance. The contemporary development of laser techniques is moving towards amelioration of laser beam characteristics and performance, stability (of frequency, polarization and mechanical one), increasing the range of applications and the choice of wavelength in the total range of the EM spectra and power (ranging from kW to MW). The emission and absorption processes in the atmosphere should be constantly studied and evaluated. That influences both the quality and the power magnitude of the laser beams entering the rocket engine from the station, located on the surface of the Earth. The contemporary development of material technology will lead to many new discoveries in the construction of new adequate materials with high hardness to temperature and pressure.

### *Acknowledgements*

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# Суб-микрометарске паралелне површинске структуре индуковане фемтосекундним ласерским снопом у форензици

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**Анстракт**— Један од ефеката интеракције ултра-кратког ласерског снопа са материјалима је формирање паралелних структура на површини (laser-induced parallel surface structures - LIPSS), чији је период мањи од таласне дужине снопа. Уколико се ради о вишеслојним танким филмовима метала, квалитет формираних структура је бољи. Узорак од пет двослојних танких филмова Al и Ti на супстрату Si смо изложили фемтосекундном снопу и запазили формирање две врсте структура које се разликују по облику. Обе су врсте вероватно узроковане појавом површинског плазмона-поларитона на површини најгорњег слоја. Појава плазмона поларитона на површини танких металних филмова и наночестица може да ограничи простирање електромагнетног поља и да појача флуоресцентни сигнал из молекула хемикалије на површини. У зависности од структуре интерфејса за одређивање циљне хемикалије на металној површини флуоресценција побољшана плазмоном (plasmon-enhanced fluorescence, PEF) је привлачан метод за скраћење времена и појачање осетљивости разних аналитичких технологија које се користе у форензици.

**Кључне речи**— фемтосекундни ласер; вишеслојни танки филмови; периодичне површинске структуре; флуоресценција.

## I. УВОД

Интеракција ласерског снопа са површином проузрокује многе ефекте, међу којима је у последње време пажњу заокупило формирање паралелних структура (LIPSS – laser induced parallel surface structures) под дејством ултракратких снопова [1]. Због карактеристике да им је периодичност мања од таласне дужине снопа, поље примене је веома широко [2]. Интеракција са металима може побољшати триболошке карактеристике, а на ламеларним материјалима, као што

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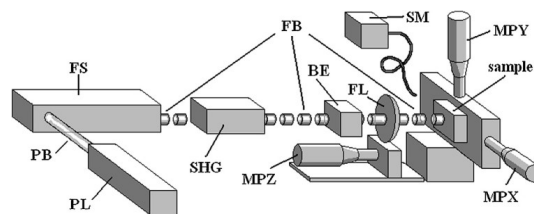
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су вишеслојни танки метални филмови, формирани LIPSS има добар квалитет и може да побољша особине површине [3-4]. Снопови различитих карактеристика (флуенца, таласна дужина, дужина импулса, поларизација, ...), разни материјали (диелектрици, полупроводници, метали, ...), али и стање амбијента током интеракције – све то утиче на карактеристике LIPSS [5]. Генерално, уочена је појава две врсте структура, са нижом просторном учестаношћу (LSFL – low spatial frequency LIPSS) и са вишом просторном учестаношћу (HSFL – high spatial frequency LIPSS) [6].

## II. ЕКСПЕРИМЕНТАЛНА ПОСТАВКА

Узорци који су коришћени су добијени депоновањем Al и Ti на подлогу од Si (1 0 0) помоћу Balzers Sputron II апарата који користи 1,3 keV аргонске јоне и са 99.9% чистоте Al и Ti мета. Две врсте узорака су подвргнуте интеракцији са фемтосекундним снопом. Први тип је био силицијумска подлога на којој је депоновано пет Al/Ti двослоја чиме је достигнута укупна дебелина од ~130 nm (сваки слој од ~13 nm). Други тип је такође био силицијумска подлога, али на којој је био депонован један слој Al дебелине ~130 nm.

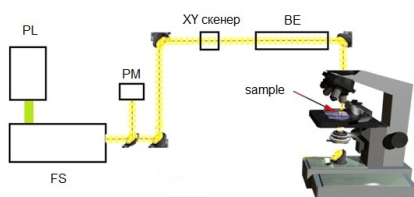


Сл. 1. Дијаграм експерименталне поставке за статичну интеракцију; PL – ласер за пумпање, PB – снап за пумпање, FS – извор фемтосекундног снопа, FB – фемтосекундни снап, SHG – удвајач учестаности, BE – проширивач снопа, FL – фокусирајуће сочиво, MPX/MPY/MPY – микропозиционери, SM – спектрометар, sample – узорак.

Извор фемтосекундног снопа је био Coherent Mira 900F систем чија је фреквенција удвојена са Inrad 5-050 ултрабрзим генератором другог хармоника. Карактеристике снопа су биле: таласна дужина 390 nm (удвојено) и 800 nm (основни хармоник), дужина импулса ~150 fs, репетиција 76 MHz (период између импулса ~13 ns), снага на мети 160–260 mW, линеарна поларизација у

горизонталној равни, Гаусов елиптични профил. Експозиције су биле од 1 до 10 s. Таласна дужина снопа је контролисана фибер-оптичким спектрометром Ocean Optics HR2000CG-UV-NIR. Интеракције су обављане у ваздуху фокусирањем фемтосекундног снопа под нормалним углом на површину првог слоја узорка (Al), сл. 1. Резултати су анализирани скенирајућим електронским микроскопима (SEM) JEOL JSM 6560 LV који има Oxford Instruments EDS анализатор и FEI SCIOS2, као и помоћу микроскопа атомских сила (AFM) NT-MDT NTEGRA Prima.

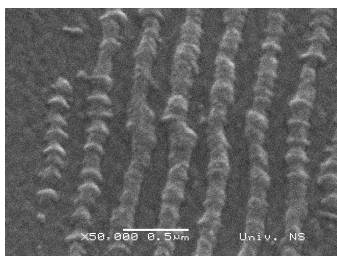
На сл. 2 је дијаграм поставке модификоване за потребе скенирања снопа преко узорка. Изостављен је удвајач учестаности, чиме је интеракција пренесена у блиску инфрацрвену област, ради скенирања се сноп уводи у огледални 2D скенер и у проширивач снопа, а фокусирање се обавља објективом модификованог микроскопа (40 $\times$ , NA=0,65).



Сл. 2. Дијаграм експерименталне поставке за интеракцију уз скенирање снопом; PL – ласер за пумпање, FS – извор фемтосекундног снопа, PM – мереље снаге снопа, XY скенер – скенер снопа, BE – проширивач снопа, sample - узорак.

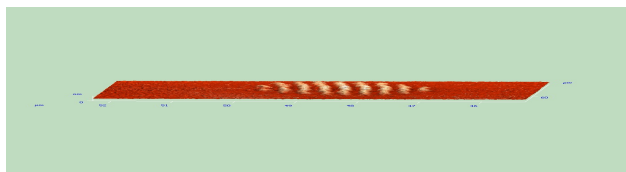
### III. РЕЗУЛТАТИ И ДИСКУСИЈА

На сл. 3 је приказ слике SEM дела области интеракције са 5 $\times$ (Al/Ti). Фемтосекундни сноп таласне дужине 460 nm је имао флуенцу од 13,6 mJ/cm<sup>2</sup> а време експозиције 10 s. У области интеракције се виде паралелне структуре са периодом од око 300 nm. Структуре су формиране у виду издигнућа на површини.



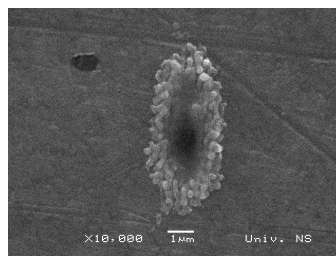
Сл. 3. Приказ слике SEM дела области интеракције са 5 $\times$ (Al/Ti) на Si; флуенца 13,6 mJ/cm<sup>2</sup> и 10 s време експозиције.

На сл. 4 је приказана AFM слика области интеракције приказаној на сл. 2. Овде се боље види да су структуре формиране као издигнућа на површини.



Сл. 4. Приказ AFM слике дела области интеракције приказане на сл. 2: 5 $\times$ (Al/Ti); флуенца 13,6 mJ/cm<sup>2</sup> и 10 s време експозиције.

Узорак другог типа – један слој танког филма (~130 nm) Al – је био изложен флуенци од 8,6 mJ/cm<sup>2</sup> исте таласне дужине и времену експозиције од 10 s. Резултати у виду слике SEM дела области интеракције су приказани на сл. 5. У централном делу је дошло до аблације топљењем, а аблирани материјал се распоредио у околини централне зоне интеракције. Дошло је до расподеле у виду LIPSS, али квалитет облика није тако добар као у случају првог узорка. Просторни период структура је око 300 nm.



Сл. 5. Приказ слике SEM области интеракције са Al на Si; флуенца 8,6 mJ/cm<sup>2</sup> и 10 s време експозиције [4].

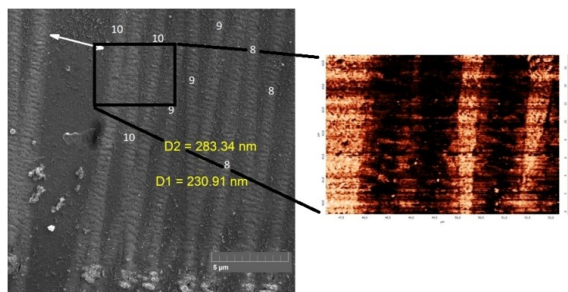
Упадни ултракратки сноп изазива на интерфејсу између металне и диелектричне површине појаву површинског плазмона (SP), а његовом интеракцијом са упадним снопом долази до формирања површинског плазмона поларитона (SPP) [2, 7], који узрокује статичну периодичну расподелу енергије на површини, периода мањег од таласне дужине упадног снопа. Дубина продирања ласерског снопа од 390 nm се израчунава [8] за Al на око 3 nm, а за Ti на око 9,3 nm.

Присуство подслоја Ti узрокује повећање прага оштећења првог (горњег) слоја, Al. Температура решетке се формира кроз два механизма: електронско-фононски (који узрокује локализацију загревања), и транспорт електронима везан за топлотну проводност електрона (који односи топлоту од зоне интеракције) [9]. Разлика у електронско-фононској спреси између два материјала води до стрме промене температуре решетке унутар подслоја (Ti). Као последицу, електрони из горњег слоја (Al) брзо преносе енергију следећем (подслоју) и тиме се термална енергија преноси кроз интерфејс Al/Ti, спреже са решетком и на тај начин односи из зоне интеракције. У првом следећем слоју (подслој, Ti) долази до гомилања термалне енергије периодично у латералном смеру, али због високе температуре топљења Ti не долази до топљења. Праг оштећења првог слоја је тиме повећан јер се енергија углавном гомила у првом подслоју, који већ

има висок праг оштећења. Одсуство подслоја Ti омогућава гомилање енергије у слоју Al и изражено топљење (сл. 5).

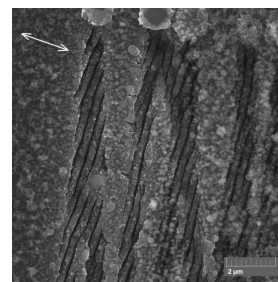
У поређењу са Al/Ti вишеслојним узорком, оптичка дубина продирања за Al је нижа, што резултује већим вредностима апсорбоване енергије и акумулације топлоте. Топљење и аблација су израженији него код вишеслојног.

Осим статичког озрачивања, вршена су и динамичка, када је снап скениран преко површине узорка. У овом случају је таласна дужина била 800 nm, а флуенца 153 mJ/cm<sup>2</sup>. На одређеним правцима је скенирање извршено вишеструким прелетом снопа преко површине узорка; на сл. 6 је приказана област дела интеракције где је снап скениран 8, 9 и 10 пута. Просторни период структуре је око 283 nm (мање од половине таласне дужине упадног снопа). У инсету на сл. 6 је приказана AFM слика дела интеракције, формирана по интензитету тунелске струје између врха пробе и узорка. Примећује се да је струја кроз формиране структуре (формиране у виду уздигнућа на површини) мања (еквивалентно тамнијој боји) него струја у деловима који нису трансформисани (светлија боја). То наводи на закључак да је дошло до смањења проводности у тим деловима, за које се може претпоставити да је узрок јача оксидација која је услед већег гомилања енергије.



Сл. 6. Приказ слике SEM (лево) и AFM (инсет-десно) дела области интеракције 5×(Al/Ti) на Si; поновљено скенирање, 8—10 пролаза, флуенца 153 mJ/cm<sup>2</sup>; бројеви 8, 9 и 10 представљају број прелета снопа преко истог правца; правац поларизације је означен белом стрелицом.

Модел изложен у [10] предлаже формирање једне врсте LIPSS на металним површинама интеракцијом са ултракратким снопом у ваздуху. У првом кораку расејање на површинским нерегуларностима индукује појаву SPP и периодичне расподеле енергије на површини. У другом кораку, на местима где се енергија акумулира долази до загревања и реакције метала са кисеоником из ваздуха. Због ултракратког импулса је оксидација бржа од термалне дифузије, и дебљина оксида расте на местима акумулиране енергије. Тиме се одржава нанометарска локализација. Како структура расте у висину, расејање се појачава и тиме је остварена позитивна повратна спрега. У трећем кораку – како структура расте – због све већег присуства оксида се смањује продирање кисеоника у структуру и тиме се цели процес зауставља. Трећи корак има негативну повратну спрегу.



Сл. 7. Приказ слике SEM дела области интеракције 5×(Al/Ti) на Si; поновљено скенирање, 10 пролаза, флуенца 215 mJ/cm<sup>2</sup>; правац поларизације је означен белом стрелицом.

На сликама 3 и 6 је приказано формирање једне врсте LIPSS, „гребен“, настале оксидацијом и надоградњом материјала. Првац простирања гребенских LIPSS је паралелан правцу поларизације упадног снопа (HSFL). За нешто веће флуенце снопа, сл. 7, долази до аблације материјала, а у аблираној области се појављује друга врста LIPSS, „канални“. Таласна дужина је 800 nm, флуенца 215 mJ/cm<sup>2</sup>, а као резултат је просторни период од 370 nm. Ширина канала је 80 nm. Аблација је наступила услед топљења/испаривања материјала. Правац каналских LIPSS управан на правац поларизације упадног снопа (LSFL).

Појава плазмона – колективне осилације наелектрисања и придруженог електромагнетног поља на површини металних филмова и наноструктура – омогућава ограничење простирања светлости у мале области и тиме је дало нова решења у оптичким спектроскопијама, као што су површински-побољшана Раманова спектроскопија (in surface-enhanced Raman spectroscopy, SERS) површински-побољшана инфрацрвена спектроскопија (surface-enhanced infrared spectroscopy, SEIRA) и плазмонски-побољшана флуоресцентна спектроскопија (surface plasmon-enhanced fluorescence spectroscopy, PEF), које се користе у низу форензичких аналитичких технологија за детекцију хемијских и биолошких агенаса релевантних у важним областима медицинске дијагностике, контроле хране и безбедности [12]. У PEF се флуорофорни означивачи спрежу са ограниченим пољем површинских плазмона што се може конструисати да веома појача интензитет емитованог флуоресцентног светла ради детектовања врло малих количина анализата са побољшаном границом детекције и скраћењем времена анализе. Просторно преклапање и усклађивање фазе између поларитона води до успостављања спрегнутих симетричних и антисиметричних модова површинског плазмона поларитона. Симетрични тип мода се може побудити на металним површинама са густим решеткама испод нивоа дифракције. Дифракција на таквој периодичној модулатији дозвољава да плазмони поларитони који се простиру у супротном смеру интерагују, стварајући појачане интензитета поља локализоване или у удубљењима („канални“) решетке или на врховима („гребени“) периодичне модулатије.

## IV. ЗАКЉУЧАК

Приказано је испитивање површинских модификација индукованих на танким филмовима – узорци од пет (Al/Ti) бислојева (укупне дебљине 130 nm) на Si као и узорци од једнослојног Al (дебљине 130 nm) на Si – помоћу фемтосекундног ласерског снопа таласне дужине од 390 nm и 800 nm и репетиције 76 MHz (~13 ns између импулса). За статичку интеракцију су формиране површинске периодичне структуре (LIPSS) на вишеслојном Al/Ti при флуенци једног импулса од 10,3–14 mJ/cm<sup>2</sup>, а на једнослојном Al при флуенци једног импулса од 8,6 mJ/cm<sup>2</sup>. Присуство подслоја повећава праг оштећења најгорњег слоја, што побољшава квалитет формираних LIPSS. За динамичку интеракцију (скенирање снопа преко површине узорка) за 153 mJ/cm<sup>2</sup> је дошло до формирања гребенских LIPSS, а за већу флуенцу једног импулса, 215 mJ/cm<sup>2</sup>, до каналских LIPSS. На основу правца простирања LIPSS се закључује њихова врста, LSFL (ниске просторне учестаности) или HSFL (високе просторне учестаности). За формирање LIPSS се механизам се види у појави површинског плазмона поларитона који изазива периодичну расподелу енергије. Присуство подслоја одговарајућих термичких карактеристика омогућава лако преношење термалне енергије у дубље слојеве. Код гребенских LIPSS има три корака везаних за продирање кисеоника из ваздуха у материјал (оксидација), док код каналских LIPSS долази до аблације топљењем/испаривањем и уклањања материјала.

Појава плазмона поларитона је важна при неким флуоресцентним техникама, као што је PEF, који је унапредио осетљивост и скратио време анализе процедура за детекцију важних анализата укључујући биомаркере, патогене и токсине. Ова једињења су детектована на ниским фемтомоларним концентрацијама, а анализа је веома скраћена. Једна од плазмона који се појављује је тесно везан са појавом периодичних структура са периодом мањим од таласне дужине (LIPSS). Индуковање LIPSS може бити интересантно у великом броју примена – медицинским, декоративним, триболошким, и др. – због појаве плазмона поларитона којим се светлост конфинира и омогућава побољшање у плазмонски побољшаној флуоресцентној спектроскопији.

## ЗАХВАЛНИЦА

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## ABSTRACT

One of the effects of the interaction of ultrashort laser beam with materials is the forming of laser-induced parallel surface structures (LIPSS), with period less than beam wavelength. For multilayer thin metal films, the quality of formed structures is better. The sample of five bilayers of Al and Ti on Si substrate was exposed to femtosecond beam and noticed the forming of two types of structures different in shape. Both are most probably the product of surface Plasmon polariton on the surface of most top layer. The occurrence of Plasmon polariton on the surface of thin metal layers and nanoparticles can confine the propagation of electromagnetic field and to amplify the fluorescent signal from molecules of the chemical compound on the surface. Depending on the interface structure for determining the target chemical on metal surface, Plasmon enhanced fluorescence is an attractive method for shortening the time of detection and increasing the sensitivity of various analytical technologies used in forensics.

### Sub-micrometer parallel surface structures induced by femtosecond laser beam in forensics

Aleksander Kovačević, Suzana Petrović, Marina Lekić, Borislav Vasić, Branislav Salatić, Jelena Potočnik

# Deskripcija, heritologija i metrologija boje

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**Apstrakt**—Ljudski osećaj za boje ima mnogo aspekata, počevši od čovekovog poimanja sveta oko sebe, do medicinskih pojmova koji uključuju i daltonizam, ali (u svakodnevnici) najpozitivniji, najhumaniji je čovekov osećaj za deo dana ili noći (meseca ili godine), za uživanje u heritološkom blagu, koje su nam ostavili preci, ali ne samo crno-belo. Istraživanja, kojima je procenjivan ljudski vek, postojanje i razvoj humanih bića, kao i nastajanje i evolucija flore i faune, sve ovo mora da bude tretirano multidisciplinarno, bez obzira da li se polazi od stena, stalaktita i stalagmita, morskih dubina, tragova u atmosferi ili potrage za planetom koja je slična Zemlji. U radu se daje nekoliko prilaza boji i kolorimetriji, sa aspekta raznih naučnih disciplina. Prikazuje se uloga lasera u novim problematikama, kvantitativne definicije boje i njenih pokazatelja. U području merenja, daje se prilaz sa aspekta filtera i korektnog opisa stanja i delovanja odabranih filtera sa savremenim izražavanjima rezultata, kao i merne metode. Konstatcija boje i njena deskripcija, ima veliku ulogu u svakodnevnom životu, kao i u pojedinim tehničkim i mass media primenama, uključivši i humanističke i tehničke nauke.

**Ključne reči**— laseri; heritologija; nove tehnologije.

## I. UVOD

Spektroradiometrijska metoda, kao fundamentalna metoda kvantifikacije, deskripcije objektivnog merenja boje, prema trenutnom stanju razvijenosti tehnologije izrade prijemnika optičkog zračenja i ostale merno-tehničke opreme, ima kao glavni zadatak postizanje zadovoljavajuće tačnosti i ponovljivosti rezultata merenja, prema postavljenim zahtevima korisnika.

Spektroradiometrijske metode merenja boje, zasnovane su na određivanju spektralne raspodele zračenja, na osnovu čega se formira boja koja se meri. Trihromatske vrednosti boje se nalaze iz poznatih krivih mešanja boja (slika 1). Za primarne svetlosne izvore, veličina koja se meri je spektralna raspodela energije, a za sekundarne se koristi spektralni koeficijent propustljivosti i koeficijent jednog od procesa interakcije, ovde koeficijent refleksije optičkog zračenja (koje se reflektuje ili propušta kroz određene sredine).

U ovom delu rada, izabran je slučaj kada ne postoji poznata raspodela zračenja izvora. Koristi se raspodela zračenja crnog

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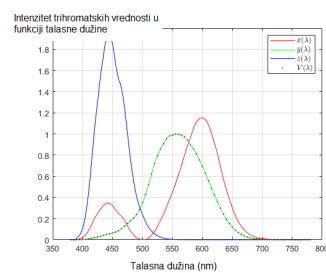
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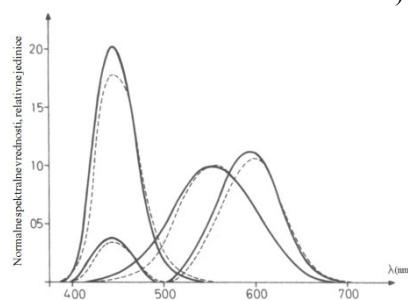
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tela i spektralni koeficijent propustljivosti staklenih uzoraka. Na taj način, proračun trihromatskih vrednosti uzima u obzir pored spektralnog koeficijenta propustljivosti i spektralnu raspodelu snage zračenja apsolutno crnog tela (ACT). Spektralna raspodela snage zračenja se množi poznatim spektralnim koeficijentom propustljivosti  $\tau(\lambda)$ . Na sl. 1 je predstavljena spektralna karakteristika trihromatskih vrednosti u funkciji talasne dužine.



a)



b)

Sl. 1. a) Krive mešanja boja; b) Krive za polje viđenja 100° u odnosu na isprekidane krive za 20°.

Proračun trihromatskih vrednosti u CIE (XYZ) standardnom kolorimetrijskom koordinatnom sistemu, za boje svetlosti, koje se propuštaju kroz posmatrani uzorak, sledi procedura:

$$X = \int_{350nm}^{750nm} E_{\lambda} \tau_{\lambda} \overline{X}_{\lambda} d\lambda; Y = \int_{350nm}^{750nm} E_{\lambda} \tau_{\lambda} \overline{Y}_{\lambda} d\lambda; Z = \int_{350nm}^{750nm} E_{\lambda} \tau_{\lambda} \overline{Z}_{\lambda} d\lambda. \quad (1)$$

Kod praktičnih proračuna, integracija se svodi na sumiranje proizvoda vrednosti spektralnog koeficijenta propustljivosti i spektralne snage zračenja ACT za nizove talasnih dužina:

$$X = \Delta\lambda \sum_{\lambda} E_{\lambda} \cdot \tau_{\lambda} \cdot \overline{X}_{\lambda}; Y = \Delta\lambda \sum_{\lambda} E_{\lambda} \cdot \tau_{\lambda} \cdot \overline{Y}_{\lambda}; Z = \Delta\lambda \sum_{\lambda} E_{\lambda} \cdot \tau_{\lambda} \cdot \overline{Z}_{\lambda}. \quad (2)$$

Interval  $\Delta\lambda$ , bira se 1-20 nm, u zavisnosti od željenih nesigurnosti tipa A i B, koje se zahtevaju za određivanje hromatskih koordinata. Kako su krive mešanja boja specifikovane u obliku relativnih vrednosti na ordinati, trihromatske vrednosti izračunate pomoću njih imajuće relativ-

ni karakter kao na slici u poglavlju rezultata (sl. 6) (primer proračuna trihromatskih vrednosti za odabrani filter) [1-4].

Zahtev je da se odrede trihromatske vrednosti i hromatske koordinate boje standardnog svetlosnog izvora tipa  $A$  sa spektralnom raspodelom energija  $E_A$ , posle prolaska kroz uzorak, koji ima određen spektralni koeficijent propustljivosti  $\tau$ . Posle transmisije kroz uzorak, relativna spektralna raspodela jačine zračenja ima oblik prikazan krivom  $\tau E_A$ . Svaka ordinata ove karakteristike, množi se odgovarajućom ordinatom svake od krivih mešanja boja  $\bar{X}$ ,  $\bar{Y}$ ,  $\bar{Z}$ . Šrafirane površine ispod krivih, dobijenih posle množenja, proporcionalne su odgovarajućim trihromatskim vrednostima  $X$ ,  $Y$  i  $Z$ . Hromatske koordinate  $x$  i  $y$  se izražavaju dalje:

$$x = \frac{X}{X+Y+Z}, \quad y = \frac{Y}{X+Y+Z}. \quad (3)$$

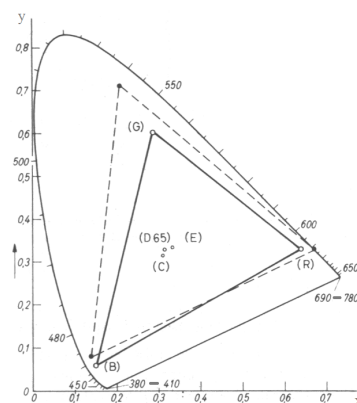
Ordinate krivih mešanja boja i ordinate spektralnih raspodela energije za standardni izvor, potrebne za posmatrani metod proračuna, obično se daju tabelarno.

Kod primarnih izvora svetlosti, postavka metode, zasniva se na spektrometru koji se koristi za merenje relativne spektralne ozračenosti (iradijancije). Kod sekundarnih izvora, merenje se vrši pomoću spektrofotometara, gde se monohromator sa odgovarajućim sistemom osvetljavanja (u zavisnosti od geometrije merenja), koristi za osvetljavanje datih uzoraka, a reflektovano, odnosno propušteno zračenje se meri fotodetektorom u odgovarajućoj geometriji.

## II. DRUGI PRILAZ BOJI

Boja se može pojaviti kao kategorija ili pojam u vrlo složenim multidisciplinarnim problemima, a često se prema naučnoj disciplini, koja se oslanja na doživljaj boje živih organizama (sa medicinske tačke gledišta, sa filozofske tačke gledišta, sa gledišta heritologije, sa ekološkog gledišta i dr.). Šire gledano, radi se o interakciji zračenja elektromagnetne ili druge prirode sa *prijemnim* aparatom. Sa tehničke strane objektivizacije, kolorimetrija ima zadatak da se kroz različite formirane formalizme opisuje boja putem trougla i koordinata, ili na drugi način, kroz drugi formirani sistem višeg ili nižeg nivoa, sl. 2. U tabeli 1 se daju ilustracije nivoa bioloških procesa uzrokovanih zračenjem i vremenskih konstanti odgovarajućih procesa sa odgovarajućim nivoom [5]. Sa obzirom na *mass media* primene (TV, novine, ...), kulturne manifestacije, znake opasnosti, dozimetrijska pravila u primeni određene ELIONSke tehnike, boja mora da ima dobro definisane koordinate u širem smislu, zasnovane na izvorima (obično tri), čijim se mešanjem postiže efekat određene (željene) boje. Da li će se koristiti odgovarajući softver uz profesionalnu mernu aparaturu, ili će se sve automatizovati kao *user-friendly*, zavisi od trenutnog administrativnog zahteva ili trenutnog nadahnuća umetnika [6].

Prema visini stepena tolerancije, institucije koje se kod nas bave *deskripcijom boje*, kao i njenim promenama, u zavisnosti od izloženosti objekta, odnosno bio-objekta, postoje stroge procedure i sistemi gde se to može izvršiti, a ako nije potrebna



Sl. 2. Normiranje boje putem EBU trougla boja (neprekidno) i FCC (isprekidano) [2].

TABELA I

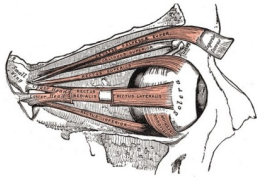
Nivo	Vreme na nivou (s)	Procesi na nivou, modifikacije
Fizički	$10^{-18} - 10^{-8}$	Eksitacije, jonizacije, elastični sudari, formacija radikala visoke reaktivnosti i kratkoživećih SR u vodi i organskim molekulima
Hemijski	$10^{-14} - 10^{-4}$	Reakcije SR sa organelama formiraju primarno oštećenje DNA, dimerizacija, modifikacije sa temp. MT, i $O_2$
Biohemijski (subćelijski)	$10^{-4} - 10^5$	Reparacija, interakcije oštećenih mikrocentara; mutacije, aberacije, MT i dr. agensi
Biohemijski (ćelijski)	$10^3 - 10^7$	Deoba ćelija i molekula, Mutacije

Prema visini stepena tolerancije, institucije koje se kod nas bave *deskripcijom boje*, kao i njenim promenama, u zavisnosti od izloženosti objekta, odnosno bio-objekta, postoje stroge procedure i sistemi, gde se to može izvršiti, a ako nije potrebna velika preciznost, korisnici formiraju svoja sopstvena merna rešenja [7]. U ovim merenjima ima mnogo mesta za definisanje osvetljenosti, u širem smislu, određenog izložbenog prostora i kontrolu stalnog fluksa, koji se, u zavisnosti od mešanja prirodne i veštačke osvetljenosti, mora kontrolisati. U zavisnosti od objekta, mora se obratiti pažnja na mere zaštite od preterane ekspozicije.

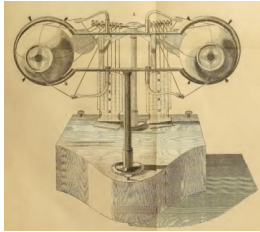
Paralelno anatomija oka, njegovog funkcionisanja, mišićnog sistema i osetljivost (kao prijemnika) na definisana zračenja i boje vidljivog spektra su predmet mnogobrojnih istorijskih radova, uključujući i današnja istraživanja na mehanizmima funkcionisanja očnog aparata, modelovanja i kvalitativno/kvantitativno povezivanje sa tehnikama merenja i povezivanja sa *tehničkim uglovima gledanja*, sl. 3 i 4. Poređenje osetljivostu pojma viđenja u oblasti fizike, metrike i psihologije, razlikuje kvantitativnom, kvalitativno i vrednovanje sa tri hromatske koordinate

V. REZULTATI

Merene vrednosti tabele 2 su prikazane delimično i na sl. 6, odnosno  $\tau(\lambda)$ ,  $x(\lambda)$ ,  $y(\lambda)$ ,  $z(\lambda)$ , prema propisanoj proceduri u postupku određivanja karakteristika staklenog filtra MEL-B-1.



Sl. 3. Anatomija oka, koja je još kod Helmholtza imala posebne predstave [8].



Sl. 4. Istorijska slika modelovanja očnog aparata [9].

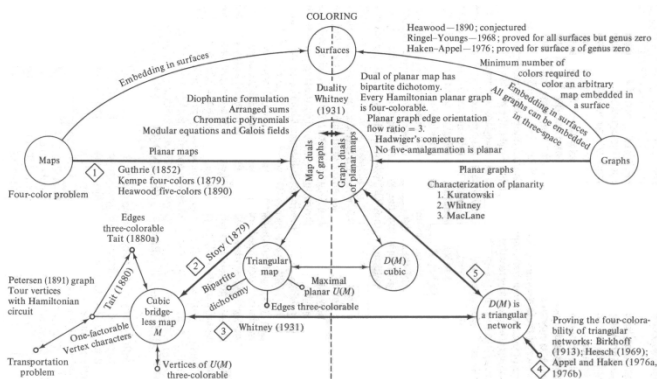
Poređenje osetljivostu pojma viđenja u oblasti fizike, metrike i psihologije, razlikuje kvantitativnom, kvalitativno i vrednovanje sa tri hromatske kordinate.

III. ULOGA IZVORA

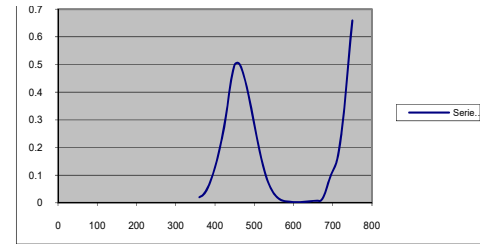
Pri definiciji boje u bilo kom sistemu širina spektralnih linija (spontane ili koherentne svetlosti) igra presudnu ulogu. Kao rezultat toga su nastali RGB laseri, od specijalnog značaja za HDTV. Od novijih tipova lasera, pokazalo se da su laseri na bazi polimera pogodni za kalibraciju, odnosno deskripciju boje [5, 10].

IV. TEOREMA-PROBLEM ČETIRI BOJE

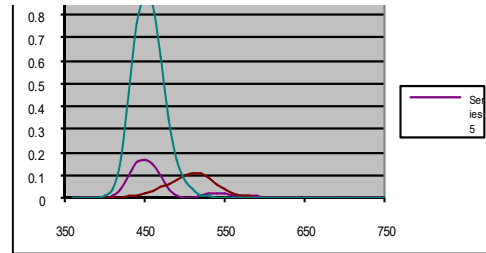
Klasični problem četiri boje zahteva mnogo raznih pogleda koji su ilustrovani sl. 5 datom u originalu, vezanu za teoremu i problem četiri boje, gde kao dokaz mora da bude uključena i savremena računarska tehnika; to ne uključuje nelinearne relacije i osećaje boje uz velike intenzitete, o čemu su istraživanja morala da se intenziviraju posle razvitka snažnih laserskih snopova [11-17].



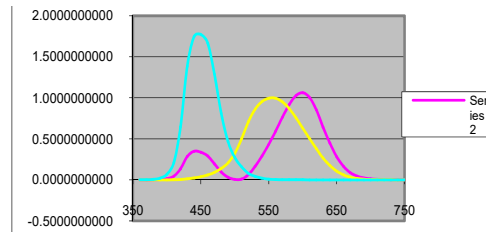
Sl. 5. Prikaz deskripcije problematike teoreme četiri boje [17].



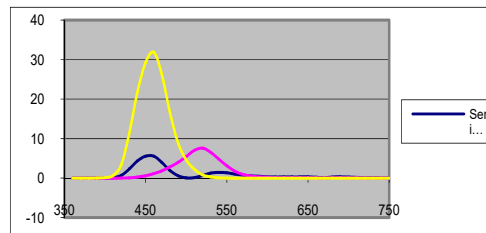
a)



b)



c)



d)

Sl. 6.  $\tau(\lambda)$ ,  $x(\lambda)$ ,  $y(\lambda)$ ,  $z(\lambda)$ ,... su prikazane na a), b), c), d), redom.

Pored merenja u tabeli II, izvršena su merenja i za seriju drugih filtera: MEL-B-2, MEL-G-2, MEL-G-4, itd.



TABELA II  
VREDNOSTI SPEKTRALNE PROPUSTLJIVOSTI STAKLENOG FILTERA MEL-B-1

$\lambda$ (nm)	$\tau(\lambda)$	$x(\lambda)$	$y(\lambda)$	$z(\lambda)$	$x$	$y$	$z$	$E_{e,1 \text{ rel}}$	$X$	$Y$	$Z$
360	0,020104	0,00013	3,29E-6	0,000606	2,61E-6	7,87E-8	1,22E-5	6	1,61E-5	4,84E-7	7,49E-5
370	0,028827	0,000415	1,24E-5	0,001946	1,20E-5	3,57E-7	5,61E-5	8	9,35E-5	2,79E-6	0,000439
380	0,049412	0,001368	3,90E-5	0,00645	6,76E-5	1,93E-6	0,000319	10	0,000662	1,89E-5	0,00312
390	0,084852	0,004243	1,20E-4	0,02005	0,00036	1,02E-5	0,001701	12	0,004353	1,23E-4	0,020569
400	0,128765	0,01431	3,96E-4	0,06785	0,001843	5,10E-5	0,008737	15	0,027105	7,50E-4	0,128517
410	0,184124	0,04351	1,21E-3	0,2074	0,008011	2,23E-4	0,038187	18	0,141639	3,94E-3	0,675152
420	0,251353	0,134338	4,00E-3	0,6456	0,033777	1,01E-3	0,162273	21	0,709313	2,11E-2	3,407743
430	0,332946	0,2839	1,16E-2	1,3856	0,094523	3,86E-3	0,46133	25	2,331892	9,53E-2	11,38101
440	0,435524	0,34828	2,30E-2	1,74706	0,151684	1,00E-2	0,760887	29	4,353339	2,87E-1	21,83744
450	0,498869	0,3362	3,80E-2	1,77211	0,16772	1,90E-2	0,884051	33	5,549847	6,27E-1	29,25324
460	0,504937	0,2908	6,00E-2	1,6692	0,146836	3,03E-2	0,842841	38	5,553325	1,15E+0	31,87624
470	0,476985	0,19536	9,10E-2	1,28764	0,093184	4,34E-2	0,614185	43	3,994789	1,86E+0	26,33011
480	0,424312	0,09564	1,39E-1	0,81295	0,040581	5,90E-2	0,344944	48	1,958043	2,85E+0	16,64357
490	0,355894	0,03201	2,08E-1	0,46518	0,011392	7,40E-2	0,165555	54	0,614152	3,99E+0	8,925058
500	0,283355	0,0049	3,23E-1	0,272	0,001388	9,15E-2	0,077073	60	0,083112	5,48E+0	4,613563
510	0,212164	0,0093	5,03E-1	0,1582	0,001973	1,07E-1	0,033564	66	0,130345	7,05E+0	2,217261
520	0,147607	0,06327	7,10E-1	0,07825	0,009339	1,05E-1	0,01155	73	0,677084	7,60E+0	0,837393
530	0,095958	0,1655	8,62E-1	0,04216	0,015881	8,27E-2	0,004046	79	1,256667	6,55E+0	0,320127
540	0,058893	0,2904	9,54E-1	0,0203	0,017103	5,62E-2	0,001196	86	1,469962	4,83E+0	0,102756
550	0,033716	0,443345	9,95E-1	0,00875	0,014614	3,35E-2	0,000295	93	1,357805	3,12E+0	0,02741
560	0,017632	0,5945	9,95E-1	0,0039	0,010482	1,75E-2	6,88E-5	100	1,048222	1,75E+0	0,006876
570	0,009053	0,7621	9,52E-1	0,0021	0,006889	8,62E-2	1,90E-5	107	0,739466	9,24E-1	0,02038
580	0,005345	0,9163	8,70E-1	0,00165	0,004898	4,65E-3	8,82E-6	114	0,560484	5,32E-1	0,001009
590	0,00365	1,0263	7,57E-1	0,0011	0,003746	2,76E-3	4,02E-6	122	0,456	3,36E-1	0,000489
600	0,002479	1,0622	6,31E-1	0,0008	0,002633	1,56E-3	1,98E-6	129	0,339787	2,02E-1	0,000256
610	0,002195	1,0026	5,03E-1	0,00034	0,002201	1,10E-3	7,46E-7	136	0,300044	1,51E-1	0,000102
620	0,002549	0,85445	3,81E-1	0,00019	0,002178	9,71E-3	4,84E-7	144	0,312803	1,39E-1	6,96E-5
630	0,0031	0,6424	2,65E-1	0,00005	0,001991	8,22E-4	1,55E-7	151	0,300369	1,24E-1	2,34E-5
640	0,004279	0,4479	1,75E-1	0,00002	0,001917	7,49E-4	8,56E-8	158	0,302779	1,18E-1	1,35E-5
650	0,007347	0,2835	1,07E-1	0	0,002083	7,86E-4	0	165	0,343737	1,30E-1	0
660	0,007538	0,1649	6,10E-2	0	0,001243	4,60E-4	0	172	0,213749	7,91E-2	0
670	0,008502	0,0874	3,20E-2	0	0,000743	2,72E-4	0	179	0,132839	4,86E-2	0
680	0,03321	0,04677	1,70E-2	0	0,001553	5,65E-4	0	185	0,288016	1,05E-1	0
690	0,078896	0,0227	8,21E-3	0	0,001791	6,48E-4	0	192	0,343735	1,24E-1	0
700	0,115048	0,011359	4,10E-3	0	0,001307	4,72E-4	0	198	0,259096	9,36E-2	0
710	0,149673	5,79E-3	20,9E-3	0	0,000867	3,13E-4	0	204	0,177154	6,40E-2	0
720	0,226687	2,90E-3	1,05E-3	0	0,000657	2,37E-4	0	210	0,138257	4,99E-2	0
730	0,354033	1,44E-3	5,20E-4	0	0,00051	1,84E-4	0	216	0,110177	3,98E-2	0
740	0,507815	6,90E-4	2,49E-4	0	0,00035	1,27E-4	0	222	0,077677	2,81E-2	0
750	0,659319	3,32E-4	1,20E-4	0	0,000219	7,91E-5	0	227	0,049734	1,80E-2	0
									36,70767	5,06E+1	158,6117

Kao primer merne nesigurnosti za drugu vrstu filtra data je tabela III. Za sva merenja je potrebno dati ocenu nesigurnosti tipa A i B, a za slučaj filtra MEL-Y-2 ocena je u Tabeli III.

Merna nesigurnost ovako dobijenih vrednosti hromatskih koordinata boje propustljivih stakala u boji, svodi se na mernu nesigurnost etalonskog spektrofotometrijskog sistema u

pogledu merenja spektralnog koeficijenta propustljivosti i tačnosti zauzimanja talasne dužine, kao i merne nesigurnosti zbog rasipanja rezultata (nehomogenog filtra). Ukupna procenjena merna nesigurnost dobijenih rezultata se pridružuje dobijenim vrednostima hromatskih koordinata; u zavisnosti od opsega hromatskih koordinata komponente

merne nesigurnosti će biti različite posebno za svaku od koordinata,  $x$  i  $y$ . U ovom radu je predstavljena samo ukupna vrednost merne nesigurnosti. Budžeti merne nesigurnosti za pojedine filtere se proračunavaju na isti način kao u tabelama 2 i 3.

Razvojem spektrofotometrijskog sistema u laboratoriji Analysis d.o.o, ostvarena je cela procedura etaloniranja različitih transparentnih (propustljivih) uzoraka, za različite primene. Poznavanjem svojstva materijala (spektralni koeficijent propustljivosti, spektralna osetljivost detektora, odnosno spektralna raspodela izvora zračenja, može se realizovati još jedna karakteristika materijala – boje. Na osnovu dobijenih vrednosti, moguće je definisati boju datih uzoraka u najširem smislu i tako dobiti novu karakteristiku kvantitativno/objektivno.

TABELA III

PRIMER PRORAČUNA BUDŽETA MERNE NESIGURNOSTI STAKLENOG FILTERA U BOJI MEL-Y-2

Komponenta merne nesigurnosti	Tip A	Tip B
Merna nesigurnost spektrofotometrijskog sistema: -Prosečno rasipanje rezultata -Pomeraj svetlosnog snopa -Orijentacija uzorka u odnosu na optičku osu sistema -Nesigurnost korekcije na interrefleksiju -nelinearnost sistema -Neuniformnost uzoraka	0,15 %	
Merna nesigurnost zauzimanja $\lambda$		0,001 %
Merna nesigurnost izvora zračenja (apsolutno crnog tela) iz literature		1 %
Ukupna relativna merna nesigurnost	1,12 %	

## VI. ZAKLJUČAK

Shvatanje boje je složeno pitanje zavisno od prilaza pojedine naučne discipline, ali je multidisciplinarni problem koji zahvata od heritologije i umetnosti do matematike, elektrotehnike, i pojmova osvetljenosti, interakcije sa zračenjima razne prirode. Kad se priđe sa gledišta koherentnosti, unitarnosti izvora osvetljenja i podseti se da se u doživljavanju boje pojavljuju razne terminologije, propisi i uobičajene, ali i specifične merne tehnike, danas se situacija komplikuje sa linearnim i nelinearnim procesima, koje donose laseri velikih gustina snopa i kratkih impulsa, pa se i aberacije hromatske prirode još više komplikuju, a i doživljaji bioloških objekata. Uz klasične aberacije pojavljuju se nove klase.

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## ABSTRACT

The human sense of color has many aspects, from man's understanding of the world around him to medical terms that include colorblind people, but (in everyday life) the most humane is man's sense of that part of the day or night (month or year), to enjoy heritage , left to us by our ancestors. Research assessing human life span, the existence and development of human beings, as well as the origin and evolution of flora and fauna, all this must be treated multi disciplinary, whether we start from rocks, stalactites and stalagmites, sea depths, atmospheric traces or the search for an Earth-like planet. The paper gives several approaches to color and colorimetry, from the aspect of various scientific disciplines. The role of lasers in new problems, quantitative definitions of color and its indicators are presented, and in the field of measurement, an approach is given from the aspect of filters and a correct description of the condition and operation of selected filters with modern expression of results and measurement methods. The perception of color and its description play a major role in everyday life, as well as in certain technical and mass media applications, including the social and technical sciences.

Key words: lasers, heritology, new techniques/methods.

## Deskripcija, heritolog and color metrology

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Milena Davidović, Suzana Polić

# Različiti režimi rada kvantnih generatora kao instrument za modifikacije u stomatologiji

Aleksandar Bugarinović<sup>1,2</sup>, Željka Tomić<sup>3</sup>, Sanja Jevtić<sup>4</sup>, Aleksander Kovačević<sup>5</sup>, Svetlana Pelemiš<sup>6</sup>, Zoran Nedić<sup>7</sup>, Dragan Družijanić<sup>1</sup>

**Apstrakt** - Upotrebom kvantnih generatora u raznim oblastima, a i u stomatologiji, bavi se veliki broj istraživača sa perspektivom da taj broj bude i veći. Ciljevi su različiti: od dobijanja materijala za dalje formiranje do učesća u formiranju modifikovanih materijala, modifikacije osobina već tradicionalnih materijala do postizanja zadataka koji se pojavljuju u vezi sa zahtevima heritologije / forenzike ili za masmedija primenu. Specijalno, dijagnostičke tehnike na bazi jačih ili slabijih interakcija elionskih snopova sa materijalom, postaju sve više tražene merne tehnike zbog vremena same dijagnostike i mogućnosti ispitivanja karakteristika uzoraka i u malim lokalnim površinama, a skeniranjem po potrebi i u većim. Podrazumevaju se i kvantitativne i kvalitativne analize, a rad sa više od jednog snopa (manje i veće gustine snage), pored mogućnosti praćenja interakcije sa teoretske strane, pruža i praktičan deo aplikacije laserske ili uopšte elionske tehnike, koji je ušao u praksu ili je od potencijalnog interesa za protetičke i druge svrhe. Šta bi onda moglo biti generalno novo u ovoj oblasti? Novi dinamički režimi rada kvantnih generatora, oblici impulsa, njihovo trajanje i frekvencija, donose novi kvalitet, koji može proizvesti drugačije efekte pri interakciji laserskih snopova sa materijalom, uspostaviti nove standarde i/ili potisnuti stare. Rad se bavi interakcijom laserskih snopova, u različitim režimima rada, sa odabranom klasom materijala. Diskutovani su modeli za opis i rezultati interakcija, analizirana se oštećenja i dat je osvrt na trenutna i potencijalna polja primene i izabrane paralele sa drugim snopnim tehnikama.

**Ključne reči** - kvantni generatori, režimi rada, interakcije, modeli, polje primene.

## I. UVOD

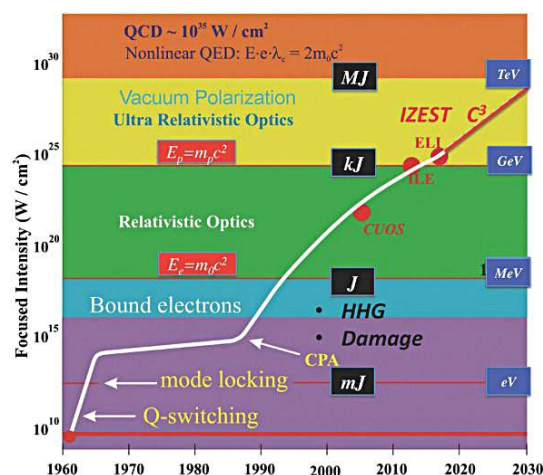
Primena laserske tehnike ili, u širem smislu, kvantnih generatora, treba da, na prvi pogled, menja značenje *modifikacije* sa kvantifikacijom od minornih uticaja na materijal mete do totalne ili lokalne destrukcije mete, zavisno od režima rada sistema kojim se vrši tretman.

Pošto je stvarnost omogućila da se interakcija koristi: a) u dijagnostičke svrhe, b) u strogo *modulacione* svrhe c) za spajanje materijala iste vrste ili različitih tipova d) za razdvajanje materijala, to postoji i grupisanje gustina energije, snage, primena *cw* ili impulsnog rada sa različitim parametrima, slike 1 i 2.

Vremenska baza impulsa (širine impulsa) je u rasponu od prvih milisekundnih impulsa sa stotinak spajkova [1] do *fs* režima.

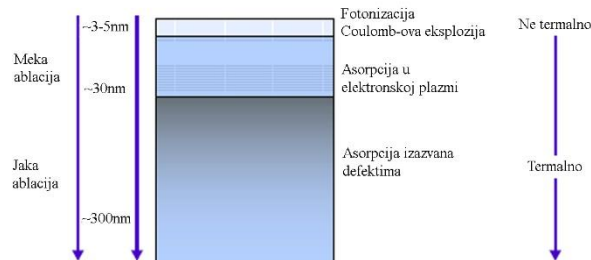
U pogledu materijala, koji su od interesa za stomatologiju, veoma je široka njihova paleta, zato što se danas koristi laserska tehnika u svakodnevnoj kliničkoj praksi: (a) za

saniranje *biološkog aparata*, uključujući i meka i tvrda tkiva, (b) u protetičke svrhe.



Slika 1. Istorija i predviđanje razvoja fokusiranog intenziteta od prvih demonstracija lasera (sa različitim režimima rada).

U svim tim procesima, postavljali su se i postavljaju se i dalje objektivizacije opisa procesa, pored toga što se veliki broj interakcija koristi u svakodnevnoj praksi, s obzirom na višedecenijsku primenu lasera u stomatologiji, zavisno od zemlje i njene regulative. To znači da su uključeni skoro svi glavni tipovi materijala: metali, dielektrici, specijalne klase keramike i dr. Posebno su važne i primene za paradentozne tretmane u kojima su korišćeni laseri malih snaga (reda mW) i gde se radilo samo o ozračavanju tkiva u vremenu predviđenom protokolom (kada je odobrena ta terapija u pojedinoj državi). Za simulaciju procesa interakcije prema očekivanom izlazu, koriste se modeli od termalnog do modela teorija sličnosti i zavisno da li se radi o proceni naponskog stanja protetske konstrukcije, do holografskih studija lokalnog opterećenja tačaka od interesa.



Slika 2. Procesi ablacije izazvanih laserima i neki procesi uz slojeve materijala od površine.

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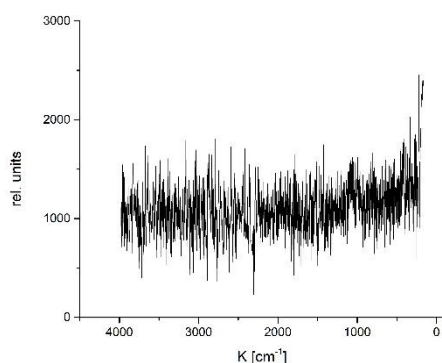
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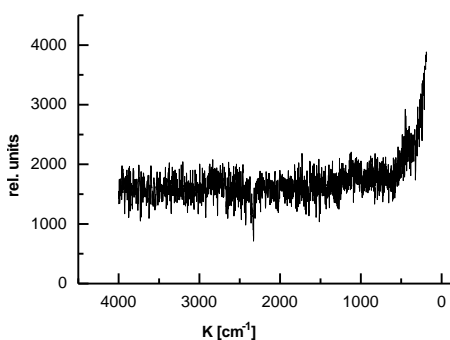


Slika 6. Mikrograf povrede na uzorku HAP-a. (Nd<sup>3+</sup>:YAG, Q-switch, E = 35 mJ, površinska modulacija, OM 40 x)

Na slikama 7 i 8 dati su IC spektri za uzorke gutaperke i HAP-a.



Slika 7. Infracrveni spektar (IC) uzorka gutaperke u dijapazonu 4000 - 180 cm<sup>-1</sup>.



Slika 8. Infracrveni spektar (IC) uzorka HAP-a u dijapazonu 4000 - 180 cm<sup>-1</sup>.

Idealno bi bilo da se spektri uvek rade pre i posle interakcije, zato što bi zavisno od površine povrede mogao da se sprovede formalizam za dobijanje optičkih konstanti materijala, kao što su koeficijenti refleksije, koeficijenti dielektričnih osobina, vezano za optičke osobine i kompleksan zapis. Ovde bi od koristi bio i *Raman*-ov spektar, koji bi i samim poređenjem sa IC spektrima dao neki kvalitativan odgovor bez sprovođenja primene algoritama za analizu strukture materijala.

Pretpostavka o novim materijalima, čiji broj raste u stomatologiji, je uvek potrebna, a za poznate materijale potrebna je iz razloga kontrole postojećih podataka i

izbegavanje razlika u tehnologiji izrade protetskog materijala. Čini se da je dobro ponoviti uvek provere i za poznate materijale.

Od interesa bi bila i tehnika termovizije, koja je skoro dve decenije bila prisutna sa prvim primenama lasera u stomatologiji, u vreme kada je ekonomska strana nabavke te kontrole predstavljala veći problem. To su eksperimenti koji mogu pratiti proces za određene dinamičke dijapazone kvantnih generatora, kad se radi o dugim impulsima ili srednjim vrednostima, pri većim repetitivijama, kao kontrolna relativna tehnika.

Važna oblast bi bilo i određivanje boje kod zuba ili protetičkih materijala, gde bi moglo biti od interesa vezivanje sa trenutnim stanjem deskripcije boje, u kojima glavne boje pripadaju kategoriji lasera (za televizijske svrhe HDTV, odavno je razvijen tip RGB lasera).

### III. SIMULACIJA

Kako je u uvodu konstatovano, posle izabranog modela, za određene režime rada kvantnih generatora [9] se konstatuje da i više modela mogu da se koriste za određene režime, koji odgovaraju tipu grejanja materijala do tačke ključanja. To su slučajevi izlaganja patogenog tkiva (paradentozna) koje se već više od deceniju koristi za saniranje stanja. Slučajevi bušenja (otklanjanja materijala), zavisno da li se problemu prišlo sa redosledom grejanje-topljenje ili grejanje-sublancija i sl., bi morali da koriste odgovarajuće algoritme kod programa kod kojih su oni razvijeni [5].

Pošto je za bušenje najbitnije pitanje dimenzija (dubina, širina), onda se za preliminarnu ocenu koriste formule koje zavise od parametara izlaganja, termodinamičkih i optičkih osobina materijala.

U širokoj paleti materijala, čini se da je situacija *Status quo* i da se može naći mnogo praktičnih korekcija za izabrane režime rada, gde će se za promenu koeficijenata u formulama za standardne uslove rada približiti simulacija eksperimentu. Pri interakciji je jedna od važnih činjenica pitanje maksimalne gustine snage / energije, koja neće dovesti do praga za „lom”. Sve to pripada složenoj problematici „laser damage”, gde se uključuje statistika malih brojeva i statistika velikih brojeva, koje se različito modeluju.

### IV. ZAKLJUČAK

U današnjem stepenu laserske tehnike postoji nekoliko definisanih režima rada kvantnih generatora, koji potencijalno mogu da se primene u biomedicinske svrhe. U praksi, iako postoji dosta dobro razvijena industrija laserskih sistema okrenutih medicini, pokazalo se da, generalno, postoji niz pozitivnih novih pravaca za primenu lasera. Ipak, od stanja same države i stepena njenog instrumentarijuma, vezano za klasičnije metode (mehanička bušilica, rentgenografija, poznate klase protetskih materijala sa komercijalnom podrškom), zavisi koliko će u njoj biti brzo uklapanje novih „optičkih” metoda u sve četiri uslovno podeljene oblasti: dijagnostika, modulacija u užem smislu (tkiva ili materijala), invazivna interakcija (hirurgija) i saniranje i formiranje ispuna i pravljenje mostova i dr. U ovom radu je pokazana interakcija sa tipovima odabranih materijala (keramičkog tipa i dr.), izvršene su analize sa strane optičke i elektronske mikroskopije, definisanje materijala mete putem IC spektroskopije. Ukazano je na

potrebu daljeg eksperimentalnog rada, vezano za stanje uzoraka i za, potencijalno, organizaciju daljih ispitivanja ili primena holografskih i tomografskih tehnika za definisanje dinamike opterećenja, uključujući tehnike brzog starenja materijala, koje bi služile za ocenu, vek trajanja protetskog nastavka i pravac traženja, kroz krojenja novih vrsta materijala od interesa za protetiku i prihvatanje „stranog tela”. Po pitanju samog HAP-a, bile su razvijene dve teorije koje su primenjivane na mehanizam stapanja sa organizmom.

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## ABSTRACT

The use of quantum generators in various fields, as well as in dentistry, is dealt with by a large number of researchers with the perspective that the number will be even higher. The goals are different: from obtaining materials for further formation to participating in the formation of modified materials, modifying the properties of already traditional materials to achieving tasks that appear in connection with the requirements of heritology / forensics or for mass media application. In particular, diagnostic techniques based on stronger or weaker interactions of elion beams with material are becoming more and more sought-after measurement techniques due to the time of diagnostics and the possibility of testing sample characteristics in small local areas, and scanning if necessary in larger ones. Quantitative and qualitative analyzes are included, and work with more than one beam (lower and higher power densities), in addition to the possibility of monitoring the interaction from a theoretical point of view, provides a practical part of the application of laser or elion techniques in general, which has entered into practice or is of potential interest for prosthetic and other purposes. What, then, could be generally new in this area? New dynamic modes of operation of quantum generators, pulse shapes, their duration and frequency, bring new quality, which can produce different effects when

laser beams interact with material, establish new standards and / or suppress old ones. The paper deals with the interaction of laser beams, in different operating modes, with the selected class of materials. Models for the description and results of interactions are discussed, damages are analyzed and an overview of current and potential fields of application and selected parallels with other beam techniques are given.

**Key words** - quantum generators, modes of operation, interactions, models, field of application.

### Different modes of operation of quantum generators as an instrument for modifications in dentistry

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# Luminescentni efekti materijala i primena

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Predrag Drobnjak<sup>6</sup>

**Apstrakt**—Luminescentne pojave se neizbežno javljaju u mnogim oblastima, od svakodnevnog života do sofisticiranih aplikacija u nauci i tehnici. Odavno su predmet izučavanja po kome se modeluju teorije i realizuju praktični sistemi.

Kako je izučavanje materijala vezano kako za sve veći broj novih materijala, tako i za veliki broj materijala sa dugom istorijom, a sa druge strane postoji više uzročnika pojave luminescencije, ova oblast je multidisciplinarna tj. stalno prisutna u kombinaciji sa mnogim naučnim disciplinama.

U ovom radu su razmatrani novi materijali razvijeni na različite načine. Neki od njih su sintetizovani od češće prisutnih hemijskih jedinjenja, a neki su vezani za retke zemlje u obliku malih primesa, čime su prema današnjem trendu vezani za lasere - kvantne generatore, kao i različite tipove senzora, detektora i slično. Promena koncentracije za koje su aktivni materijali kod kvantnih generatora vezani, direktno su donele poboljšanje koeficijenta korisnog dejstva izlaganjem aktivnih materijala snopovima elektromagnetnog zračenja, kao i izlaganjem nuklearnim zračenjima.

Razmatrani su eksperimentalni rezultati luminescentne pojave na izabranim uzorcima, na aparaturama - sistemima gde je temperatura bila parametar, a laserski izvor je bio u okviru upotrebljenih spektrofluorometarskih sistema. Sa koherentnom pobudom, mereni su luminescentni spektri. Iz dobijenih spektara određena su karakteristična vremena vezana za oblike dobijenih spektara u vremenskom domenu.

**Ključne reči**—luminescentne pojave, novi materijali, retke zemlje, promene koncentracije, pojačanje efekata.

## I. UVOD

Neće se ulaziti u daleku istoriju luminescentnih pojava, ali nije mali broj nauka u kojima je luminescencija odigrala veliku ulogu. Iako na mnogo mesta postoje definicije luminiscentnih pojava na materijalima pri raznim ekscitacijama, ipak postoji više termina i podela luminescentnih pojava prema raznim parametrima i kvantitativnim pokazateljima njenog opisa, pema oblasti primene ili vremenima karakterističnim za izazivanje emisije fotona pri raznim ekscitacijama.

Prilikom kategorizacije luminescentnih osobina materijala, oslonili smo se na razne podele vezane za materijale, kako *stare oprobane* tako i nove materijale. Praćenjem odziva materijala na razne tipove ekscitacije fluorescencije od posebnog interesa je poređenje i analiziranje raznih vrsta luminescencije u odnosu na materijale. Kroz istorijski osvrt teorijskog i eksperimentalnog razvoja uz praćenje aplikativnosti svakog pojedinog otkrivenog procesa uz razvoj novih oblasti, važan momenat bi bio u periodu nastanka kvantne elektronike.

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U ovom radu ćemo se potsetiti na jednu od definicija luminescencije. Luminescencija se definiše kao proces, gde je vreme između apsorpcije i emisije zračenja veće od trajanja perioda proizvedenog zračenja.[1] Podsećamo da se prema vremenu provedenom u pobuđenom stanju razlikuju fluorescencija i fosforescencija. Fluorescencija se vezuje za trajanje procesa za vreme dok je uzorak izložen nekom elektromagnetnom zračenju, dok fosforescencija traje i po prekidu *pobude*. Ovde bi mogla da se povede diskusija u odnosu na druge pobude luminescencije ili za modifikacije naziva i prema drugim procesima.

Teorijski se može procenjivati koncentracija aktivnih primesa koje učestvuju u luminescenciji uz sagledavanja distribucija naseljenosti u materijalima jednostavnom primenom distribucija naseljenosti u materijalu koja je tipa, ..., i uključivanjem pravila baziranih na verovatnoćama dozvoljenih i nedozvoljenih prelaza s obzirom na Paulijev princip kojim se definišu. Dozvoljeni prelazi su vezani za terminologiju prelaz singlet - singlet, sa velikom verovatnoćom za njihovo dešavanje. Pobuđena stanja su u ovim opcijama vezana za materijale koji imaju kratka vremena života, reda ns.

Materijali od potencijalnog interesa za luminescentne pojave su veoma različiti, neki od njih su interesantni u cilju istraživanja različitih efekata i karakteristika primenljivih u raznim oblastima i naučnim disciplinama. U ovom radu, korišćeni su materijali dopirani retkim zemljama u formi metalnih pločica, soli ili bronzi. Ispitivani su luminescentni efekti sa definisanim pobudama.

Složene veze među osobinama materijala u raznim fazama (agregatnim stanjima) postaju još složenije, ako se radi o jednjemima sa više elemenata. Dopirani materijali sa malo procenata retkih zemalja predstavljaju već odavno široko polje istraživanja, gde je i dobijanje koherentnih zračenja, odavno dobilo presudnu ulogu, pored istorijskih proučavanja luminescencije raznih tipova. Bez obzira na nalaženje razlika u terminologiji danas su razvijene mnoge grane u vezi sa koherentnim zračenjima, a i nuklearnim. Posebno je interesantno proučavati luminescenciju bioloških materijala. Od interesa bi sigurno bilo da se na istom materijalu, po mogućnosti ispitaju i korektno metrološki dobiju osobine sa strane luminescencije izazvanih raznim mehanizmima ekscitacije. To bi doprinelo dobijanju kompletnih odziva materijala u kvantitativnom obliku. Interesanto je polje istraživanja vezano i za razne procese podvrgavanja materijala raznim upadnim zračenjima koja podrazumevaju i promene temperature, pritiska okoline, ali i čisto mehaničkih napreznja, ozračavanja raznim snopovima

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iz mikrotalasnog područja, drugim koherentnim izvorima, akustičkim poljima. Izaazvani luminescentni spkteri i korektna obrada rezultata bi bili realni pokazatelji ponašanja materijala u vezi sa promenama tretiranja uzoraka i njihovom prethodnom modifikacijom.

Poređenje spektara dobijenih od tretiranih i netretiranih uzoraka bi bio materijal za interpretaciju daljeg trenda na intenziviranju ili gušenju luminescentnih osobina uzoraka.

U ovom radu su vršene analize na uzorcima, koji su pripremani u laboratorijskim uslovima sa raznim tehnologijama, gde su izazivana nova hemijska jedinjenja ili se samo radilo o tankim premazima pločica lakih metala sa primesama retkih zemalja.

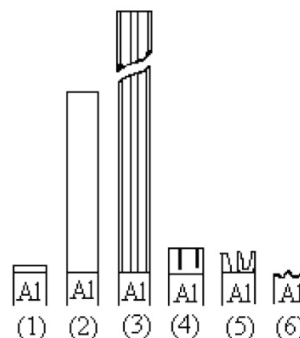
Za neke od merenja je izabrana oblast koja traži zavisnost luminescentnih osobina od temperature na kojoj se nalazi uzorak. Vršena su merenja luminescentnih svojstava sintetizovanih materijala, odmah po završetku tehnološkog procesa (tretiranja materijala i raznim parametrima ambijenta sa raznim uzročnicima luminescencije), pa su ponovo proverena luminescentna svojstva, odnosno, vršeno je ispitivanje luminescentnih svojstava prekursora i dobijenih bronzi, na temperaturama pre i posle faznih prelaza.

## II. MATERIJALI

Upotrebljeni materijali u ovom radu pripadaju različitim grupacijama, kako prema hemijskom sastavu, tako i prema obliku i načinu dobijanja. Za prvu grupu odabrani su laki metali dopirani retkim zemljama, za drugu kompresovani prah sa primesom mangana i za treću, fosfat – volframove bronzne dopirane jonima Li, Mg, Eu.

*Laki metali dopirani europijumom* - Eksperimenti su rađeni na pločicama Al dopiranim sa 10% Eu koje su anodirane kiselinama: fosfornom, sumpornom, hromnom, oksalnom i bornom. Anodizacija[2] je vršena na uzorcima koji su prvo odgrevani 5 časova na temperaturama od 150 °C, 250 °C, 350 °C i 450 °C, a onda sporo hlađeni. Za anodizaciju su pripremani na tri načina: 1) elektropolirani u HClO<sub>4</sub> / C<sub>2</sub>H<sub>5</sub>OH rastvoru u zapreminskom odnosu 1:4, propuštanjem konstantne jednosmerne struje gustine 100 mA / cm<sup>2</sup>. Nakon toga su prani u etil alkoholu i sušeni toplim vazduhom. Zaostali oksidni sloj je skidan u smeši od 20 g / l hromne kiseline i 35 ml / l koncentrovane fosforne kiseline na 80 °C za vreme od jednog minuta. 2) hemijski čišćeni potapanjem u smešu od 20 g / l hromne kiseline i 35 ml / l koncentrovane fosforne kiseline na 80 °C za vreme od pet minuta, a zatim prani u destilovanoj vodi i sušeni strujom toplog vazduha, 3) odmašćeni acetonom u ultrazvučnoj kadi. Da bi se tačno definisala površina uzorka koja se anodizira i da bi se izbegao efekat puzanja elektrolita u toku anodizacije, na gornji deo uzorka, izuzev mesta električnog kontakta, nanošena je granična maska od laka za nokte.

Generalna klasifikacija anodnih oksidnih slojeva na Al formiranih anodizacijom u galvanostatskom režimu obuhvata 6 karakterističnih vrsta oksidnih slojeva, slika 1 [3,4].



Slika 1. Struktura oksidnih slojeva formiranih u raznim elektrolitima [3,4].

(1) Površina Al prekrivena formiranim oksidnim slojem koji formiraju izolatorske anodne oksidne slojeve jonskim prevođenjem pod uticajem jakog električnog polja. Zbog visokog afiniteta Al prema kiseoniku, Al površine su uvek pokrivene prirodnim visokootpornim oksidnim slojem debljine 2 - 5 nm.

(2) Barijerni oksidni slojevi u elektrolitima koji ne rastvaraju novostvoreni oksidni sloj, debljine 700 –1000 nm. Elektroliti za njihovo stvaranje su: vodeni rastvori borne kiseline, amonijum borata, amonijum tartarata (pH 5 – 7) i neki organski elektroliti (citrična, malična i glikolna kiselina).

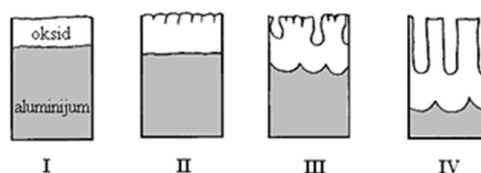
(3) Dupleks (porozni) oksidni slojevi nastali u elektrolitima koji slabo rastvaraju novostvoreni oksidni sloj su mnogo veće debljine od grupe 2. Karakterišu ih dva sloja: unutrašnji tanak, kompaktni sloj tiora 2 i spoljašnjeg, debljeg i poroznog, čija debljina ne zavisi od napona anodizacije nego od gustine struje anodizacije, vremena anodizacije, temperature i koncentracije elektrolita u odnosu na prvi. Tipični elektroliti su vodeni rastvori sumporne, fosforne, hromne i oksalne kiseline.

(4) Oksidni slojevi koji nastaju u jakim elektrolitima kao što su koncentrovana sumporna, fosforna i perhlorna kiselina i neki alkalni elektroliti. Koriste za dobijanje glatkih visoko – reflektujućih površina.

(5) Oksidni slojevi nastali u nekim mono – karbonskim kiselinama (mravlja, sirćetna), i u nekim elektrolitima sa dodatkom hlorida.

(6) Koroziono ponašanje aluminijuma u jakim alkalnim rastvorima i halidima i karakteriše se malim početnim naponom koji se dalje značajno ne menja.

Na slici 2. [1] su prikazane faze rasta poroznih oksidnih slojeva na aluminijumu



Slika 2. Faze rasta poroznih oksidnih slojeva na Al

Galvanoluminescencija (GL) ili elektroluminescencija



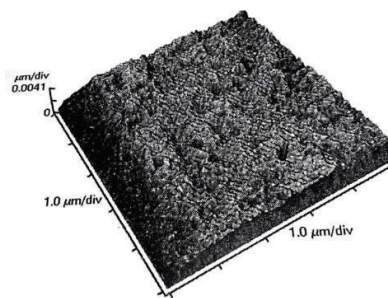
[2,3], 1883. G. I Sluganova su dosta proučavanenom sa raznih strana, te se u ovom radu nećemo baviti detaljnom analizom ovih pojava.

*Drugu grupu materijala* prikazanih u ovom radu činili su komprimovani prahovi, dobijeni u Laboratoriji za radijacionu hemiju i fiziku Instituta na nuklearne nauke "Vinča" gde su izvršena merenja na dva jedinjenja,  $\text{Li}_2\text{SnO}_3$  0,5% Mn i  $\text{LiLaP}_3\text{O}_{12}$  0,5% Mn. Eksperimentalni rezultati prikazani u ovom radu, odnose se na prvo jedinjenje. Materijali ovog tipa daju odličan odziv i veoma su pogodni za ispitivanja na velikom opsegu, ali naročito na niskim temperaturama, od 10 do 300K. Zbog veoma izražene zavisnosti luminescentnih efekata od temperature, mogu se smatrati osnovnim materijalima u ispitivanju i primeni termoluminescentnih efekata, te ih je lako primeniti u svim situacijama gde merenje temperature nije moguće primenom klasičnih metoda.

*Treća, možda najinteresantnija grupa materijala su fosfat volframove bronz dopirane jonima Li, Mg, Eu.* - Heteropoli kiseline pripadaju familiji polioksometalata sa ugrađenim heteropoli anjonima, tipa metal-kiseonik, koji predstavljaju osnovnu strukturnu jedinicu Keggin-ovog anjona.[5] Oktaedri su međusobno povezani preko atoma kiseonika i formiraju veoma stabilan i kompaktni skelet heteropolianjona. Katjoni mogu biti: vodonik, alkalni metali i drugi metalni joni. Više od dvadeset tipova struktura, uključuje četiri do četrdeset atoma metala i od jednog do devet heteroatoma pa su poznati molibdenovi, volframovi, vanadjumovi i niobijumovi heteropolianjoni Heteropoli kiseline i soli heteropoli kiseline se mogu koristiti i kao polazni materijali za dobijanje volframovih bronzi. Heteropoli kiseline su poznate više od sto godina i kao novi materijali, superjonski provodnici, interesantni su radi njihove praktične primene kao čvrstih elektrolita u gorivnim ćelijama, sensorima i displejima. One imaju važnu ulogu u industriji boja, komercijalnih katalizatora za mnoge reakcije, koriste se za dobijanje antikorozivnog pigmenta, zatim kao fotooksidacioni agensi u procesu razvijanja fotografija. Od posebnog značaja je primena u biologiji, farmaciji i medicini (antikoagulantna sredstva, antireumatici, antineoplastici, antivirolici, a postoje i pokušaji da se iskoriste u lečenju HIV infekcija). Utvrđeno je da su ove kiseline potentni inhibitori ćelijske, bakterijske i virusne DNK i RNK polimeraze. Volframove bronz imaju specifičnu strukturu koja nastaje rušenjem Keggin-ovog anjona na temperaturi od 602 °C. Ta struktura je slojevita i sastoji se od međusobno povezanih  $\text{PO}_4$  tetraedara i  $\text{WO}_6$  oktaedara. U takvoj strukturi formiraju se pentagonalni i heksagonalni otvori (šupljine, kanali) koji omogućavaju lakši transport alkalnih i zemno alkalnih metala, kojima su procesom jonske izmene u potpunosti ili delimično zamenjeni  $\text{H}^+$  joni u WPA. Pored jonske provodljivosti koja je karakteristična za ove materijale, oni pokazuju i elektronsku provodljivost zbog velike elektronske gustine uslovljene prisustvom kiseonika i elektronske konfiguracije volframa, koji ima veliku statističku težinu slobodnih d-elektrona.

Volframove bronz obuhvataju veoma veliku grupu stehiometrijskih i nestehiometrijskih jedinjenja sa empirijskom formulom  $\text{MxWO}_3$  (M je obično jedan od elemenata alkalnih metala, zemnoalkalnih metala, amonijum jon, metalni joni retkih zemalja gde x ima vrednost između 1

i 0). Prvu volframovu bronzu sintetisao je Wöhler 1823. godine [6], zbog relativno velike stabilnosti katjona volframa. Bronze karakteriše metalni sjaj, boja (od zlatno-žute do crveno-crne), dobra električna provodljivost, ili su poluprovodnici zavisno od M i vrednosti za x. Volframove bronz najčešće imaju kubnu strukturu, ali ako su promenjene x vrednosti može doći do faznih transformacija bronz, pa su tako nađene i tetragonalna i heksagonalna struktura. U slučaju fosfat volframovih bronzi je moguće dobiti strukture čiji dizajn na nivou čestice i subčestice pokazuje dobru saglasnost, između teorijskih proračunatih i dobijenih eksperimentalnih vrednosti. Slaganje eksperimentalnih i teorijskih podataka je u granicama greške od 6,7 do 19 %. Asemblirani filmovi pokazuju morfologiju površine koja odgovara u celosti morfologiji čestica iz koje se filmovi sastoje. Veoma su niske hrapavosti, reda veličine ispod 80 nm. U procesu sinteze/nanošenja tankih filmova na površinu substrata (kvarcna pločica) samoasembliranjem monokliničkih fosforvolframovih bronzi, dobijeni su filmovi dobro definisane morfologije, koju čine čestice praha sa njihovim subelementima/subčesticama, koje se raspoređuju na veoma homogen način duž cele površine substrata, slika 3.[5]

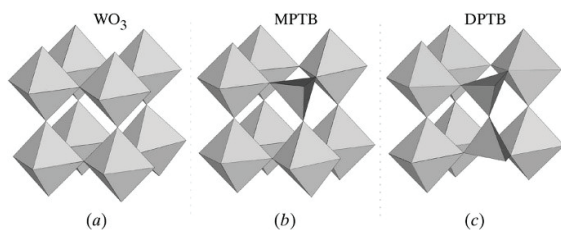


Slika 3. AFM mikrofotografija tankog filma fosforvolframove bronz na površini substrata

Merenje hrapavosti dobijenih filmova pokazuje da njihova hrapavost osciluje u uskom intervalu od nekoliko nm do najviše 80 nm, zavisno od mesta na kome se meri i od zakrivljenosti površine na tom mestu. Pri tome zakrivljenost površine prati morfologiju samih samoasembliranih čestica. Srednja vrednost je ispod 10 nm.

Volframove bronz pokazuju makroskopske kvantne osobine, na primer, elektronsku nestabilnost koja je odgovorna za pokretanje spinske gustine talasa (SGT) i gustine naelektrisanja talasa (GNT) [7 - 12].

Raspoloživi (dostupni) modeli za mehanizme nastanka GNT su neadekvatni, te su sprovedena sistematska istraživanja GNT u jedinjenjima koja pokazuju niske dimenzionalne elektrofizičke osobine, ali imaju različite sastave i strukture. Među široko korišćenim modelom objekata nalaze se fosfor volframove bronz koje sadrže perovskitni tip slojeva, građeni od  $\text{ReO}_3$  tipa, slojevi  $\text{WO}_6$  oktaedara odvojeni su slojevima koji se sastoje od monofosfatnih grupa ili difosfatnih jedinica (slika 4).[5]



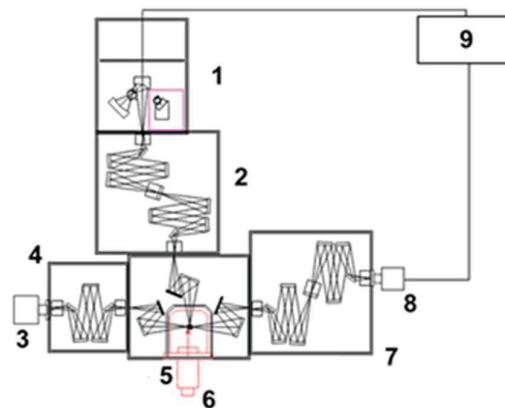
Slika 4. Zamena  $\text{WO}_6$  oktaedra u strukturi  $\text{ReO}_3$  tipa: a) klasičan perovskitni tip; b) jedan  $\text{WO}_6$  oktaedar zamenjen je sa  $\text{PO}_4$  tetraedrom i dobijaju se MPTB; c) dva susedna  $\text{WO}_6$  oktaedra zamenjena su sa  $\text{P}_2\text{O}_7$  difosfatnom grupom i dobijaju se DPTB.

Manje varijacije termodinamičkih parametara (temperature, pritiska, hemijskog potencijala, itd.) mogu izazvati značajne promene kod fizičkih osobina ovih jedinjenja. Zbog ove termodinamičke osetljivosti ovi neorganski materijali mogu poslužiti kao model objekata za ispitivanje različitih nestabilnosti.

Dobijanje složenih metalnih oksida sa karakterističnim električnim, optičkim i superprovodnim osobinama polazi od molekularnih prekursora koje je potrebno transformisati u krajnje proizvode. Trenutno razvijene tehnike dobijanja polaze iz rastvora, ali su samo aerosolni procesi uspeli da sjedine precipitaciju, termolizu i sinterovanje u jedan kontinualni proces. Došlo je do pojave velikog broja pojmova koji su imali za cilj da razdvoje aerosolne procese bilo po mehanizmu nastajanja aerosola ili po vrsti prekursorske termolitičke reakcije (razlaganje isparavanjem, sprej piroliza, termoliza aerosol, reakciono raspršivanje, ultrazvučna sprej piroliza). Pojam ultrazvučna sprej piroliza se koristi za opisivanje ovih procesa. Dobijanje složenih metalnih oksida sa karakterističnim električnim, optičkim i superprovodnim osobinama polazi od molekularnih prekursora koje je potrebno transformisati u krajnje proizvode. Od svih, do sada razvijenih tehnika, samo su aerosolni procesi uspeli da sjedine precipitaciju, termolizu i sinterovanje u jedan kontinualni proces. Tokom ultrazvučne sprej pirolize rastvor se raspršuje u sitne kapi, koje se potom uvode u reakcionu zonu i koje tada predstavljaju veliki broj mikro reaktora. U okviru svake kapi dolazi do isparavanja i precipitacije rastvorene supstance unutar kapi, sušenje, razgradnja precipitacione čestice na visokim temperaturama, formiranje mikroporozne čestice definisanog faznog sastava i na kraju, sinterovanje mikroporozne čestice u gustu česticu. Ultrazvučna sprej piroliza omogućava kompletno održanje stehiometrije na nivou kapi, pa je zbog toga izuzetno pogodna za sintezu mešovitih metalnih oksida.

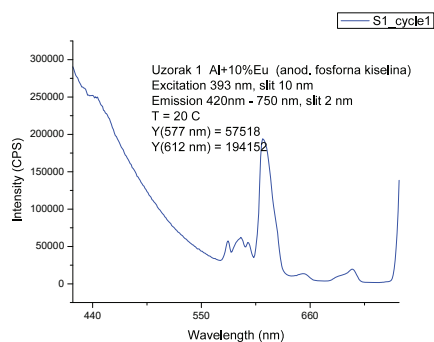
### III. EKSPERIMENT

*Prva grupa materijala* - Merenja na ovim uzorcima su vršena u Laboratoriji za Metrologiju Fizičkog fakulteta u Beogradu. Priprema uzoraka takođe je bila rezultat rada u ovoj laboratoriji. Osnovnu aparaturu činio je spektrofotometar, Slika 3, čiji glavni deo aparature je standardna volframska lampa Wi 17/G.



Slika 5. Šema spektrofotometra: 1 – Xe impulsna ili kontinualna lampa ili laser, 2 – ekscitacioni monohromator, 3 – detektor, 4 - emisioni monohromator, 5 – ćelija sa uzorkom, 6 – emisioni monohromator, 7 – detektor, 8 – računar sa softverom za obradu podataka. [13]

Dobijeni spektri predstavljaju zavisnost intenziteta populacije ekscitovanih fotona od talasne dužine na različitim temperaturama. Iz odnosa dva karakteristična maksimuma moguće je izračunati vreme života. Karakterističan spektar uzorka na temperaturi 20 oC, slika 4.

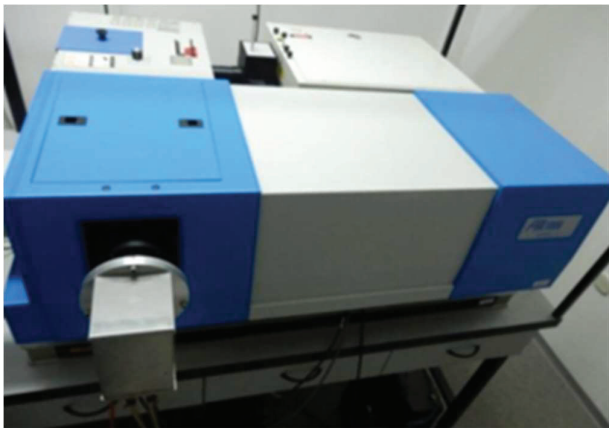


Slika 6. Spektar uzorka Al 10%Eu na 20 °C

Korišćeni su Al uzorci dimenzija 25 mm x 10 mm x 0.12 mm sa zaobljenim ivicama čime su uklonjene mehaničke deformacije koje bi mogle biti uzrok neželjene luminescencije – efekat šiljka (deformacije mogu biti uzrok jakog električnog polja (Stojadinović –teza).

*Druga grupa materijala* - Prikazani su rezultati za **Li<sub>2</sub>SnO<sub>3</sub>0,5%Mn**. Uzorci su bili izloženi koherentnoj svetlosti ekscitacione talasne dužine 532 nm, slit 200 μm, 1800 zarez. Kao izvor pobudne svetlosti korišćen je OPO laser (Optički parametarski oscilator) koji se sastoji od optičkog rezonatora i nelinearnog kristala. Postoje kontinualni i impulsni OPO laseri, s tim što je impulsna verzija lakša za izgradnju jer su za nelinearne efekte potrebne veće gustine energije pumpe. Za merenje vremena života pobuđenih stanja na temperaturama od 10 do 300 K korišćen je spektrofotometarski sistem Horiba Jobin-Yvon Model FHR-1000 sa iCCD detektorom (slika 7.) [14] Sistem sadrži i termo-električno hlađeni fotomultiplikator (hlađenje sa Peltije-ovim elementom).

Monohromator je jednostruki sa fokalnim rastojanjem od jednog metra, takođe sadrži dve difrakcione rešetke od 300 i od 1800 zarezova po milimetru, koje se mogu birati zavisno od rezolucije koju želimo postići. Veća spektralna rezolucija postiže se korišćenjem difrakcione rešetke od 1800 zarezova, ali je signal nekoliko puta slabiji.



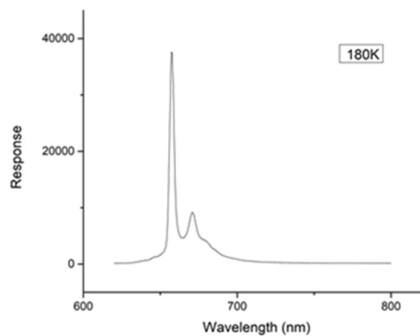
Slika 7. Spektrofluorimetar Horiba Jobin-Yvon Model FHR-1000

U delu za postavljanje uzorka postavljen je Kriostat Advance Research Systems sa zatvorenim sistemom napunjenim helijumom i omogućava postizanje minimalne temperature od 9 K (slika 8)[14]

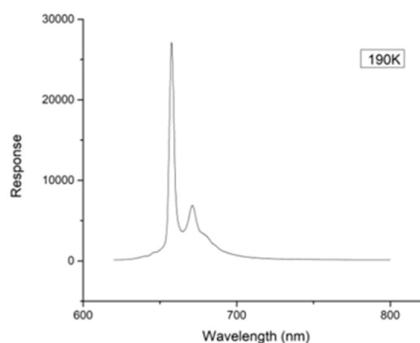


Slika 8. Krio Advance Research Systems

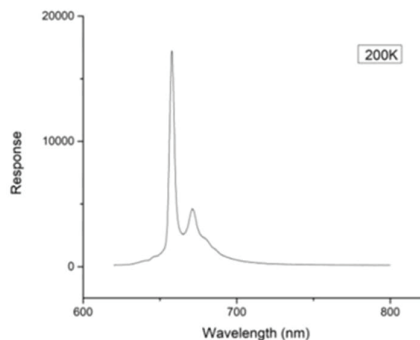
Dobijene zavisnosti prikazane su na slikama 9 , 10, 11 i 12.



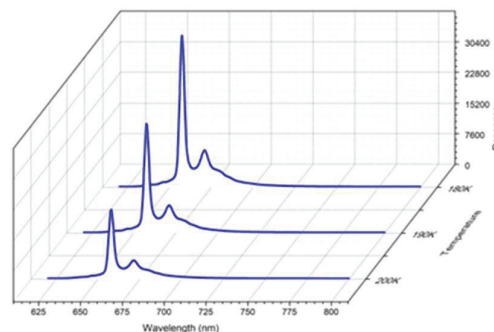
Slika 9. Zavisnost intenziteta populacije ekscitovanih fotona od talasne duzine na 180 K



Slika 10. Zavisnost intenziteta populacije ekscitovanih fotona od talasne duzine na 190 K



Slika 11. Zavisnost intenziteta populacije ekscitovanih fotona od talasne duzine na 200 K

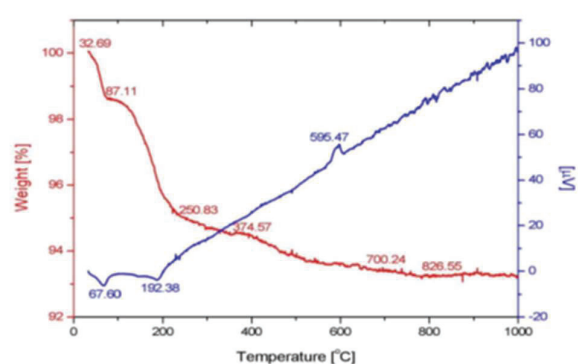


Slika 12. Zavisnost intenziteta populacije ekscitovanih fotona od talasne duzine na temperaturama 180, 190 i 200K

*Treća grupa materijala* - Eksperimentalni rezultati su korišćeni za karakterizaciju litijumove fosforvolframove bronzne koja je dobijena iz soli  $\text{Li}_3\text{PW}_{12}\text{O}_{40}\cdot n\text{H}_2\text{O}$  i  $\text{Li}_3\text{PW}_{12}\text{O}_{40}\cdot n\text{H}_2\text{O}$  u  $\text{SiO}_2$  supstratu, kao prekursorima za dobijanje bronzni u procesu kalcinacije i ultrazvučne sprej pirolize (USP).

Termijska analiza uzorka  $\text{Li}_3\text{PW}_{12}\text{O}_{40}\cdot n\text{H}_2\text{O}$  rađena je sa ciljem dobijanja podataka o termalnoj stabilnosti ove soli, a egzotermni pik na datoj temperaturi označava strukturni fazni prelaz ove soli u fosfat volframovu bronzu dopiranu sa litijumom, to jest Li-PWB. DTA i TGA krive za  $\text{Li}_3\text{PW}_{12}\text{O}_{40}\cdot n\text{H}_2\text{O}$  so od sobne temperature do  $1000\text{ }^\circ\text{C}$  prikazane su na slici 13.

Dva endotermna fazna prelaza, na  $67,6$  i  $192,4\text{ }^\circ\text{C}$ , odgovaraju procesu dehidratacije fizički vezane vode i kristalne vode, pri čemu se gubi tri odnosno osam molekula vode. To znači da ova so kristališe sa osam molekula kristalne vode. Ovaj proces je više ili manje kontinualan, pa se proces dehidratacije završava na oko  $300\text{ }^\circ\text{C}$ .



Slika 13. DTA i TGA krive za  $\text{Li}_3\text{PW}_{12}\text{O}_{40}\cdot n\text{H}_2\text{O}$  –

#### IV. DISKUSIJA

Iz dobijenih spektara dobijana su karakteristična vremena vezana za oblike dobijenih spektara u vremenskom prostoru.

Pored toga, ovaj rad predstavlja i novi prilog rasvetljavanju problema vezanih za strukturu, identifikaciju provodnih vrsta i ispitivanje faznih transformacija WPA do formiranja bronzni različitim metodama, i dizajniranja dobijenih čestica.

Ovde su prikupljeni i sistematizovani teorijski i eksperimentalni podaci o sintezi, strukturi, osobinama i primeni fosforvolframovih bronzni. Posebno se obratila pažnja na odnos između strukturnih karakteristika i elektrofizičkih osobina ovih jedinjenja. Razmatrane su i mogućnosti praktične primene fosfor volframovih bronzni.

#### V. ZAKLJUČAK

Čini se da ipak ne postoje sveobuhvatne analize gde je jedan materijal podvrgavan raznim vrstama pobude, što pogotovo važi za novorazvijane materijale. Zato bi tom tipu istraživanja trebalo posvetiti posebnu pažnju

Od posebnog interesa bi svakako bilo ispitivanje na istom materijalu, po mogućnosti da se ispitaju i korektno metrološki

dobiju osobine sa strane luminescencije izazvanih raznim mehanizmima ekscitacije. To bi doprinelo dobijanju kompletnih odziva materijala u kvantitativnom obliku.

Dalji tok istraživanja ići će u smeru ispitivanja luminescentnih svojstava prekursora i bronzne na temperaturama pre i posle faznog prelaza. Luminescentni procesi već sada nalaze široku primenu u merenjima temperature, kako na mernim instrumentima dostupnim mestima, tako i na formacijama nepristupačnim za klasične metode merenja. Pored etaloniranja metrološkom kategorizacijom obojenih organskih rastvora, sve veća primena luminescentnih efekata ogleda se u identifikaciji materijala od interesa za kulturnu baštinu [15] i dejstva nuklearnih zračenja termoluminescentnih karakteristika.

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#### ABSTRACT

Luminescent phenomena inevitably occur in many areas, from everyday life to sophisticated applications in science and technics. They have long been the subject of study, based on which theories are modeled and practical systems are implemented.

Since the study of materials is related to both an increasing number of new materials and a large number of materials with a long history, and on the other hand there are several causes of the appearance of luminescence, this field is multidisciplinary, i.e. constantly present in combination with many scientific disciplines.

In this work, new materials developed in different ways were considered. Some of them are synthesized from more commonly present chemical compounds, and some are connected to rare earths in the form of small impurities, which according to today's trend are

related to lasers - quantum generators, as well as different types of sensors, detectors etc. The change in concentration, to which active materials in quantum generators are connected, directly improved the coefficient of useful effect by exposing active materials to beams of electromagnetic radiation, as well as by exposing them to nuclear radiation.

The experimental results of the luminescent phenomenon on the selected samples, on the apparatus - systems where the temperature was a parameter, and the laser source was within the spectrofluorometric systems used, were considered. With coherent excitation, luminescence spectra were measured. From the obtained spectra, the characteristic times related to the shapes of the obtained spectra in the time domain were determined.

Keywords—luminescent phenomena, new materials, rare earths, concentration changes, enhancement of effects.

### **Luminescent effects of materials and applications**

Milanka Pećanac, Bećko Kasalica, Aleksander  
Kovačević, Zoran Nedić, Miodrag Malović, Predrag  
Drobnjak

# Efekte luminescencije, materijali i razne pobude

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## Apstrakt

Luminiscentni spektri materijala su se ne mogu izbeći u velikom delu teorijskih i praktičnih deskripcija kvantitativne i kvalitativne prirode. Pobuda luminiscentnih spektara, vreme gašenja, vreme uspona se u merenjima već odavno prate i određuju brzine, koje vladaju u nekom ansamblu. U raznim vrstama pobuda luminescencije (termoluminescencija, radioluminescencija, hemoluminescencija i dr) pojavljuju se karakteristične krive koje se na početku interpretacije i obrade signala dokazuju sa svojim analitičkim modelima. Za interpretaciju urađenih merenja ili pripremi materijala za merenja, razmatraju se eksperimentalni uslovi za dobijanje stanja materijala u zavisnosti od potrebne aparature i očekivane/dobijene zavisnosti. Pored ostalih materijala na kojima su mereni termoluminescentni spektri, razmatraju se i materijali fosfat volframove bronz dopirane retkim zemljama. Uzorci predstavljaju nekomercijalne materijale, sa postupcima dobijanja u laboratorijskim uslovima. Radi se o solima, koje su dobijene termičkim tretmanom, o solima heteropoli kiselina. Vrš se sinteza kiselina i rezultat su prahovi različitih dimenzija do većih domena (centimetar do dva, zavisno od brzine rasta kristala stajanjem na sobnoj temperaturi, kada prelaze u prah). To su jedinjenja sa velikim brojem molekula vode. U matičnom rastvoru soli heteropoli kiselina, kristali imaju 29 molekula vode, spajanjem u ambijentalnim uslovima broj molekula vode opada na 21, pa 14 itd. Najstabilnija forma je sa 6 molekula vode, a komercijalni uzorci kiselina imaju 8 – 10 molekula vode. Hemijskom analizom se vidi i u kojim temperaturnim procesima jonske izmene heteropolne kiselina sa hloridima retkih zemalja, dobijamo soli jedinjenja. Sa dobijenih termograma se određuje temperatura na koji se ruši Keginov anjon i jedinjenje prelazi u bronzu (stabilno jedinjenje).

**Ključne reči:** termoluminescencija, radioluminescencija, hemoluminescencija, spektri, karakteristične krive, fosfat – volframova bronz.

## I. UVOD

Luminescencija (od latinske reči lumen, luminis = svetlost) predstavlja pojavu hladnog zračenja svetlosti (fotona). Ove pojave ne nastaju povišenjem temperature, nego od više drugačijih uzroka, te tako postoji više vrsta luminescencija. Od toplotnog zračenja se razlikuje po načinu nastajanja, svojstvima i vremenu trajanja.

Termin hladna nije sasvim opravdan. On se koristi zato što energija kojom se podstiče (izziva) luminescencija ne potiče od pretvaranja toplotne energije u svetlosnu, već od drugog oblika energije. Luminescentna svetlost ne podleže zakonima toplotnog zračenja. Ta svetlost se može javiti vrlo intenzivno i na niskim temperaturama i u svim agregatnim stanjima.

Luminescentne spektre materijala je nemoguće izbeći u velikom delu teorijskih i praktičnih deskripcija kvalitativne i kvantitativne prirode.

Luminescencija može biti izazvana na razne načine i glavni su pobrojani u Tabeli 1:

TABELA I. NAČINI DOBIJANJA LUMINESCENCIJE I ODGOVARAJUĆI NAZIVI

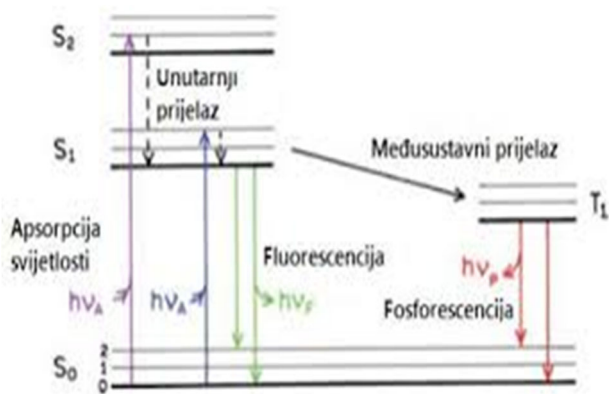
Proces	Naziv
Hemijski	Hemoluminescencija
Biološki	Bioluminescencija
Delovanje $\alpha$ i $\beta$ zaka	Radioluminescencija
Delovanjem svetlosti	Fotoluminescencija
Delovanjem električne struje	Elektroluminescencija
Delovanjem toplote	Termoluminescencija
Mrvljenjem	Triboluminescencija

Drugu podelu je moguće napraviti s obzirom na vreme trajanja sekundarnog zračenja, što je prikazano u Tabeli 2:

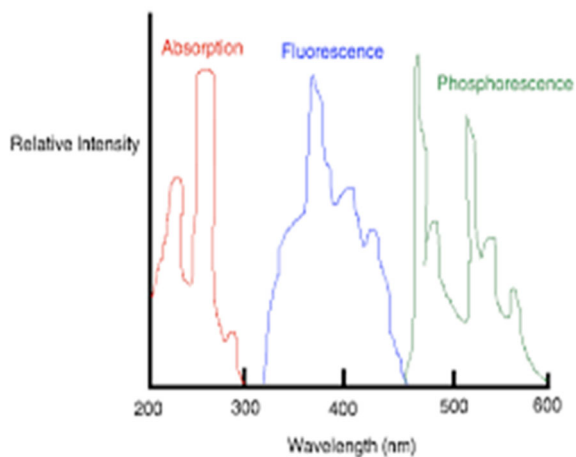
TABELA II. VRSTE LUMINESCENCIJE PREMA TRAJANJU SEKUNDARNOG ZRAČENJA

Naziv	Trajanje sekundarnog zračenja
Fluorescencija	Dok deluje pobuda
Fosforescencija	I nakon prestanka pobude

Na slici 1 (a, b, c i d)[3] dati su fluorescencija i fosforescencija – dijagrami Jablonskog.

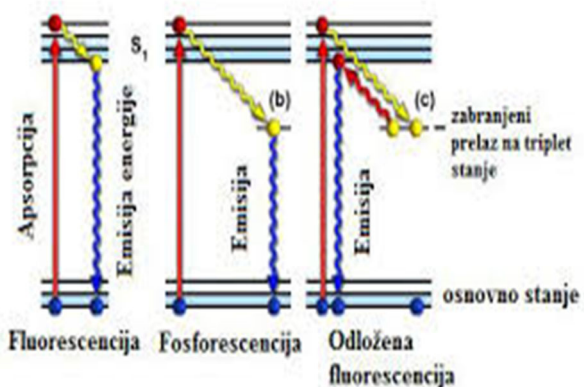


a)

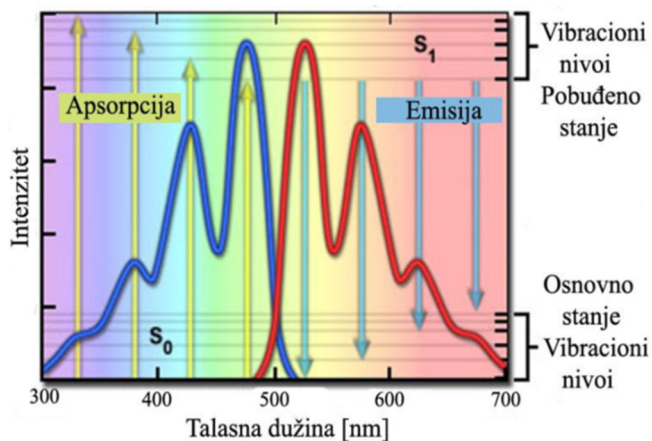


d)

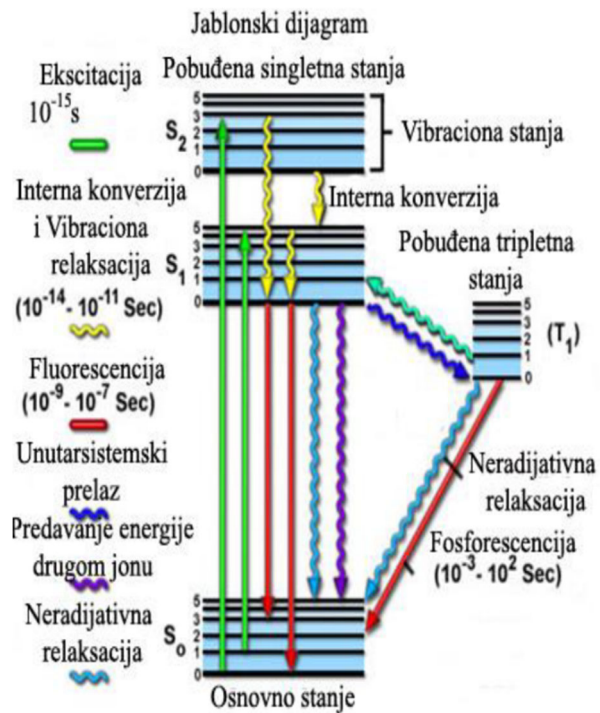
Sl. 1 Dijagrami Jablonskog



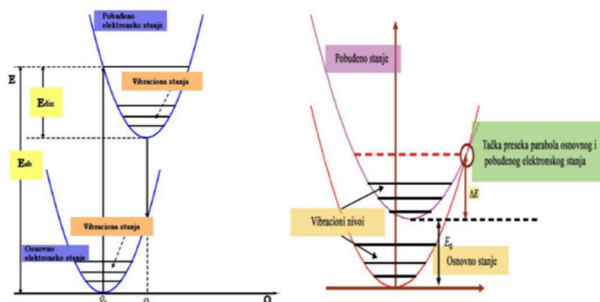
b)



Sl. 2 Šematski prikaz apsorpcionog i emisionog spektra [3]



c)



Sl. 3 Parabole osnovnog i pobudnog stanja – konfiguraciono koordinatni model luminescentnog jona[3]

## II. KARAKTERISTIČNE KRIVE U ZAVISNOSTI OD POBUDE

U raznim vrstama pobuda luminescencije (termalna, radio, hemo, foto i druge), pojavljuju se karakteristične krive (funkcije) koje se na početku interpretacije i obrade signala dokazuju svojim analitičkim modelima.

Teorijski, jedna od najčešćih linija, od kojih se druge dve krajnje linije dobijaju limesom, sa razvijenim formalizmima, su Voigtova, Lorencova i Gausova linija. Izvode se prema dinamici procesa u nekom ansamblu odabranih mikro čestica, u najširem smislu. Indikatori se odnose i na kosmos (astronomiju), dinamiku rastvora u tečnostima, sa malim ili makromolekulima. Razvijeni formalizmi u oblasti kritičnih pojava, gde su merenja mnogo teža, u slučaju magneta ili neutrona, sa razvijenim teorijama, uz merenja u „lakšim oblastima” metrologije, omogućavaju mnoge praktične odgovore i potvrde, kroz odbacivanje hipoteza u oblasti kritičnih pojava (BL, Stanli).

Osnovna matematička formula za opis linije odnosi se na različite oblasti elektromagnetnog spektra, koje se protežu od reda kHz, MHz, do širina koje su praktično izražene u Kajzeru.

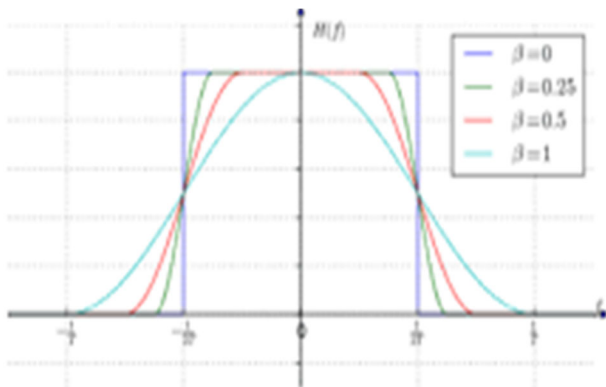
Detaljno proučavanje promena oblika i glavnih karakteristika linija, za razne oblasti, u spektroskopima različitih tipova (atomske, molekularne i dr.), praktično je savladana savremenim paketima programa i aparata, sa direktnim konstantama određenih vrsta materijala i korišćena za mnoge senzore temperature, prljavštine, proces koagulacije itd. S druge strane, prema vrsti mikročestica koje učestvuju u luminescentnim pojavama, u najširem smislu, kroz PSD tehnike (Pulse Shape Discrimination). PSD - Puls se izračunava pomoću formule:

$$f(t) = \exp\left(\frac{-(t - t_0)^2}{(2dt^2)\cos(2\pi f_0(t - t_0))}\right)$$

gde je:  $f_0$  noseća frekvencija,  $t_0=1/(2*\pi*df)$  je poluširina impulsa,  $df$  je poluširina opsega.

Spektar ovog impulsa je Gauss-ov sa centralnom frekvencijom  $f_0$  i standardnom devijacijom  $df$ .

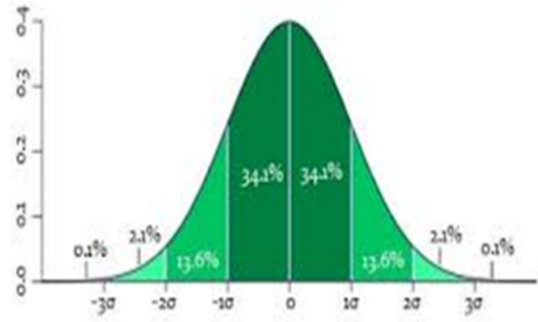
Oblikovanje impulsa je posebno važno u RF komunikaciji za uklapanje signala unutar određenog frekventnog opsega i obično se primenjuje nakon linijskog kodiranja i modulacije, Slika 4.[ ]



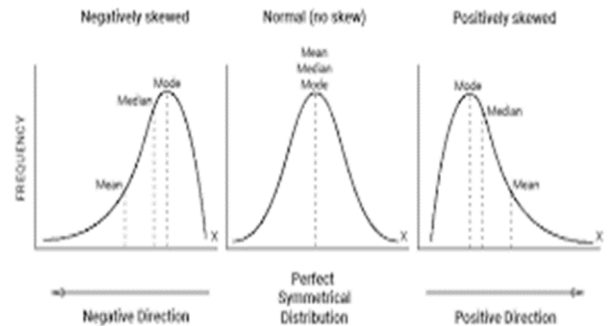
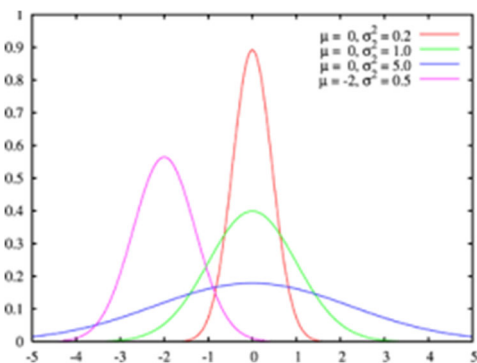
Sl. 4 PSD - oblikovanje impulsa

Gauss-ova kriva, na Slici 5. predstavljena je formulom i slikom koja dočarava idealnu raspodelu:

$$f(x) = \frac{1}{\sigma\sqrt{2\pi}} e^{-\frac{1}{2}\left(\frac{x-\mu}{\sigma}\right)^2}$$



Sl. 5 Gausova raspodela (formula i idealna raspodela)

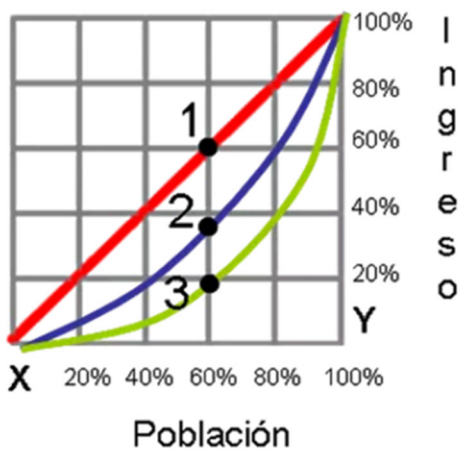


Sl. 6 Primena (snimljenji emisijski spektar i razlaganje)

Lorencova kriva (izgled), dat je na Slici 7.

Ovo je prostor za navođenje sponzora i/ili finansiranja. Ukoliko nema sponzora i/ili finansiranja obrišite ovaj 'text box'. (sponsors)





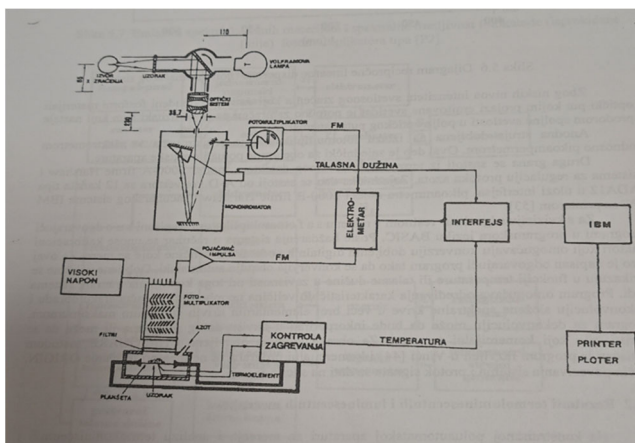
Sl. 7 Lorencova kriva

### III. RAZNI TIPOVI LUMINESCENCIJE

Pre Aparatura za merenje rendgenoluminescencije i termoluminescencije, prikazan je na Slici 8. Ovaj tip aparature omogućava:

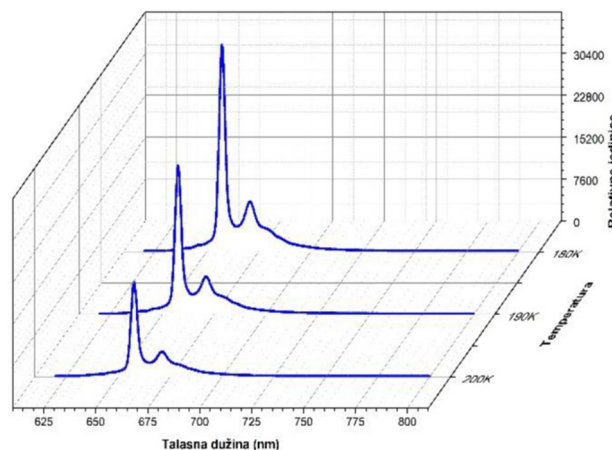
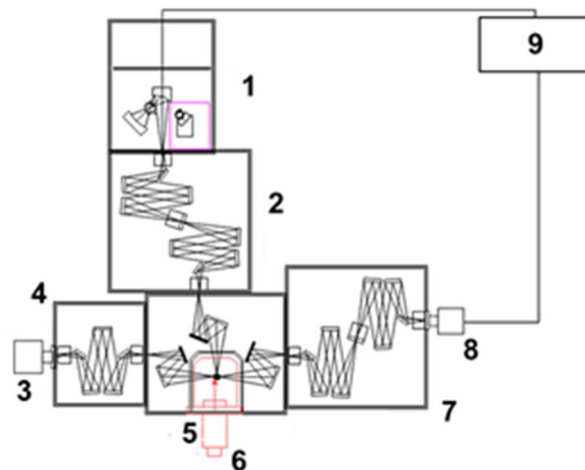
- Uporedna merenja i analizu termo i rendgenoluminescentnih spektara;
- Merenje spektara različitih polikristalnih uzoraka koji mogu da budu u prahu ili nanoseni na nosač u vidu ekrana.
- Ispitivanjem pojave luminescencije (u zavisnosti od upotrebljene pobude), moguće je ispitati postojanje lokalnih defekata u kristalnoj strukturi;

Aparatura se sastoji od dve grane: prva – za merenje spectra luminescentnog zračenja pobuđenog X ili UV zračenjem; druga – za merenje termoluminescentne (TL) krive isijavanja fosforescentnog zračenja. Izlaz iz obe grane ide na automatsko snimanje i analizu spektara.

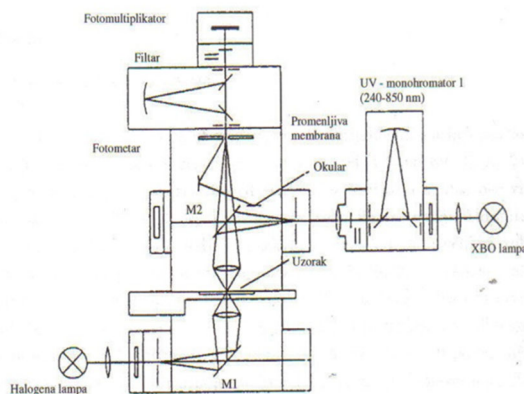


Sl. 8 Blok šema aparature za merenje rendgeno i termoluminescencije

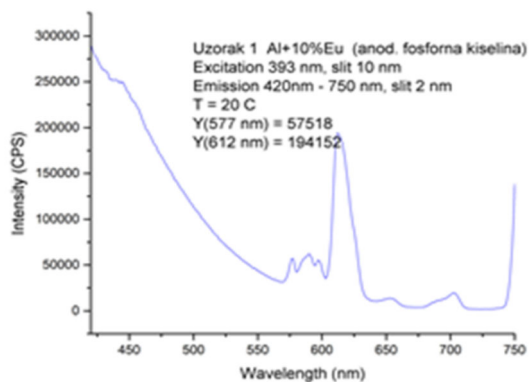
Na Slici 9 i Slici 10.[12], prikazane su aparature i dobijeni emisijski spektri u laboratorijama Fizičkog fakulteta Univerziteta u Beogradu i Instituta Vinča.



Sl. 9 Aparatura i emisijski spektar uzorka  $\text{Li}_2\text{SnO}_3$  0,5 %Mn u 3D predstavi na temperaturama 180, 190 i 200 K (Institut Vinča)



- Mikrospektrofluorometar sa promenljivim ekscitacionim i detekcionim talasnim dužinama (Carl Zeiss UMSP 80)



Sl. 10 Aparatura (mikrospektrofotometar) i emisijski spektar uzorka Al+10%Eu, anodizovano fosfornom kiselinom (Fizički fakultet Univerziteta u Beogradu)

#### IV. MATERIJALI

Za pripremu materijala za merenje i interpretaciju merenja, razmatraju se eksperimentalni uslovi za dobijanje stanja materijala u zavisnosti od potrebne aparature i očekivane dobijene zavisnosti definisanih ključnih veličina.

Pored mnogo klasičnih materijala na koje bismo se oslonili pri merenju luminescentnih spektara, u razmatranju su materijali fosfat volframove bronz dopirane retkim zemljama.

Uzorci predstavljaju nekomercijalne materijale sa postupkom dobijanja u laboratorijskim uslovima. Radi se o solima koje su dobijene termičkim tretmanom heteropoli kiselina.

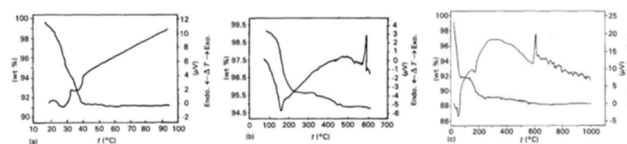
Vrši se sinteza kiselina i rezultat su prahovi čestica raznih dimenzija, do većih domena (1 do 2 cm, zavisno od brzine rasta kristala stajanjem na sobnoj temperaturi, kada prelaze u prah).

To su jedinjenja sa velikim brojem molekula vode. U matičnom rastvoru heteropolikiselina, kristali imaju 29 molekula vode. Spajanjem u ambijentalnim uslovima, broj molekula vode opada na 21, pa na 14 itd. Najstabilnija forma je sa 6 molekula vode, a komercijalni uzorci kiselina imaju 8 – 10 molekula vode.

Poznato je da, u principu, heteropoli kiselina grade više kristalohidrata sa 21, 14 i 6 molekula vode. Različite modifikacije kristalohidrata heteropoli kiselina morale bi se zapaziti pri termičkim promenama, gde se mogu pratiti metodama termičke analize (TGA i DTA). Međutim, u literaturi postoji dosta neslaganja o broju molekula vode prisutnih u pojedinim fazama kod ovih jedinjenja (29 – 31, 24 – 18, 14 – 13 i 6 -5).

Rezultati termijske analize H<sub>3</sub>PW<sub>12</sub>O<sub>40</sub>• 29H<sub>2</sub>O, od sobne temperature do 1000 °C su prikazani na Slici 11.[13]

Pri brzini odgrevanja od 10 °C /min na DTA krivoj , pojavljuju se tri pika za H<sub>3</sub>PW<sub>12</sub>O<sub>40</sub> . 29H<sub>2</sub>O. Zapažamo jedan dublet sa maksimumom na 54°C (endotermijski pik). Ovaj dublet može se razdvojiti u dve komponente, na oko 30 °C i 40 °C. Pri brzini zagrevanja od 1 °C /min, odgovaraju prelazi krive kristalohidrata od 29 do 6 molekula vode, uz međufaze kristalohidrata sa 21 i 14 molekula vode.



Sl. 11 DTA i TGA krive za H<sub>3</sub>PW<sub>12</sub>O<sub>40</sub>•29H<sub>2</sub>O: a) oblast od sobne temperature do 100 °C (1°C/min); b) oblast od 100-620 °C (5°C/min); c) oblast od sobne temperature do 1000 °C (10 °C/min)

#### Heteropoli kiselina

Zbog svojih osobina heteropoli kiselina i njima srodna jedinjenja predstavljaju veoma značajnu grupu jedinjenja, čija se primena zasniva na njihovim redoks osobinama i velikom jonskom naelektrisanju.

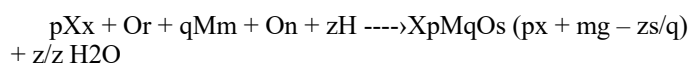
Pripadaju familiji polioksometalata sa ugrađenim heteropoli anjonima, tipa metal – kiseonik, koji predstavljaju osnovnu strukturnu jedinicu Keggin-ovog ajona.

Oktaedri su međusobno povezani preko atoma kiseonika i formiraju veoma stabilan i kompaktan skelet heteropolianjona.

Katjoni mogu biti vodonik, alkalni metali i drugi metalni joni. Više od dvadeset tipova struktura uključuje četiri do četrdeset atoma metala i od jednog do devet heteroatoma, pa su poznati molibdenovi, volframovi, vanadžijumovi i niobijumovi heteropolianjoni.

Najrasprostranjenija jedinjenja pripadaju Keggin-ovoj i Silverton-ovoj strukturi [zasićenom 12 – očlanom nizu (M:X=12)], koja ima najveći značaj za katalizu. Ovaj keggin-ov tip heteropoli kiselina je najstabilniji i najdetaljnije proučavan.

Dobro definisana Keggin-ova struktura je sastavljena od XO<sub>4</sub> tetraedra okruženog sa 12 ivica i uglom koji formiraju metal – kiseonikovi oktaedarski (MO<sub>6</sub>) ligandi, koji su kao anjoni uvek negativno naelektrisani, pri čemu njihova konkretna gustina naelektrisanja zavisi prvenstveno od elementarnog sastava i molekulske strukture. heteropoli anjoni su polimerni oksoanjoni i formiraju se kondenzacijom više od dva različita mononuklearna oksianjona u kiseljoj sredini kako je prikazano sledećom jednačinom:



Klasifikacija heteropoli jedinjenja – heteropoli jedinjenja mogu biti klasifikovana prema odnosu centralnih atoma i perifernih molibdenovih ili sličnih atoma. Jedinjenja koja imaju isti broj atoma u anjonu, obično su izomorfna i imaju slične hemijske osobine. Obično heteropolimolibdati i heteropolivolframati sadrže neprelazne elemente kao centralne atome i imaju više strukturnih sličnosti od prelaznih elemenata koji mogu biti centralni atomi. Keggin-ov tip heteropoli jedinjenja sastojinse od tri vrste struktura. Veoma je važno praviti razliku između primarne, sekundarne i tercijarne strukture koje su izgrađivački delovi kompleksa.

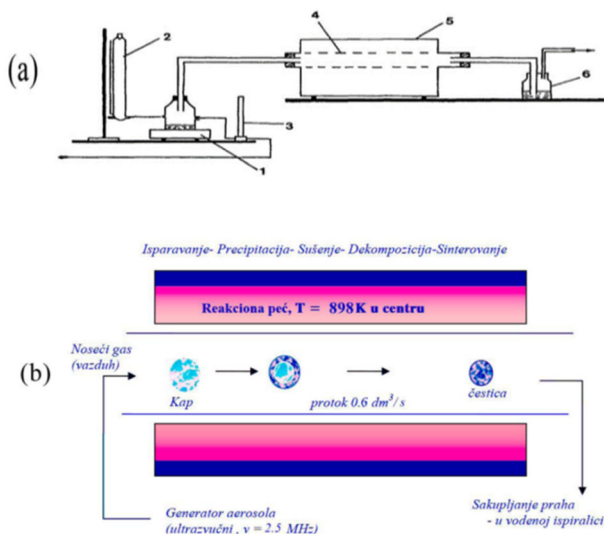
#### Dizajniranje materijala

Prva faza procesa sinteze obuhvata pripremu 20 % vodenog rastvora 12-volframfosfornog kiselina (H<sub>3</sub>PW<sub>12</sub>O<sub>40</sub>•29H<sub>2</sub>O), koja je poslužila kao prekursor za dobijanje odgovarajuće fosforvolframove bronz sprej pirolizom.

U drugoj fazi procesa sinteze, dati rastvor je podvrgnut ultrazvučnom raspršivanju ultrazvučnim atomizerom Gapusol

9001, RBI, koji se sastoji od tri transducera koji rade na frekvenciji od 2,5 MHz i njegovom očvršćavanju unutar reakcione komore, koju čini peč sa kvarcnom cevi (Heraeus Rof7/50) koja se nalazila na temperaturi od 625 °C.

Aerosol nastao tokom ultrazvučne pobude rastvora nošen je strujom vazduha (gas nosač) brzinom protoka od 0,66 dm<sup>3</sup>/s kroz reakcionu komoru u kojoj je dolazilo do očvršćavanja praha sve do posude za taloženje čestica. Vreme boravka kapljice aerosola u peći iznosilo je 20 s. Na ovaj način dobijen je prah fosforvolframove bronz. Šematski prikaz dobijanja praha metodom reakcionog raspršivanja, predstavljen je na Slici 12.[13]

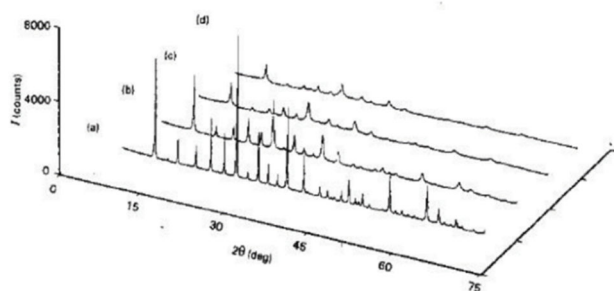


Sl. 12 Šematski proces dobijanja praha metodom reakcionog raspršivanja

(a) 1 – generator aerosola (GAPUSOL 9001 tip RBI); 2 – posuda sa konstantnim nivoom; 3 – fluometar; 4 – kvarcna cev (prečnika 40 nm); 5 – cevasta cev (HERAEUS ROF Z/50); 6 – vodena ispiralica;

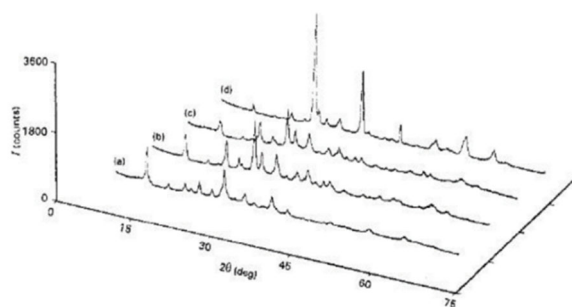
(b) kvarcna peć (proces dobijanja praha)

Difraktogrami su predstavljeni na Slici 13.i Slici 14.[13]



Sl. 13 Difraktogrami odgrejane 6-WPA faze na različitim temperaturama

a) 6-WPA na 170 °C; b) 0-WPA na 250 °C; c) 0-WPA na 350 °C; i d) 0-WPA na 400 °C.



Sl. 14 Difraktogrami 0-WPA (H3PW12O40) faze transformisane na različitim temperaturama: a) 0-WPA na 400 °C; b) D-WPA na 450 °C; c) D-WPA na 500 °C; i d) D-WPA na 550 °C.

### Primena volframovih bronz

Potencijalna primena bronz je u njihovoj ugradnji u baterije i gorive čelije, koje imaju veliku primenu u komunikaciji. Potrebne su jeftinije katode za ove sisteme, jer sadašnje katode koriste platinu ili legure platine, koje su veoma skupe i povećavaju cenu proizvoda. Poteškoće pronalaženja odgovarajućih katalizatora za odgovarajuće electrode, odnose se na činjenicu da elektronski provodnici korodiraju na potencijalima na kojima se kiseonik redukuje (1,23 V u kiselej sredini). Njihova hemijska i elektrohemijska stabilnost je veoma značajna za njihov izbor i za zamenu postojećih katalizatora koji se koriste u navedenim sistemima.

Volframova bronza zadovoljava neke od uslova koji su karakteristični za kiseoničnu elektrodu (dobra elektronska provodljivost i velika stabilnost na najnižoj pH vrednosti). Čelija je kubna i zapreminski centrirana sa atomom volframa u centru, koji je ortogonalno okružen sa šest atoma kiseonika koji se nalaze na bočnim stranama. Uglovi kocke predstavljaju osam interstacionalnih mesta (položaja) koja su dostupna, ali nisu kompletno zauzeta, sa atomima natrijuma. Volframove bronz su dobri katalizatori za elektrodukciju.

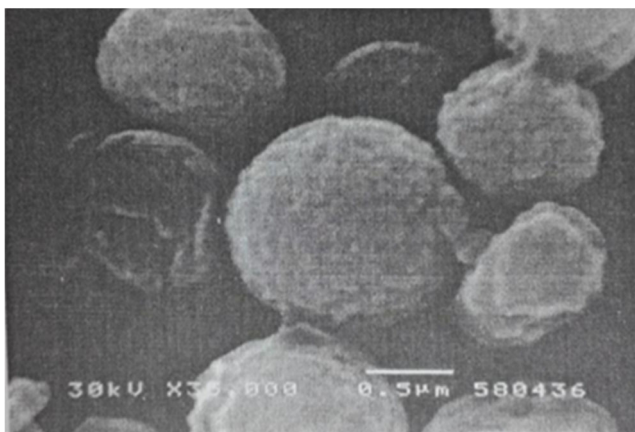
### ZAKLJUČAK

Pokazano je da je u slučaju volframovih bronz moguće dobiti strukture čiji dizajn na nivou čestice i subčestice pokazuje dobru saglasnost između teorijski proračunatih vrednosti po datom modelu i dobijenih eksperimentalnih vrednosti.

Srednja, eksperimentalno određena veličina čestica praha iznosila je 1000 nm, odnosno 1 μm, a na osnovu proračuna po datim modelima ta veličina je 1066 nm, dok je srednja veličina subčestica 45 nm, a proračunom dobijena vrednost je 55 nm.

Spektar diskretnih vrednosti čestica praha obuhvata raspodelu između 890 i 1220 nm, dominantno za eksperimentalno dobijene podatke, a 850 do 1320 nm za teorijski procenjene vrednosti. Slaganje eksperimentalnih i teorijskih podataka je u granicama greške od 6,7 do 19 %.

Asemblirani filmovi pokazuju morfologiju površine koja odgovara u celosti morfologiji čestica iz koje se sastoje filmovi. Veoma su niske hrapavosti (reda veličine ispod 80 nm), Slika 15.[13]



Sl. 15 SEM mikrofotografija čestica i subčestica fosfor volframove bronz

Dobra saglasnost između teorijski izračunatih i eksperimentalno dobijenih vrednosti, navodi nas na dalji tok istraživanja i daje jasan signal da ispitivanja luminescentnih svojstava prekusora i bronz na temperaturama pre i posle faznog prelaza, otvaraju mogućnost za dizajniranje novih materijala i njihovo podvrgavanje različitim vrstama pobude kao i tumačenje kroz veoma jasno definisanu matematičku analizu. Pored sve šireg spektra primene luminescentnih svojstava materijala, značajna je sve veća primena u identifikaciji materijala od interesa za kulturnu baštinu.

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#### ABSTRACT

Luminescent spectra of materials cannot be avoided in a large part of theoretical and practical descriptions of a quantitative and qualitative nature. Excitation of luminescent spectra, quenching time, rise time have long been monitored in measurements and determine the velocities that rule in an ensemble. In various types of luminescence excitations (thermoluminescence, radioluminescence, chemoluminescence, etc.), characteristic curves appear, which are proven with their analytical models at the beginning of signal interpretation and processing. For the interpretation of the measurements made or the preparation of the material for the measurements, the experimental conditions for obtaining the state of the material depending on the required apparatus and the expected/obtained dependence are considered. In addition to other materials on which the thermoluminescence spectra were measured, the materials of phosphate tungsten bronze doped with rare earths are also considered. The samples represent non-commercial materials, with procedures for obtaining them in laboratory conditions. These are salts obtained by heat treatment, salts of heteropoly acids. Acid synthesis is carried out and the result is powders of different dimensions up to larger domains (centimeter to two, depending on the speed of growth of crystals standing at room temperature, when they turn into powder). These are compounds with a large number of water molecules. In the parent solution of the heteropoly acid salt, the crystals have 29 water molecules, by combining in ambient conditions the number of water molecules decreases to 21, then 14, etc. The most stable form is with 6 water molecules, and commercial acid samples have 8-10 water molecules. Chemical analysis also shows at what temperature processes of ion exchange of heteropoly acid with chlorides of rare earths, we get salts of compounds. From the obtained thermograms, the temperature at which Kegin's anion breaks down and the compound turns into bronze (stable compound) is determined.

Key words: thermoluminescence, radioluminescence, chemoluminescence, spectra, characteristic curves, phosphate - tungsten bronze

# Luminiscencija kroz karakteristične krive i analitičke formulacije

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**Apstrakt**—Generalizovani prilazi karakterističnim krivama iz raspodela raznih individualnih objekata u ansamblima različite dinamike i služe da bi se došlo do glavnih karakteristika zakona, koji određuju čestice/objekte ansambala u makroskopskoj skali. Statistički prilaz je i nastao iz potreba da se nalaze „stariji” atributi, koji će opisati ansambl. Time se u neorganskom, organskom, odnosno biosvetu i dobijaju razne zavisnosti koje su opisane matricnim formalizmom sa rangom po potrebnim i dovoljnim parametrima. Procesima rasejanja raznih snopova elektromagnetskog, akustičkog ili drugog zračenja se iz mnoštva čestica, kapljica, centara rasejanja u molekularnom, atomskom i ćelijskom zapisu, dobija kategorija povezana sa makroskopskim opisima temperature, pritiska i drugih parametara, *atributa*. Tako se dobijaju kvantitativni prilazi, koji vode do različitih interpretacija o statusu, pojedinog elementa ansambla sve do ćelijskog nivoa. Drugi prilaz sledi za interpretacije i generalne postavke, koje se fituju sa Voigtovom krivom, a Gaussian i Lorenzian se pojavljuju kao dva limesa. U mnogo primena, interpretacija rezultata merenja iz raznih *klasičnih* i savremenih mernih metoda, pojavljuje se potreba za traženjem generalizacije raspodela. U ovom radu se analizira jedno od rešenja generalizovane funkcije sa fitovanjem po 4 parametra. Merenja autora u raznim laboratorijama i različitim tehnološkim postupcima iz kojih su dobijeni prahovi, kapljice, to jest objekti/članovi skupa, pojavljuju se praktični zahtevi za generalizacijom prilaza. Sa druge strane, za *stara* merenja, traženi su programi za dobijanje karakterističnih tačaka (gustina uzetih tačaka sa kontinualnih krivih, koja će što manje uticati na promenu generalne analitičke krive. Analiziran je generalniji prilaz familiji krivih, kako bi se fitovali rezultati jedne metode merenja na analogan način sa podešavanjem fitovanja putem četiri parametra.

**Ključne reči**—luminiscencija, biosvet, rasejanje, Voigtova kriva, ansambl

## I. UVOD

### *Intenzitet luminescentnog zračenja i dimenzije čestica*

Intenzitet luminescentnog zračenja i dimenzije čestica koje zrače su suštinski povezani na nekoliko načina, u zavisnosti od specifičnog tipa luminescentnog materijala, dinamike uslovnog ansambla čestica i mehanizma luminiscencije. Evo nekoliko mogućih prilaza:

- Velika površina za apsorpciju:** Kod nekih materijala, kao što su polimeri ili nanomaterijali, veće dimenzije čestica obično imaju za rezultat veću *površinu za apsorpciju zračenja raznih vrsta*. To dovodi do većeg intenziteta luminescentnog zračenja, jer se omogućava kvantitativno veća apsorpcija energije/fotona.
- Kvantni prinos luminescencije:** Kvantni prinos luminescencije kao odnos između broja emitovanih fotona i broja apsorbovanih fotona materijala, će po definiciji biti modulisan veličinom čestica i tako uticati na kvantni prinos luminescencije. Uz date pretpostavke, manje čestice mogu imati veći kvantni prinos luminescencije zbog svojih specifičnih kvantnih svojstava.
- Maksimalna talasna dužina apsorpcije:** Zavisno od veličine čestica, luminescentni materijali mogu imati različite karakteristične maksimalne talasne dužine apsorpcije. Ovaj pojam zaslužuje posebnu diskusiju, što će se u nekom modelovanju usvojiti. Promena u veličini čestica može dovesti do promene maksimalne talasne dužine apsorpcije, što ima uticaj na intenzitet luminescentnog zračenja.
- Stabilnost i distribucija veličine čestica:** Promena dimenzija čestica, može uticati na stabilnost materijala i distribuciju veličine čestica, a to se dalje prenosi na efikasnost luminescentnog procesa i intenzitet zračenja.

Ovi faktori su samo neki od mnogih, koji utiču na povezanost intenziteta luminescentnog zračenja i dimenzija čestica koje zrače. Za svaki specifični materijal i luminescentni proces, potrebno je izvršiti eksperimentalne i teorijske studije, kako bi se bolje razumeo mehanizam i povezanost pojmova. S obzirom na razne mehanizme i procese pobude, istorijski je, čini se, više rađeno na tipu pobude luminiscencije i traženju formalnih matematičkih relacija za ograničen pristup pobude i materijala.

Ispitivanje dimenzija čestica praha na osnovu luminiscencije, može biti izvedeno na nekoliko načina, u zavisnosti od specifičnih karakteristika materijala i tehnika koje se koriste. U savremene metode spadaju:

1. **Spektroskopija luminiscencije:** Tehnika uključuje merenje emitovane svetlosti iz praha posle izlaganja nekom obliku energije; u sprezi koherentne/nekoherentne pobude pojavljuje se zračenje najčešće u vidljivoj ili UV oblasti. Dimenzije čestica mogu uticati na intenzitet i formalni detektovan izlaz u frekventnom ili vremenskom domenu. Detektovan odziv materijala će imati specifične karakteristike, gde se „kriju” razne informacije o materijalu. Promene u ovim karakteristikama, mogu se koristiti za procenu dimenzija čestica.
2. **Time-Resolved Luminescence (TRL):** Razvijene aparature na današnjem stupnju elektronike i računarskih algoritama uspostavljaju vremenski profil luminiscencije posle izlaganja izabranom definisanoj energiji pobude u odgovarajućoj dinamici. Time se obezbeđuje i razlikovanje dinamika manjih i većih čestica bez traženja posebnih uslova. Analizom vremenskog profila luminiscencije, može se proceniti raspodela veličine čestica u prahu.
3. **Luminiscentni markeri:** Ako se organizuje u eksperimentu odabir pogodnih luminiscentnih markera, smatra se da je emisija markera u zavisnosti od sopstvenih dimenzija. Merna aparatura prati spektralni oblik kroz intenzitet što je baza za interpretaciju dimenzija čestica.
4. **Fluorescentna mikroskopija:** Kombinacija fluorescentnih markera sa mikroskopijom, omogućava direktno vizuelizaciju i merenje kroz ocenu dimenzija čestica praha.
5. **Upotreba kalibracionih standarda:** Kalibracioni standardi čestica etalonskih dimenzija uz luminiscentne signale izabranih uzoraka, organizovanom komparacijom, dovode do realnih dimenzija u uzorku.

Na našim, a i na inostranim prostorima su velike hemijske farmaceutske kompanije (instituti i fakulteti) imali ograničen broj aparatura za primenu statičkog rasejanja, a za izbor kapljica ili prahova sa dinamičkim rasejanjem su se više bavili poljoprivredni, veterinarski, vojni instituti i ambulante [1-9].

S obzirom da je stara kategorizacija *fine* čestice prebacila u oblast nanotehnologija, smanjene su znatno dimenzije čestica, pošto se proširio broj tehnika kojima se ciljano dobijaju druge kvantitativne vrednosti karakteristike ansambala. Ove i druge tehnike za karakterizaciju praha u području farmaceutskih proizvoda i nanomaterijala su danas proširene i odgovaraju strožijim zahtevima u smislu mernih nesigurnosti.

Ove tehnike mogu biti korisne za karakterizaciju praha u mnogim primenama, uključujući farmaciju, materijale, nanotehnologiju i mnoge druge oblasti. Međutim, važno je napomenuti da su potrebni odgovarajući instrumenti i metodologije za preciznu i pouzdanu analizu.

## II. GRAFIČKA PREDSTAVA REZULTATA MERENJA

Tokom godina, menjala se tehnička podrška i prvobitni relativno jednostavni algoritmi za dokaz standardnijih predstava spektra lasera i spektra ansambla objekata tipa praha (raspršenog), molekula, makromolekula i daljih formacija do koloida. Objekti/elementi ansambala su se proširili sa molekula u biološke ćelije, viruse. U vreme korone, tehnike su prema literaturi uključivale i neelastična rasejanja i Ramanove spektre za razlikovanje kovida-19 od drugih oblika virusa.

Grafici koji se odnose na luminiscenciju često prikazuju intenzitet emitovane svetlosti u funkciji nezavisne promenljive (vreme, talasna dužina, frekventni pomak). Oblici grafika su različite matematičke funkcije, koje predstavljaju luminiscenciju, sa specijalnim tipovima relaksacije materijala: fluorescencija, fosforescencija, elektroluminiscencija i dr. Literatura se odlikuje sa dosta različitih definicija pri poređenju strogih izražavanja sa izabranom matematičkom predstavom ili zadovoljavanja sa samo parametrima karakterističnih krivih kao što je vreme relaksacije.

Grafik fluorescencije, može prikazati intenzitet emitovane svetlosti od strane fluorofora (molekula koji emituje svetlost posle apsorpcije fotona) u zavisnosti od talasne dužine svetlosti ili od vremena, pošto je fluorofor izložen pobudi.

Grafik fosforescencije prikazuje slične podatke, ali za fosforescentne materijale koji emituju zračenje i posle prestanka pobude.

Elektroluminiscentni grafici prikazuju intenzitet svetlosti koji se emituje iz OLED-a ili drugih sličnih uređaja u zavisnosti od električnih parametara.

U ovim i sličnim slučajevima u merenju se traže grafici istog tipa za precizno definisane: pobude, vrstu materijala i time se koriste za analizu karakteristika materijala i procesa koji uključuju luminiscenciju.

## III. MATEMATIČKE FUNKCIJE

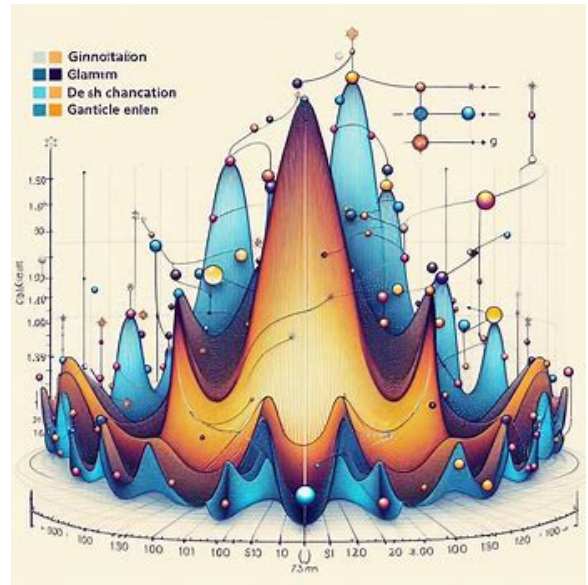
Postoji niz matematičkih modela, koji opisuju vezu i formalizme intenziteta luminescentnog zračenja, dimenzija čestica i drugih mogućih mernih parametara. Precizna matematička funkcija, koja bi opisala veze direktno će varirati zavisno od konkretnih karakteristika materijala i luminescentnog procesa, odnosno pobude. Primena odabrane metode merenja koja bi se koristila u izabranom slučaju bi se uključila sa interfejsom dela programa za interpretaciju mernih rezultata, ako se ne radi samo o zahtevu glavnih statističkih sakupljenih podataka i obrade na uobičajen način određene laboratorije. Među modelima su česte podele na empirijske, fizičke i teorijske (ova podela bi mogla da bude predmet diskusije):

**Empirijski modeli** se temelje na eksperimentalnim podacima i koriste za opisivanje veze između dimenzija čestica i intenziteta luminescentnog zračenja. Koriste se funkcije u obliku polinoma, eksponencijalne funkcije ili drugi oblici funkcija, koje se prilagođavaju eksperimentalnim podacima vezano za prethodno fitovanje mernih podataka i pretpostavljenu klasu funkcija koja se bira.

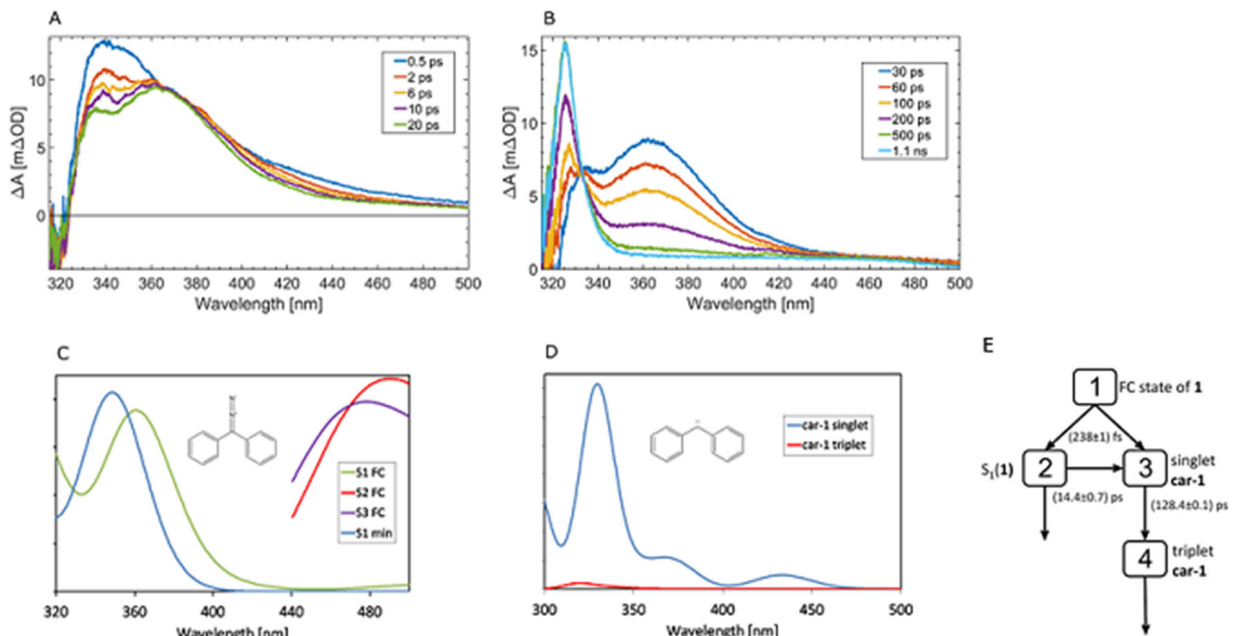
**Fizički modeli** se temelje na fundamentalnim fizičkim principima, koji opisuju interakciju svetlosti s materijalom i procese koji dovode do luminiscencije. Posebno se razvijaju modeli koji mogu uključivati kvantno-mehaničke efekte, efekte površine, strukture materijala i drugih atributa/parametara.

**Teorijski modeli** koriste analitičke koncepte i računarske simulacije kako bi se predvidelo ponašanje luminescentnih materijala na osnovu njihovih strukturnih i elektronskih svojstava. Uključeno je modelovanje energijskih nivoa u materijalu, interakcije fotona i elektrona i efekti veličine i oblika čestica zavisno od traženih izlaznih parametara opisa čestica po ustaljenoj nomenklaturi (80 parametara) [6].

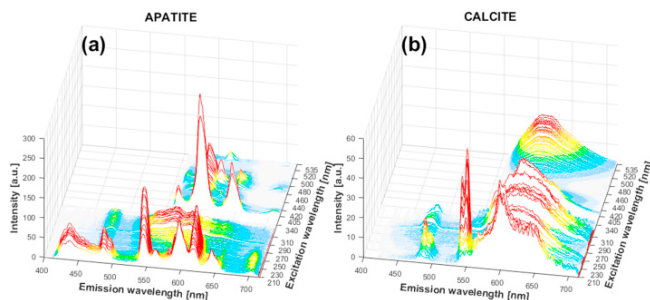
Za konkretne primene, postoji literatura, koja je isključivo vezana za određenu vrstu luminiscencije prema pobudi ili prema klasi luminiscentnih materijala. Laboratorije su razvile svoje interesne kalibracije prema svojim *budžetima* i prema praktičnim zahtevima istraživača ili massmedia primena. I uvek se sve svodi na potrebu da se dobiju korisni i praktični postupci za *friendly* upotrebu date aparature/sistema tuma, koji radi na aparaturi i priprema eksperimente. Ilustracija težine interpretacije luminiscentnih procesa i mogućih mernih rezultata je predstavljena na Sl. 1-3, od kojih su uključeni i stimulisani procesi [7].



Sl. 1 Predstava složenijeg skupa mernih podataka iz literature (3D)



Sl. 2 Drugi način predstave luminiscentnih merenja iz literature sa drugim karakterističnim krivama iz originalne literature gde su intenzitet i talasna dužina vezani za singletne i tripletne prelaze



Sl. 3 Emisije i ekscitacije luminiscencije (EEM) iz apatita (a) i kalcita (b), putem ekscitacije sa UV (210–340 nm) i vidljive svetlosti (405–535 nm) sa korakom od 1 nm. Vreme kašnjenja je bilo 50 ms, a prozor 500 ms.

#### A. Generalizovani prilaz problematici sa izborom potrebnih specifičnih funkcija

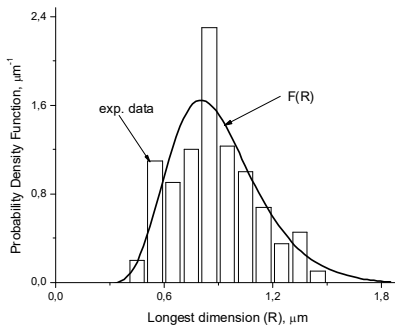
Prema postavljenim premisama u postupku generalizovanog prilaza ansamblima objekata koji mogu biti neograničenih atributa (prahovi, mikroorganizmi, kapljice,...) koji se odlikuju specifičnim oblicima: uniformnih, anizotropnih, višeslojnih struktura, koji se za praktičnu upotrebu i danas dalje razvijaju. Konkretno, može se pretpostaviti da je element skupa pravilna/nepravilna matematička aproksimacija sfere ili kugle, cilindar ili valjak i dalje praviti kombinacije sa spomenutim pretpostavkama o uniformnosti zahvaćene zapremine, višeslojnosti i sl. [4]. U osnovi razvijanja *fitovanja* pošlo se od

Gausove i Log-normalne raspodele, pošto intuitivno ne radimo sa malim brojem čestica. Iz niza predviđenih matematičkih koraka od generalizovane funkcije do LG4.

Nova generalizovana logaritamska jednačina je definisana sa četiri parametra, nazvana LG4. Formulirana je i predložena za opisivanje distribucije veličine i oblika čestica. Za poseban izbor parametara LG4 je redukovana na logaritamsku distribuciju LG2 definisanu sa dva parametra, pa se omogućava odabir srednje vrednosti raspodele kao parametra i ona se kao parametar pojavljuje eksplicitno u funkciji distribucije. LG2 je predložen kao funkcija gustine verovatnoće za veličinu i oblik raspodele čestica („četvrto stanje materije“ prema Hejvudovoj definiciji) kao, na primer, metalne i keramičke čestice koje privlače veliku pažnju javnosti, kao novi materijali visokih performansi za magnetne materijale, koji obećavaju, a zatim hemijski katalizatori, materijali koji promovišu sinterovanje, senzori, itd. Oblik i sličnost čestica za neke svrhe su veoma važni. Ukupna zapremina, ukupna površina ili bilo koje drugo korisno svojstvo uzorka može biti vezano za oblik i veličinu pojedinih čestica. Ako su sve čestice geometrijski slične, sav naknadni tretman je znatno pojednostavljen u pogledu oblika. Uvođenjem parametra oblika čestice, generalizovana sličnost i eliptički faktor sklopa čestica, primena LG2 za proučavanje raspodele oblika projektovane čestice sprovodi se formalizam u ovom radu..

Srednja vrednost, kao parametar veličine, pojavljuje se eksplicitno distribuciji LG2. Parametri se određuju fitovanjem empirijske raspodele različitih projektovanih čestica (najduže i najkraće dimenziji površina čestica). LG2 jednačina je usvojena kao model za distribuciju oblika čestice. Međutim, izvedeno modelovanje može biti korisno za različite primene bez obzira na vrste čestica. Parametar oblika čestice je uveden i zatim uopštena sličnost čime su definisani eliptički faktori sklopa čestica. Primenom LG2 distribucije dobijena je distribucija najduže i najkraće dimenzije parametara oblika. Metoda za razmatranje sličnosti čestica je razvijena. Postupak ove metode je jednostavan, lak i brz, ali metoda je ograničenog opsega primene i može biti od koristi za opis eliptične čestice.

Na osnovu prethodnih formalizama, izvršeno je fitovanje u definisanu krivu F(R) koja je fitovana prema eksperimentalnoj raspodeli najduže dimenzije čestice (Slika 4)



Slika 4. Kriva F(R) fitovana prema eksperimentalnoj raspodeli najduže dimenzije čestice

$$f_{LG4(p,x_{1p},x_{2p},\zeta_p)} = f_p(x) = \frac{x^p \exp \left[ -\frac{(\ln x - \ln x_{1p})(\ln x - \ln x_{2p})}{2\zeta_p^2} \right]}{x_{1p}^{p+1} \zeta_p \sqrt{2\pi} \exp \left\{ \frac{[2(p+1)\zeta_p^2 - \ln(x_{1p}/x_{2p})]^2}{8\zeta_p^2} \right\}}$$

$$\langle x_p \rangle = x f_p(x) dx = \frac{\int_0^\infty x^{p+1} \exp \left[ -\frac{(\ln x - \ln x_{1p})(\ln x - \ln x_{2p})}{2\zeta_p^2} \right]}{x_{1p}^{p+1} \zeta_p \sqrt{2\pi} \exp \left\{ \frac{[2(p+1)\zeta_p^2 - \ln(x_{1p}/x_{2p})]^2}{8\zeta_p^2} \right\}} dx = \sqrt{x_{1p} x_{2p}} \exp \left( \frac{(2p+3)\zeta_p^2}{2} \right)$$

$$\langle x^2 \rangle = \int_0^\infty x^2 f_p(x) dx = x_{1p} x_{2p} \exp [2(p+2)\zeta_p^2]$$

$$\langle \sigma^2 \rangle = \int_0^\infty (x - \bar{x})^2 f_p(x) dx$$

$$x_g = \exp \left( \int_0^\infty (\ln x) f_p(x) dx \right) = \sqrt{x_{1p} x_{2p}} \exp [(p+1)\zeta_p^2]$$

$$f(x) = \frac{x^{-\frac{3}{2}} \exp \left[ -\frac{(\ln x - \ln x_1)(\ln x - \ln(x^2/x_1))}{2\zeta^2} \right]}{x_1^{-\frac{1}{2}} \zeta \sqrt{2\pi} \exp \left\{ \frac{[\zeta^2 - \ln(x_1/x_2)]^2}{8\zeta^2} \right\}}$$

$$f(x) = \frac{x^{-3/2} \exp \left[ -\frac{(\ln x - \ln x_1)(\ln x - \ln x_2)}{2\zeta^2} \right]}{x_1^{-1/2} \zeta \sqrt{2\pi} \exp \left\{ \frac{[\zeta^2 + \ln(x_1/x_2)]^2}{8\zeta^2} \right\}} \quad i$$

$$\bar{x} = \sqrt{x_1 x_2}$$

Za slučaj generalizovane funkcije LG4 [1] izveden je detaljno potreban formalizam do dobijanja raspodele čestica za slučaj magnetnih čestica NdFeB, materijal koji je vrlo važan za gabarite savremenih magneta [8,9].

Pošto je iskorišćen prilaz sa generalizovanom funkcijom i deo primene sproveden sa LG4, odnosno LG2 i povezan sa eksperimentalnom krivom, vratimo se na teorijski put putem kernela. On se uvodi za rešavanje problematike koja bi povezala luminiscenciju sa raspodelom čestica koje luminisciraju.

Ako postoji f(x) zasnovano na eksperimentalnim merenjima i ako znamo funkciju transformacije parametara K(x,y) (kernel), onda se teorijski dobija funkcija g(y), koja može da predstavlja funkciju raspodele čestica. Uključena je i funkcija greške ε(x),

$$f(x) = \int K(x,y)g(y)dy + \varepsilon(x)$$

Za slučaj da je upotrebljena merna angularna raspodela za procese Mieovog rasejanja [3,4]

$$f(x) = I_{sca}(\theta)$$

Raspodela dimenzija čestica se u principu može predstaviti sledećim raspodelama: Rosin-Rammler, Nukiyama-Tanasawa,



Log-normalna, što se može direktno povezati sa razvijenom LG4 koja je definisana sa 4 parametra, na početku ovih planiranih operacija, sa varijacijama LG3, LG2.

#### Zaključak

Poznavanje dinamike procesa za slučaj velikih ansambala je složena problematika i sa eksperimentalne i sa teorijske strane. Izazivanjem procesa emisije fotona (spontano ili stimulirano) je jedan od načina za povezivanje formalizama adekvatnih spektroskopija i planiranje potrebnih osobina novih materijala. U ovom radu je na osnovu predložene generalizovane funkcije raspodele prikazan postupak i rezultati za magnetne materijale

Luminiscentne pojave imaju više različitih načina pobude i čini se da bi bilo interesantno povezati različite krive koje se pojavljuju u literaturi sa generalnijom bazom funkcija i po mogućstvu ih sa gledišta izbora sa fitovanjem sa četiri parametra, kao što je skicirano u ovom radu predstaviti za širu upotrebu.

Različite krive (matematičke formule) se pojavljuju i u savremenim knjigama posvećenim luminiscenciji, i u fitovanju za date familije luminiscentnih materijala .

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#### ABSTRACT

Generalized approaches to characteristic curves from the distribution of various individual objects in ensembles of different dynamics and serve to arrive at the main characteristics of the law, which determine the particles/objects of the ensemble on a macroscopic scale. The statistical approach was born out of the need to find "older" attributes that will describe the ensemble. In this way, various dependencies are obtained in the inorganic, organic, i.e. bio world, which are described by the matrix formalism with the rank according to the necessary and sufficient parameters. Through the scattering processes of various beams of electromagnetic, acoustic or other radiation, a category associated with macroscopic descriptions of temperature, pressure and other parameters and attributes is obtained from the multitude of particles, droplets, scattering centers in the molecular, atomic and cellular record. Thus, quantitative approaches are obtained, which lead to different interpretations of the status of a single element of the ensemble down to the cellular level. Another approach follows for interpretations and general propositions, which are fitted with the Voigt curve, and Gaussian and Lorentzian appear as two limes. In many applications, the interpretation of measurement results from various classical and modern measurement methods, there is a need to search for the generalization of distributions. In this paper, one of the solutions of the generalized function with 4-parameter fitting is analyzed. The author's measurements in various laboratories and various technological procedures from which powders, droplets, that is, objects/members of the set were obtained, show practical requirements for the generalization of the approach. On the other hand, for old measurements, programs were requested for obtaining characteristic points (the density of points taken from continuous curves, which will affect the change of the general analytical curve as little as possible). A more general approach to the family of curves was analyzed, in order to fit the results of one method of measurement on analog mode with fit adjustment via four parameters.

*Key words*—luminescence, bio world, scattering, Voigt curve, ensemble

**Luminescence through characteristic curves and analytical formulations**



Програм и Зборник апстраката са Прве  
националне конференције

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херитологији и новим  
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16. март 2019.  
Београд

**Зборник апстраката са Прве националне конференције  
Методолошка истраживања у херитологији и новим технологијама**

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## САДРЖАЈ

1. Предавање по позиву Сања Петронић, Методологија испитивања чишћења папира из XIX века применом ласерског снопа.....	8
2. Љиљана Никшић, Методолошки прилаз у стварању концепта међународне мултидисциплинарне научне изложбе „Јасеновац – право на незаборав“, УН 2018.....	10
3. Зоран Карастојковић, Павле Карастојковић, Сузана Полић, Материјали за израду иконостаса.....	12
4. Владимир Симић, Методологија изградње звучног архива као ресурса херитолошких вредности.....	14
5. Милеса Срећковић, Сања Јевтић, Драган Јевтић, Слађана Пантелић, Оптички каблови и комуникације у железници 20. и 21. века.....	15
6. Светлана Пелемиш, Владан Мирјанић, Срђан Вуковић, Nanotechnology and biomaterials in dentistry.....	17
7. Анђелка Милосављевић, Димитрије Маљевић, Методолошки приступ испитивањима обрада аустенитних материјала неконвенционалним технологијама.....	18
8. Милош Ђурић, Exercises in Bad Style: The Case of Transgressing Quantum Gravity Hermeneutics.....	19
9. Братислав Иричанин, Методолошки путеви транспоновања у делима Софије Ковалејевске и Данила Киша: једна могућа перспектива.....	21
10. Лука Јовановић, Кристина Мицошки, Херитолошки метод и економска историја: два догађаја из Краљевине Србије и Краљевине СХС.....	22
11. Вељко Зарубица, Катарина Зарубица, Савремени методолошки прилаз оцени мерне несигурности у разним методама мерења са конкретним примерима од значаја за културну баштину.....	23
12. Александра Стевановић, Документарни филм „Три логоса“ као методолошки оглед у разматрању речника технологије.....	25
13. Станко Остојић, Најзаступљеније математичке методе у постављању модела од интереса за културну баштину.....	27
14. Жељка Томић, Методолошко-предметни наслов, Хартлијева класификација и индустријско наслеђе.....	29
15. Анастасија Вигњевић, Екстензија, промишљање и артикулација херитолошких проблема.....	30
16. Матија Срећковић, Методологија у прихватању фундаменталних /егзактних метода и статистички прилаз као основа моделовања од значаја за бионауке и економију.....	31
17. Милена Давидовић, Квазирасподеле од интереса у дигиталној обради података.....	33
18. Сања Јевтић, Драган Јевтић, Методологија у индустријском наслеђу везаном за железницу.....	34
19. Сузана Полић, Универзалност херменеутичког феномена и херитологија.....	36
20. Ведран Ибрахимовић, Методологија примене микроталасних мерења и паралела са оптичким подручјем.....	37
21. Братислав Иричанин, La Place и Saint Simone: методолошке опсервације, о каузалности и метонимији.....	38

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22. Зоран Стевић, Сања Петронић, Драган Миленковић, Наташа Томић, Методологија дијагностике и заштите културног наслеђа применом нових технологија .....	39
23. Магдалена Драговић, Светлана Чичевић, Александар Чучаковић, Александар Трифуновић, Алина Дута, Нови приступи и примена савремених технологија у едукацији инжењера: практични примери из херитологије.....	40
24. Александер Ковачевић, Препознавање облика неуронском мрежом у области заштите културног наслеђа.....	42
25. Светлана Рогановић, Методологија компаративних истраживања естетика Биомбо стила и дадаистичког израза Sophie Henriette Gertrude Taeuber-Arp.....	44
26. Милеса Срећковић, Станко Остојић, Лазар Кричак, Слободан Бојанић, Електрооптички, електромагнетни, акустооптички, пиезоелектрични и други линеарни и нелинеарни процеси у дијагностицирању материјала или у системима мерења .....	45
27. Анђелка Милосављевић, Микроструктурне промене ласером заварене суперлегууре никла М-252 .....	47
28. Софија Бекрић, Драгољуб Бекрић, Наслеђе Великог рата и аквизиције у Завичајном музеју Жупе – Александровац .....	48
29. Драган Миленковић, Дијагностика стања и заштита културног наслеђа применом инфрацрвене термографије.....	49
30. Магдалена Драговић, Зоран Стевић, Борис Косић, Александар Чучаковић, Марко Радосављевић, Методе обраде дигиталног модела објекта културне баштине за сврхе реконструкције.....	50
31. Сања Петронић, Зоран Стевић, Алекса Маљевић, Важност примене ласера у чишћењу и одржавању херитолошки значајних тканина од памука, тексаса и свиле.....	52
32. Горан Трбојевић, Бард критичке педагогије и методологија академског индустријског комплекса.....	53
33. Никола Славковић, Перцепција асинхроног мотора и приказ Николе Тесле у делу Леса Драјсдејла.....	54
34. Зоран Фидановски, Бојана Фидановски, Биљана Фидановски, Софтвер од интереса за културну баштину и другу савремену проблематику .....	55
35. Драган Павловић, Јован Цветић, Утицај руских архитеката на изградњу Београда.....	57
36. Љубинко Јањушевић, Александар Седмак, Михаило Петровић и методологија изабраних проналазака у индустријској и научној баштини Србије .....	58
37. Томислав Рајић, Савремени software и методологија његове примене у енергетици мерењима.....	59
38. Ђорђе Ђорђевић, Ивана Ћировић, Гордана Ђукановић Основи заснивања методологије дефинисања идентификационе карте уметничког дела - параметризовањем његових карактеристичних геометријских-морфогених својстава, применом поступака микрофотограмetriје и математичке статистике/вероватноће.....	61
39. Бранислав Давидовић, Моделовање у простору ласерске технике у циљу модела за специфичне примене.....	65
40. Зоран Лазаревић, Милош Јечменица, Богдан Брковић, Савремени проблеми у пројектовању и даљинском управљању енергетски ефикасне микромреже на подручју Манастира Хиландар .....	67
41. Милеса Срећковић, Станко Остојић, Милена Давидовић, Функције корелације, метода момената и кумуланата, дефиниција и практичне примене у савременим	

статистичким опсервацијама .....	69
42. Анђелка Милосављевић, Утицај легирајућих елемената током ласерске обраде високотемпературних материјала .....	71
43. Вишеслава Рајковић, Студије случаја кроз призму науке о материјалима .....	72
44. Катарина Чолић, Филип Вучетић, Примена савремених оптичких метода мерења у биомедицини .....	73
45. Силвана Б. Димитријевић, Стеван П. Димитријевић, Зоран Стевић, Заштита од тамњења металних предмета културне баштине.....	74
46. Сузана Полић, Интегритет и безбедност културног наслеђа у светлу новијих методолошких и технолошких истраживања .....	75
47. Сања Петронић, Борис Рајчић, Димитрије Маљевић, Интеракција ласера са бакром и његовим легурама са аспекта херитологије.....	76
48. Катарина Чолић, Побољшање механичких карактеристика биоматеријала применом савремених ласерских метода.....	77
49. Марија Крстева, Превођење техничких терминолошких конструкција са енглеског на македонски језик.....	78
50. Предраг Јованић, Прелиминарна грађа и прилог за рестаурацију и конзервацију манастира Раковица.....	79



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технологијама“



## ПРЕПОЗНАВАЊЕ ОБЛИКА НЕУРОНСКОМ МРЕЖОМ У ОБЛАСТИ ЗАШТИТЕ КУЛТУРНОГ НАСЛЕЂА

### NEURAL NETWORK PATTERN RECOGNITION IN THE AREA OF CULTURAL HERITAGE PROTECTION

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Употреба алгоритама неуронских мрежа је све више заступљена у области заштите, конзервације и ресторације културних добара. Неуронске мреже имају предност уколико се захтева обрада великог броја узорака и уколико није могуће установити аналитичке зависности између објеката.

Формирањем база слика делова архитектонских објеката се добија основа за тренирање неуронских мрежа чиме се омогућава препознавање нових узорака и њихова класификација. Конволуциона неуронска мрежа дубоког учења је обучавана на примерима (преко 10000 слика) као што су: олтар, апсида, звоник, купола (изнутра и споља), витражи и брод. Неколико примера из јавно доступне базе података је приказано на сл. 1.



Сл. 1. Део примера на којима је обучавана неуронска мрежа за препознавање слика: купола споља (горе) и витражи (доле).

Ова методологија је показала завидну тачност и над валидационим и над испитујућим скупом (95% односно 94,6%).

У другом случају је неуронска мрежа обучавана на спектрима који су добијени LIBS (спектроскопија ласерски индукованог пробоја) техником. На основу 93 спектра је добијена тачност од 87,5%. Прелиминарна анализа тешко доступних или делимично извађених узорака је понекад неопходна да би се на терену донеле брзе одлуке о даљем наставку радова. На овај начин се олакшава идентификација области вредне истраживања.

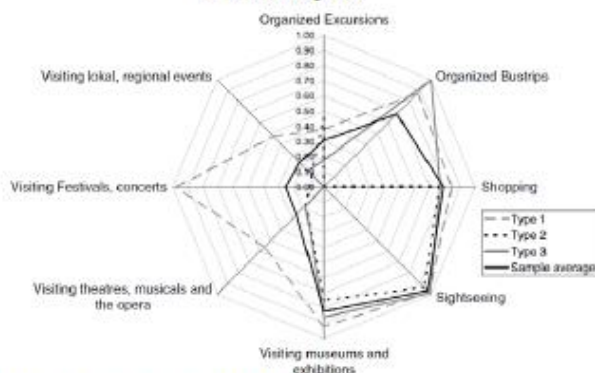
Неуронске мреже се могу употребити и са аспекта друштвених наука. У једном истраживању се користи тополошка мрежа, као ненадгледана неуронска мрежа која трага за хомогеним групама у оквиру датог скупа података. На примеру туристичког тржишта Аустрије су туристи класификовани у девет типова. Сегментација је извршена на основу Националног истраживања гостију Аустрије обављеног лета 1997 и зиме 1997/1998, где је интервјуисано преко 10000 гостију, а територија је подељена на 49 региона. Скуп података је одабран од скоро 2500 туриста који су класификовани у девет типова на основу својих активности за време туристичке посете. За ових девет кластера су дефинисане описне и нумеричке променљиве, које су праћене. Пример за три кластера је приказан на сл. 2.

Уочено је да тип дефинисан као „појединачни истраживач културе“ најбоље реагује на понуде које се јављају у току зимске сезоне.





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Сл. 2. Профили променљивих за три типа [4].

У истраживању обављеном за време изложбе „Лепота или истина“ (it. “Il bello o il vero”) [5] је понашање посетилаца моделовано неуронском мрежом. Сваки од посетилаца је представљен неуроном (чвором неуронске мреже) по најједноставнијем моделу биолошког неурона „интеграције и акције“ који укључује и временску компоненту. Синаптичка тежина између два чвора одговарају степену свиђања у односу на поједини објекат који деле два посетиоца. Праћена је узајамна комуникација преко друштвених мрежа, као и појединачне активности преко мобилне апликације (сл. 3).



Сл. 3. Главни екран мобилне апликације за оцену утиска о појединачном уметничком делу на изложби „Лепота или истина“ [5].

Анализирано је како информација дељена између посетилаца утиче на остале учеснике унутар круга друштвене мреже са темом културног наслеђа. Биолошка морфологија мреже је мапирана и дубље су истражени одговарајући параметри: ниво друштвености и матрица пријатељства. Овај систем је управљао великом количином корисничких података, као и тема апликација, као што су профилисање по контексту и повратна спрега, и системи препоручивања.

Показује се да је употреба неуронских мрежа оправдана, где год има велики број података за које нису јасно дефинисане релације у деловима области заштите, конзервације и рестаурације културних добара.

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**ПРЕДАВАЊА ПО ПОЗИВУ**

1.	Сања Петровић, Зоран Стевић	Методологија усвајања знања о ласерском чишћењу папира на основу изабраних руских научних извора	1-3
2.	Саша Милић	5G мрежа у светлу културне баштине нове индустријске револуције и интернета ствари	4-7

**СЕКЦИЈА 1**

3.	Зоран Здравковић, Јулијана Мирчевски	Дигитализација и конзервација културне баштине и историјског наслеђа	8-11
4.	Томислав Рајић	Примена софтверског алата XGSLab за процену нејонизујућег зрачења у близини далековода	12-14
5.	Милеса Срећковић, Александер Ковачевић	Ретроспектива методолошких приступа примене ласерских техника у херитологији: изабране студије	15-17
6.	Анђелка Милосављевић	Материја у методолошком приступу Анђија Bergsona – опсервација са становишта технологије материјала	18
7.	Станко Остојић	Методологија у постављању модела за припрему конзервације/рестаурације објеката индустријске и нематеријалне културне баштине	19-20
8.	Светлана Пелемиш	Нанотехнолошки материјали у културној баштини	21

**СЕКЦИЈА 2**

9.	Жељка Томић	Баштина и феномен светлости у делу Bele Hamvaša „Невидљиво збивање“: методологија истраживања	22-23
10.	Милош Ђурић	Констрактивна анализа енглеско-француских термина регистра конзервације и херитологије	24-25
11.	Анастасија Вигњевећ, Антон Ето	Изоловање нејасних значења по методи Rolana Barta и рефлексије	26-28
12.	Драган Павловић, Милеса Срећковић	Историја астрономске опсерваторије у Београду	29-31
13.	Слободан Бојанић	Хеуристика и научни рад на пољу техничких наука	32-33

**СЕКЦИЈА 3**

14.	Никола Поповић, Јулијана Мирчевски	Реконструкција и конзервација старих и избледелих докумената у државној управи	34-37
15.	Милош Ђурић	Метафразе у преводима текстова из области техничко-технолошких наука: методолошки изазови	38-39
16.	Светлана Рогановић	Интеракција етике, естетике и политике, као методолошки концепт у интердисциплинарности Grizelde Polok	40-41
17.	Горан Трбојевић	Методолошки поглед на употребу антитезе у истраживањима Stivena Grinblata о ренесансној култури	42-43

18.	Братислав Ирчанин	Филозофија у технолошком добу, дело Hansa Lenka, педесет година касније	44-46
-----	-------------------	-------------------------------------------------------------------------	-------

#### СЕКЦИЈА 4

19.	Љиљана Никшић	Методолошка искуства истраживања односа Србије и САД у контексту културе сећања	47-50
20.	Жељка Томић	Изоморфизам у контексту примене електротехнике у заштити културног наслеђа: методологија по Фукуоу	51
21.	Катарина Насрадин, Сузана Полић	Мултидисциплинарност у истраживању старих култура у стваралаштву Nagalda Natmana	52-54
22.	Бранка Радојчић, Слађана Пантелић	Значај система противпожарне заштите за очување објеката културне баштине – пример пожара у Нотр Даму	55-56
23.	Анђелка Милосављевић, Сања Петронић	Спецификум истраживања микроструктурних и микроморфолошких промена за израду протокола заштите индустријског наслеђа	57

#### СЕКЦИЈА 5

24.	Никола Славковић	Предикције примена 5G стандарда у креирању модела проширене реалности у заштити културног наслеђа	58-59
25.	Слађана Пантелић, Бранка Радојчић	Примена оптичких сензора у испитивању и обезбеђивању уметничких предмета и музеја	60-61
26.	Вељко Зарубица	Методологија за развој протокола примене мерних техника у заштити наслеђа	62-67
27.	Милош Павловић	Новија методолошка решења у примени нуклеарних техника у заштити културног наслеђа	68
28.	Љубинко Јањушевић, Александар Седмак, Сузана Полић	Методолошки приступи у херитолошкој валоризацији старих патената	69

#### СЕКЦИЈА 6

29.	Зоран Стевић, Сања Петронић, Миша Стевић	Конструкција апаратуре за дијагностику објеката културног наслеђа RTI методом	70-71
30.	Милеса Срејковић, Зоран Недић, Александер Ковачевић, Зоран Карастојковић, Милица Винић, Миливоје Ивковић	Проблеми интерпретације и идентификације примене разних дијагностичких техника у културној баштини	72-73
31.	Зоран Карастојковић, Радиша Периф, Павле Карастојковић, Сузана Полић	Методологија и дизајн у производњи златних православних крстова	74-80
32.	Сања Јевтић, Ана Илић, Драган Јевтић, Милеса Срејковић	Методологија изградње специфичних железничких система и мрежа	81-82
33.	Милена Давидовић, Милош Давидовић	Утицај квалитета ваздуха на очување културног наслеђа	83-85

## СЕКЦИЈА 7

34.	Братислав Ирчанин	Улога математике у концептуално-критичким методолошким приступима <i>Simona Mozega</i>	86-88
35.	Никола Славковић	Методичке оријентације и рефлексije научних истраживања у електротехници, погодне за креирање наставних активности у херитологији	89
36.	Љиљана Никшић	Методологија истраживања за изложбу о доприносу Пупина и Вилсона у мобилизацији и Версајском миру	90
37.	Сузана Полић	Методолошке основе у истраживању визуелних представа феномена <i>sensus communis</i>	91-93
38.	Милован Јанићијевић	Кохерентни снопови и критични феномени у материјалима са фракталном микроструктуром	94-95
39.	Лука Јовановић	Менаџмент и економија херитологије	96
40.	Бранимир Давидовић	Одржавање трасе 6 kv каблова у површинским рудницима у Србији	97-99
41.	Зоран Латиновић	Улога вештачке интелигенције у менаџменту	100
42.	Нада Ратковић Ковачевић, Зоран Стевић, Драган Шаиновић	Примена термалних - инфрацрвених сензора у херитологији	101-107



Милеса Срејкович<sup>1</sup>, Александер Ковачевић<sup>2</sup>

## РЕТРОСПЕКТИВА МЕТОДОЛОШКИХ ПРИСТУПА ПРИМЕНЕ ЛАСЕРСКИХ ТЕХНИКА У ХЕРИТОЛОГИЈИ: ИЗАБРАНЕ СТУДИЈЕ

**Апстракт:** Улога ласерских техника у херитологији се развија у складу са динамиком технолошког напретка, посебно ласерских система који су комбиновани са дијагностиком на бази кохерентног зрачења. У избору једног погледа на изабрану тематику, неколико студија примене ласерских техника у сврхе отклањања енкрустација, у овом раду се хронолошки прате важни тренуци у развоју методологије примене ласерских техника у оквиру синтезе оптичких и херитолошких анализа о спрези технологија и методологија.

**Кључне речи:** ласерске технике, уклањање наслага, методологија, оптика, херитологија

## RETROSPECTIVE OF METHODOLOGIC APPROACHES TO LASER TECHNIQUES IMPLEMENTATION IN HERITOLGY: CHOSEN STUDIES

**Abstract:** The role of laser techniques in heritology develops accordingly to the dynamics of the technological advance, specially laser systems, which are combined with diagnostics based on coherent radiation. Chosing one view to selected thematics, several studies on the implementation of laser techniques in encrustation removal, in this work important points of the development of the methodology of the implementation of laser techniques in the frame of the synthesis of optical and heritology analyses on the couplings between technologies and methodologies are chronologically monitored.

**Key words:** laser techniques, encrustation removal, methodology, optics, heritology

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### ПРОБЛЕМИ ИНТЕРПРЕТАЦИЈЕ И ИДЕНТИФИКАЦИЈЕ ПРИМЕНЕ РАЗНИХ ДИЈАГНОСТИЧКИХ ТЕХНИКА У КУЛТУРНОЈ БАШТИНИ

**Апстракт:** Спрега ласерских техника и херитологије у вишедеценијској историји је имала велики раст. Са једне стране је развијано много нових типова ласера и мерних метода базираних на кохерентном зрачењу. Са друге стране је херитологија обogaћена многим новим објектима културне баштине, пронађени су многи археолошки раритети, а појавила се и потреба за одржавањем и за обезбеђењем дугог живота историје. Постоји много избора у ласерској техници којима се може дијагностицирати дефинисано стање објекта са изабраним параметрима праћења. После извршених операција снимања стања објекта спектроскопским техникама којима се везују заостали напони и Раманово расејање, Бриугенови прагови (спектри, помаци, и ширина линија) са акустичким подацима и механиком објекта, ИЦ спектри и молекуларне особине итд...атомска спектроскопија са примесама или главним компонентама материјала. Паралелно недеструктивним методама, сада се појављују методе типа LIBS (LIPS) које уз микроскопске деструктивне ефекте дају своје одговоре о материјалу артефакта. Како постоје развијене базе података за велики број дефинисаних чистих или познатих материјала, потребно је опрезно прићи подацима за материјале артефакта са обзиром да се *a priori* тежи мањем оштећењу материјала да би могла да се употреби нека друга паралелна техника.

**Кључне речи:** спектроскопија, дијагностика, културна баштина

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because it is often sampled at multiple points and averaged (to be linked with a single outcome variable observation). The proposed method is evaluated in simulations and with experimental data obtained in South Adriatic zooplankton research. Practical advices on how to apply it using popular software tools are given.

### SS-MD1.3 MOGUĆNOST PROCENA RADIJACIONOG RIZIKA POMOĆU RETROSPEKTIVNE DOZIMETRIJE

*Aco Janićijević and Aleksandra Janićijević*

U savremenim istraživanjima aktuelna su tri pristupa za procenu rizika raka pluća ljudi nastalog od izlaganja radonu I njegovim kratkoživećim potomcima u vazduhu. Ova tri pristupa su pre svega zasnovana na: a) plućnoj dozimetriji; b) rudarskoj epidemiologiji, i c) kontroli slučajeva stambene epidemiologije. Od navedenih pristupa najdirektniji je ovaj pod c), koji izbegava mnoge nesigurnosti u pretpostavkama i kompleksnostima obuhvaćenih pod a) i b) i ima određene prednosti u odnosu na ova prva dva pristupa. Poslednjih godina raste broj epidemioloških studija koje imaju značajnu ulogu u povezivanju rezultata retrospektivne dozimetrije sa procenom rizika raka pluća stanovništva usled izlaganja radonu Rn i njegovim potomcima. Razvijeno je niz mernih tehnika koje imaju za cilj rekonstrukciju izlaganja radonu i njegovim potomcima tj. određivanje kumulativnog izlaganja tokom proteklih (dve-tri) decenije. To je bitno zato što indukcija raka pluća ima dug period skrivanja, pa se došlo do zaključka da je sveukupno izlaganje najvažnija determinanta rizika raka pluća. U ovom radu je data teorijska zasnovanost po pitanju ambijentalne prisutnosti aktivnosti  $^{210}\text{Po}$  u površinskom sloju stakla nekog prostora za profesionalni rad ili stambeni boravak.

### SS-MD1.4 CHOSEN PROBLEMS AND APPLICATIONS OF METHODS IN ENTOMOLOGY AND ENGINEERING AND THEIR COUPLING WITH NATURAL SCIENCES

*Aleksander Kovačević*

Insect collecting – capture, storage and preservation – is important method in entomology. Using insect studying in other scientific fields can be a good alternative to common methods of solving problems or can help develop various engineering applications. Moreover, some engineering methods can be used in entomology to improve studying of insects. Selected methods, like using femtosecond beam induced fluorescence, laser focusing microscopy, studying of the structure of butterfly scales, etc., are described as well as their applications, coupled in the fields of natural sciences.

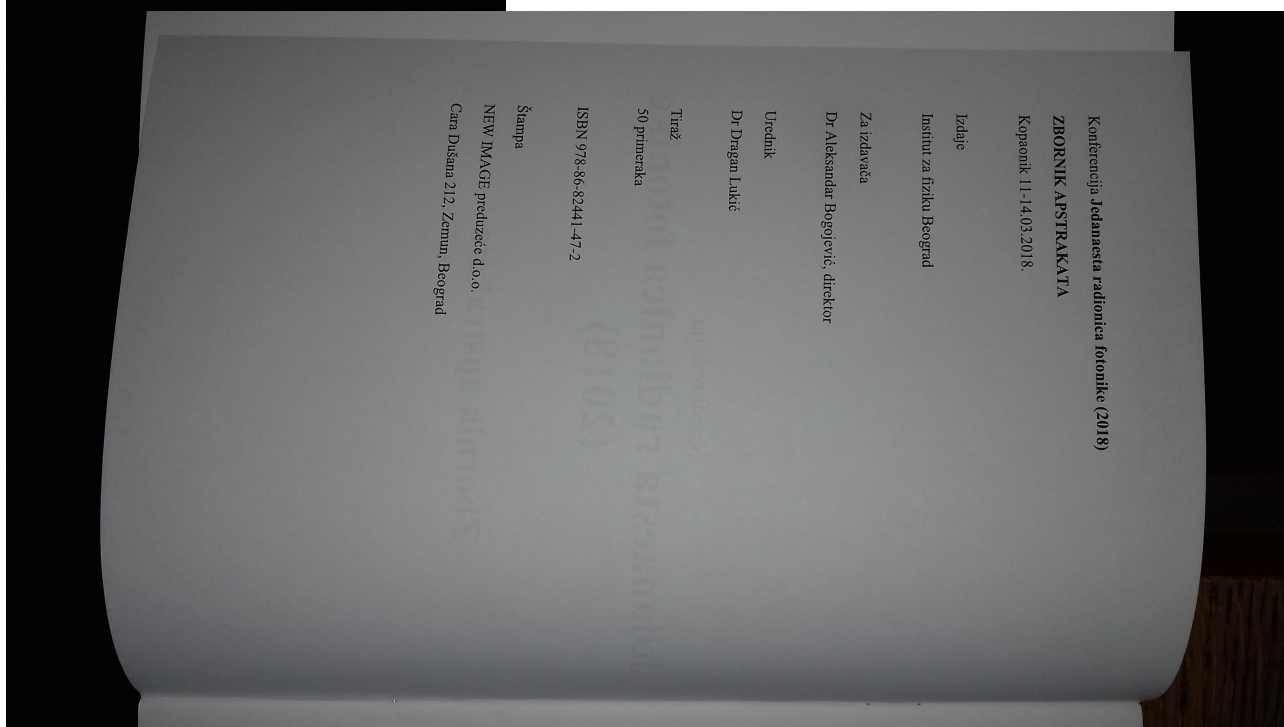
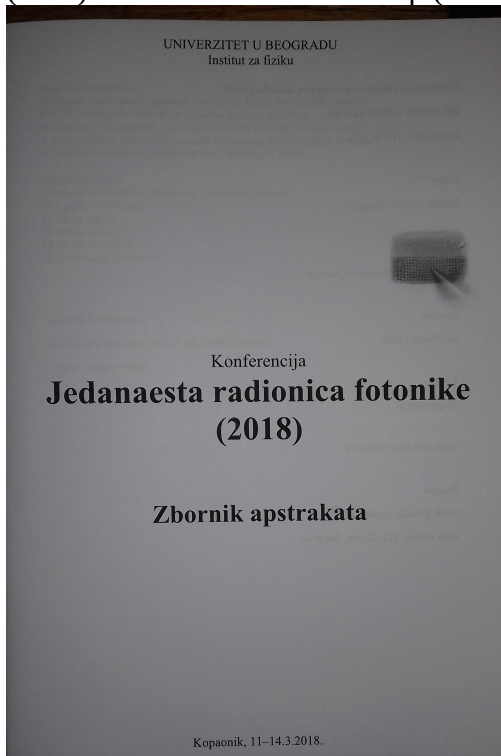
### SS-MD1.5 IMPACT OF LOW MELTING ALLOYS FROM PRINTED CIRCUITS/WASTE CARDS ON STRENGTH OF SUCH REMELTED STEEL

*Zoran Karastojković*

Modern cars contain printed circuits, which in their nature were made from a number of low melting alloys (LMA). The role of LMA in printed circuits is pretty well known and explained at an appropriate literature in PC fabrication. One of the main demand of alloys used for soldering of PC components is in their relatively low temperature of melting. The basic components for making these alloys belong to low melting metals as: Pb, Sn, Sb, Cd, Bi, In, Ge, and others. One of the main characteristics is in temperature of melting below 4500C, in PC production below 2500C. Farther, those LMA also must possess the

(M34) 11th Photonics Workshop (Conference), Kopaonik, March 11-14, 2018, p.34

(M34) 11th Photonics Workshop (Conference), Kopaonik, March 11-14, 2018, p.35



### Formation of LIPSS on Al/Ti thin metal films by scanning of low-fluence femtosecond beam during cross-directional scanning

Aleksander Kovacević<sup>1</sup>, Suzana Petrović<sup>2</sup>, Marina Lekić<sup>1</sup>, Davor Peruško<sup>1</sup>, Vladimir Lazović<sup>1</sup>, Svetlana Savić-Sević<sup>1</sup>, Borislav Vasić<sup>1</sup>, Branislav Salatić<sup>1</sup>, Radoš Gajić<sup>1</sup>, Dejan Panjelić<sup>1</sup>, Branislav Jelenković<sup>1</sup>

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**Abstract.** Formation of periodic nanostructures on thin metal films by femtosecond laser beam (laser induced periodic nanostructures – LIPSS) influences the tribological properties of the surfaces. Having excellent mechanical properties, multilayer thin films, like 5x(A1/Ti)/Si, are interesting direction of investigation, because their multilayer structure enables forming of high quality LIPSS [1]. Two types of LIPSS are described, high spatial frequency LIPSS (HSFL) and low spatial frequency LIPSS (LSFL), which differ in their spatial frequency. During exposition of thin film metal systems with femtosecond beams, we changed scanning configuration [2]. We have exposed 5x(A1/Ti)/Si multilayer system with femtosecond beam from the laser system Mira 900 in NIR. Same areas have been exposed with two different scanning directions. The direction of the second scanning over the same area was orthogonal to the first one. The polarization direction during second scanning was orthogonal to the polarization direction during first scanning. In this way, several crossed LIPSS can be seen.

**Acknowledgments.** The work has been supported by the Ministry of Science, Republic of Serbia, under No. III45016, OI171038 and OI171005.

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[2] A. G. Kovacević, S. Petrović, et al., *Appl. Surf. Sci.*, **417** (2017), 155–159.

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## Formation of LIPSS on Al/Ti thin metal films by scanning of low-fluence femtosecond beam during multi-pass scanning

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**Abstract.** During interaction of femtosecond laser beam with metal surfaces, periodic nanostructures can be formed – laser induced periodic nanostructures. LIPSS. Two types of LIPSS have been recorded, of low and high spatial frequency (LSFL and HSFL, respectively). Thin metal films in multilayer systems, like 5x(Al/Ti)/Si are convenient for forming high quality LIPSS due to their multilayer structure [1] and are interesting because of their excellent mechanical properties. We have exposed the multilayer thin film metal systems 5x(Al/Ti)/Si with femtosecond beams with various scanning configurations [2]. The laser system was Coherent Mira 900 in NIR. The same areas have been exposed to several scans of the beam (multi-pass scanning). In this way, it is possible to see that the LIPSS maintain their structure during consecutive scans.

**Acknowledgements.** The work has been supported by the Ministry of Science, Republic of Serbia, under No. III45016, OI171038 and OI171005.

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[2] A. G. Kovacević, S. Petrović, et al., *Appl. Surf. Sci.* **417** (2017), 155–159



# Inducing LIPSS on multilayer thin metal films by femtosecond laser beam of different orientations

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## Abstract

The occurrence of laser-induced periodic surface structures (LIPSS) has been known for a while. Multilayer thin films, like Al/Ti, are suitable for LIPSS formation and attractive for applications—due to their wearing behavior and corrosion resistance; LIPSS generation may improve their properties as well. LIPSS properties depend not only on the material but also on the beam characteristics, like wavelength, polarization and scanning directions, etc. After exposing with NIR femtosecond pulses from Coherent Mira 900 laser system in several beam exposures, we have analyzed the samples of thin metal film systems with Tescan Mira3 SEM and NTegra AFM. The formation of LIPSS is most probably due to the generation of surface plasmon polariton, through the periodic distribution of energy in the interaction zone which lead to thermal processes in layers and interfaces. Two types of LIPSS were generated, which differ in shape, orientation and in ablation pronounced or not. For consecutive interactions in the same direction, LIPSS maintained its orientation, while for orthogonal passes LIPSS with mutually orthogonal orientation were generated. LIPSS period fluctuated between 320 and 380 nm and structures with pronounced ablation have significantly smaller width. Probable mechanism is that for greater accumulated energy pronounced ablation takes place giving LIPSS in the form of trenches or grooves, while for less accumulated energy the buildup of the material—probably due to pronounced oxidation—lead to LIPSS in the form of hills or ridges.

**Keywords** Laser nanostructuring · Thin metal films · LIPSS · Structures orientation

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Guest Edited by Goran Gligoric, Jelena Radovanovic and Aleksandra Maluckov.

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## 1 Introduction

Interaction of pulsed laser beam with surfaces yields the appearance of LIPSS (laser-induced periodic surface structures). The occurrence of LIPSS has long been known and studied (Birnbaum 1965; Van Driel et al. 1982; Sipe et al. 1983; Young et al. 1984; Ursu et al. 1985). It has been studied on variety of materials: metals (Ursu et al. 1985; Wang and Guo 2005; Vorobyev and Makin 2007; Vorobyev and Guo 2008, 2013), semi-conductors (Von der Linde et al. 1997; Bonse and Krüger 2010; Bonse et al. 2011; Varlamova et al. 2014), dielectrics (Reif et al. 2008), graphite (Goloso et al. 2011), compounds (Kautek et al. 2005; Gakovic et al. 2011), diamond (Shinoda et al. 2009), graphene (Beltaos et al. 2014). LIPSS properties depend not only on the material but also on the beam characteristics, like wavelength, polarization and scanning directions, etc. (Kovačević et al. 2017).

Surface morphology is a key factor in controlling the optical, mechanical, wetting, chemical, biological, and other properties of a solid surface. LIPSS may improve material properties by functionalization and may widen applications: structural coloring, absorptance enhancement, antireflective films, biomedical applications, optofluidics applications, holography, anti-counterfeiting, decorating, sensing, catalysis, optical data storage (Vorobyev and Guo 2013).

The occurrence of LIPSS can be viewed as an inherent phenomena of the interaction of the ultrafast beam with solid surface, with main characteristics that the spatial period of LIPSS is less than the beam wavelength. The orientation depends on the incident beam polarization direction. Generation is explained by self-organization or by surface plasmon polaritons (SPP) (Vorobyev and Makin 2007; Reif et al. 2008). Incident wave induces oscillations of charges (surface plasmon) and SPP forms as the coupling between incident and induced waves; in this way periodic distribution of energy is formed on the surface.

Two types of LIPSS are reported: low spatial frequency LIPSS (LSFL) and high spatial frequency LIPSS (HSFL) (Bonse et al. 2005). LSFL period  $\lesssim$  wavelength and HSFL period  $<$  wavelength/2. Named after their size (magnitude of spatial frequency), their orientation in respect to the polarization direction is not yet fully understood. It seems that LSFL orientation is perpendicular to polarization for metals and semiconductors (Bonse et al. 2012). Due to SPP, periodical distribution of thermal energy on the surface can instigate thermal processes. The occurrence of metal-oxide, or thermochemical type of LIPSS has been reported on Ti, Ni, Cr and NiCr surfaces, as well as ablative LIPSS and models have been proposed (Öktem et al. 2013; Dostovalov et al. 2017, 2019a).

When creating LIPSS on multilayer thin metal films, the underneath layer has an important role. In the example of Al/Ti multilayer film (Kovačević et al. 2015), Ti and Al have different electron heat conductivity and electron–phonon coupling. Top layer (Al) electrons accept energy and quickly transfer to the next layer (Ti). Strong coupling keeps the energy in Ti and away from topmost Al. In this way, the damage threshold for Al increases which preserves LIPSS for longer expositions. In this work, we have examined the LIPSS generated upon consecutive scanning over the same area of same and of different scanning orientations. By changing the parameters of the beam (fluence, scanning speed, scanning number and directions over the same area) the formation of LIPSS was affected. Two types of LIPSS, which differ in shape, orientation to the incoming beam polarization and in ablation pronounced or not, are generated and examined during repeated consecutive scanning of same and orthogonal directions. For lower accumulation on energy, LIPSS in the form of ridges formed while for higher fluences and accumulated energies, the generation of LIPSS gave prevalence to the ablation. After repeated consecutive scanning along the

same trajectory LIPSS preserved to some extent. Also, during scanning along close parallel lines, LIPSS from one line affected generation of LIPSS from neighboring line. Overlapping scanning lines should generate LIPSS mutually perpendicular. We have examined the three cases of LIPSS: repeated consecutive scanning along same trajectory, scanning with close parallel lines, and scanning with perpendicular lines. The results can be of use in functionalization of materials by LIPSS forming with possible impact in wetting and biomedical applications.

## 2 Experiment and methods

The samples were prepared by D.C. ion sputtering in a single vacuum run, using Ar ions and switching from one target to the other. Targets were 99.9% pure Al and Ti deposited on a Si(100) wafer as a substrate. In this way, 5×(Al/Ti) multilayer structures have been generated, where each layer was 13 nm thick and total thickness of the multilayer structure was 130 nm.

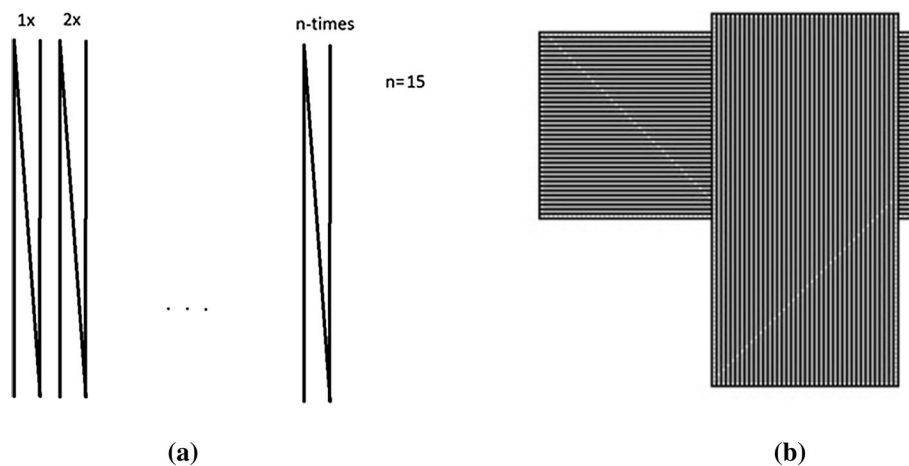
Coherent Mira 900 laser system was a source of NIR femtosecond pulses (wavelength 730–840 nm, repetition rate 76 MHz, fluence 145–260 mJ/cm<sup>2</sup>) pumped by Verdi V10 Nd:YVO<sub>4</sub> CW laser (wavelength 532 nm, power 10 W) for exposition of the samples. Steering and focusing was a part of a modified optical microscope with 2D mirror scanner (objective 40×, NA 0.65). Ocean optics HR2000CG UV-NIR fiber spectrometer was used for spectral detection. The samples have been analyzed with Tescan Mira3 SEM and NTe-gra Prima AFM under ambient conditions. The numerical simulations have been performed by COMSOL Multiphysics package, with one-dimensional two-temperature (1D TTM) model. Basic relations underlying the TTM model were proposed by Anisimov (Anisimov et al. 1974). The model observes the electron and lattice subsystems. TTM model has been used for many years to calculate the temperature of the electrons and lattice during interaction of ultrashort laser pulses with different materials. All necessary physical quantities and constants that we used in the simulation can be found in the literature (Majchrzak et al. 2010a, b). The fs beam from laser was introduced into the modified microscope onto the steering two-axis scanning mirror system and transferred through the objective of the microscope to the sample.

Patterns used for interaction are presented in Fig. 1. For consecutive repeated scanning over same trajectory, the pattern in the form of letter “N” is used (Fig. 1a). The laser beam traverses over the sample surface following the pattern of the letter. At first location, it “writes” one letter. At second (neighboring) location, it traverses the same trajectory twice, writing two letters one over another. At third location, it writes three letters, and so on. The pattern used for perpendicular overlapped scanning is composed of set of parallel lines and the sample is rotated by 90° (Fig. 1b).

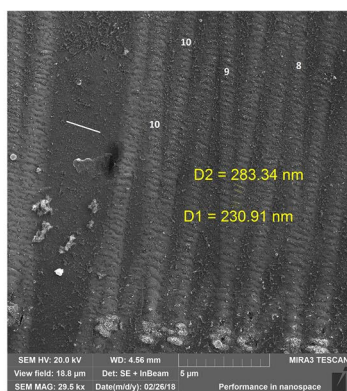
## 3 Results and discussion

The samples were exposed to laser beam of 730, 800 and 840 nm of wavelength with different fluences. Irradiated areas were examined by SEM and AFM. For specified parameters, simulations of 1D temperature distribution were performed.

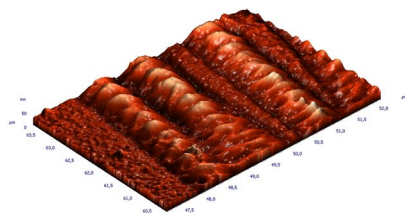
In Fig. 2, the results after beam of 800 nm wavelength and 153 mJ/cm<sup>2</sup> of fluence repeatedly scanned from 1 to 10 times over the surface are presented. The area where beam



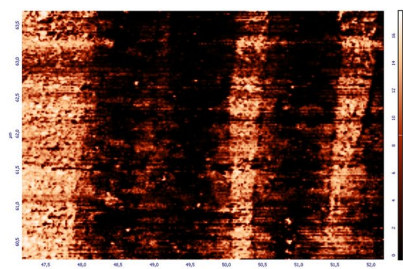
**Fig. 1** Implemented patterns of scanning: **a** for consecutive repeated scanning; **b** for perpendicular overlapped scanning



**(a)**



**(b)**



**(c)**

**Fig. 2** LIPSS generated after beam of  $153 \text{ mJ/cm}^2$  repeatedly scanned over the same trajectory: **a** SEM of the area of 8–10 passes; **b** AFM, detailed portion of the area in **a**—rendered area is  $(3 \times 5) \mu\text{m}$  and maximal height is 50 nm; **c** graphical presentation of the AFM current (a.u.) of the area in **b**

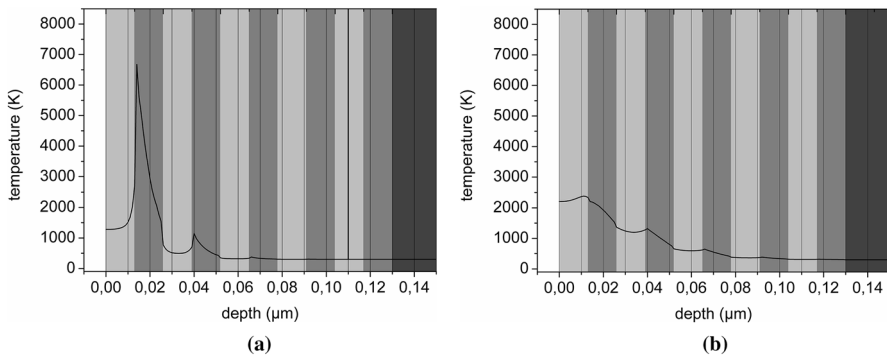
scanned 8, 9 and 10 times along the same trajectory, following the shape of the letter “N”, is shown in Fig. 2a. White line on the left side of the image presents polarization direction. The beam repetition rate was 76 MHz, diameter  $\sim 1.2 \mu\text{m}$ , scanning speed  $242 \mu\text{m/s}$ . Effective number of pulses (number of pulses which affect the area of a beam spot) for one pass is 317,000. LIPSS in the form of ridges parallel to the polarization direction with spatial period of  $\sim 283 \text{ nm}$  are generated and preserved up to 10 passes. In Fig. 2b, detailed AFM view of a part of the area from Fig. 2a which shows 10 passes is presented. AFM current of the area from Fig. 2b is shown in Fig. 2c.

The simulation of the lattice temperature from the surface to the bulk is shown in Fig. 3. Odd layers (Al) are presented with light grey bars, even layers (Ti) are presented with grey bars, while substrate (Si) is presented with dark grey bar. After 1.25 ps (Fig. 3a), the temperature reaches maximum in the second (Ti) layer. After 20 ps (Fig. 3b), the temperature reaches maximum in the first (Al) layer.

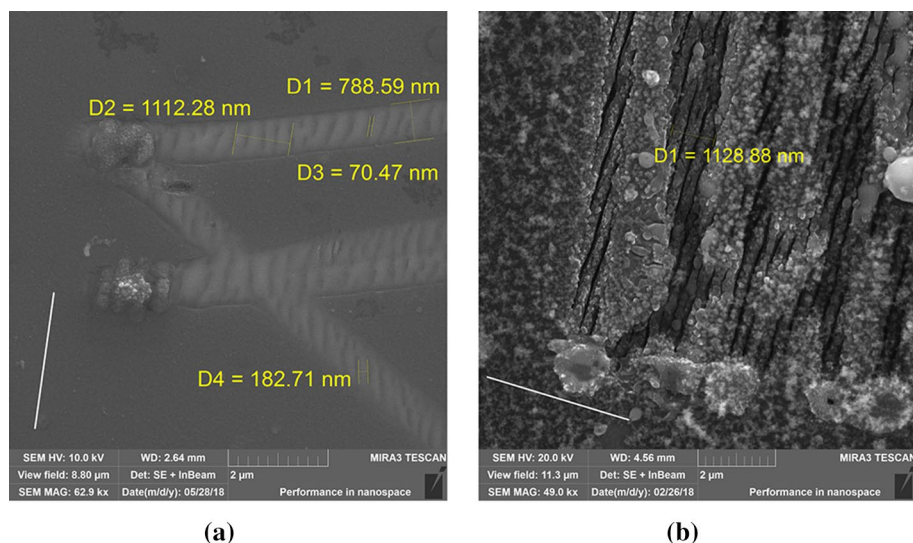
In Fig. 4, the results after beam of: (a) 730 nm wavelength and  $145 \text{ mJ/cm}^2$  fluence (repetition rate 76 MHz, diameter  $\sim 1 \mu\text{m}$ ) scanned 15 times (scanning speed  $1.14 \text{ mm/s}$ , effective number of pulses for one pass 67,000) and (b) 800 nm wavelength and  $215 \text{ mJ/cm}^2$  fluence (repetition rate 76 MHz, diameter  $\sim 1.1 \mu\text{m}$ ) scanned 10 times (scanning speed  $24 \mu\text{m/s}$ , effective number of pulses for one pass 667,000) along the same trajectories are presented. In Fig. 4a, LIPSS are in the form of ridges (spatial period of  $\sim 278 \text{ nm}$ ) parallel to the polarization direction. In Fig. 4b, LIPSS are in the form of grooves (spatial period of  $\sim 370 \text{ nm}$  and groove width of  $\sim 80 \text{ nm}$ ) perpendicular to the polarization. In both cases LIPSS are preserved up to 15 and 10 passes, consecutively. Higher fluence provoked the appearance of groove-type of LIPSS. Spatial temperature distribution is similar in shape to the distributions shown in Fig. 3.

In order to create structures of mutual perpendicular direction at the same area, we performed perpendicular consecutive scanning of two (same) patterns by sample rotation (Fig. 1b). The beam was of 840 nm wavelength and the fluence was set to  $\sim 182 \text{ mJ/cm}^2$  in order to generate groove-type of LIPSS. The beam repetition rate was 76 MHz, scanning speed  $1.5 \text{ mm/s}$ , diameter  $\sim 1.1 \mu\text{m}$ , Effective number of pulses for one pass is 51,000.

In Fig. 5a, the interaction area of the sample with two consecutive beam scanning of the same pattern (Fig. 1b) is presented. The right-hand and lower parts of the image present areas where patterns don't overlap, while central, upper and left parts present overlapped patterns. Magnified portion of the right-hand part, Fig. 5b, shows that grooves of two



**Fig. 3** Spatial temperature distribution from the surface to the bulk after exposition to the beam of 800 nm wavelength and  $153 \text{ mJ/cm}^2$  of fluence: **a** after 1.25 ps; **b** after 20 ps

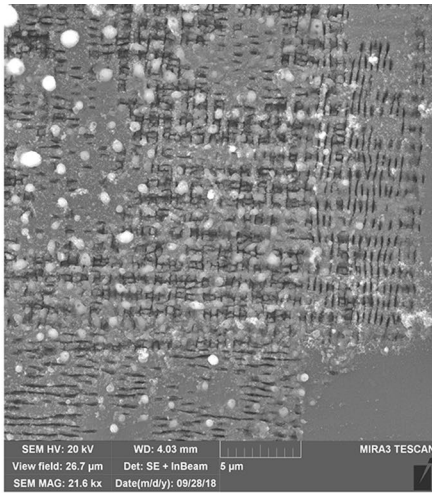


**Fig. 4** SEM micrographs of LIPSS generated after beam of: **a** 145 mJ/cm<sup>2</sup> scanned 15 passes and **b** 215 mJ/cm<sup>2</sup> scanned 10 passes. White line on the left side shows polarization orientation

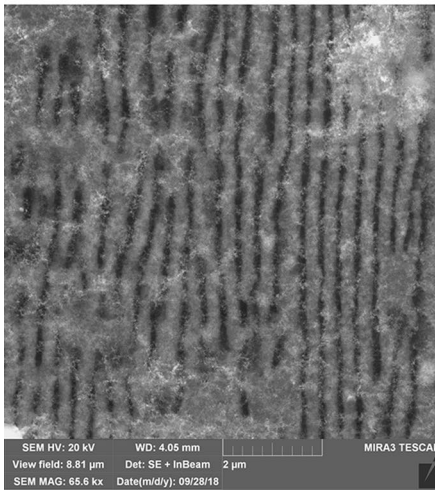
neighboring lines connect when patterns do not overlap. Where patterns overlap, Fig. 5c, grooves don't form in connected perpendicular directions; their width ranges from 98 to 126 nm.

The decrease in the AFM current (Fig. 2c) in the areas of laser exposition could be explained by increased resistivity of the exposed areas. Interaction with the beam fostered the penetration of nitrogen and/or oxygen into the first (Al) layer increasing the resistivity, which goes well with the three-step model (Öktem et al. 2013). The lattice temperature distribution from the surface to the bulk (Fig. 3) shows the influence of the multi-layer structure. After 1.25 ps (Fig. 3a), the temperature reaches maximum in the second layer (Ti). Moreover, the temperatures are higher in Ti layers than in neighboring Al layers. This is explained by the difference between two materials characteristics (Kovačević et al. 2015). Electrons from Al can quickly transfer energy to Ti layer away from the interaction zone due to the difference in electron–phonon coupling. This increases the damage threshold in Al leading to more regular ripples. The repetition rate also influences the regularity of the LIPSS, as noted in (Dostovalov et al. 2019b): higher the repetition rate, more ordered structures are formed.

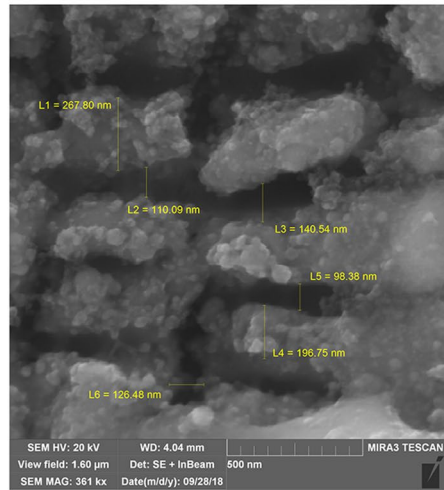
The LIPSS in the form of ridges (Figs. 2a, 4a) are most probably generated by the penetration of the nitrogen and/or oxygen from the ambient (air) into the material—thermochemical LIPSS (Öktem et al. 2013; Dostovalov et al. 2017). For higher fluences, LIPSS formed in the form of grooves by ablation mechanisms, which can be deduced by scattered ejected material seen in Fig. 4b. Slower scanning speed and low melting point of Al induced energy accumulation sufficient for Al melting and ablation, which gave the prevalence of the generation of grooves (ablative LIPSS) over ridges (thermochemical LIPSS). The comparison of the spatial periods—283 nm (Fig. 2a) and 278 nm (Fig. 4a) for ridges versus 370 nm (Fig. 4b) for grooves—suggests grooves could be classified into LSFL and ridges into HSFL; this could be also supported by their orientation in respect to the beam polarization direction (Bonse et al. 2013).



(a)



(b)



(c)

**Fig. 5** SEM micrograph of LIPSS generated by consecutive pattern scanning and sample rotation: **a** wide area; **b** right-hand part of the area in **a**; **c** magnified part of the central area in **a**

The attempt to generate intersecting perpendicular grooves was not successful. The reason is twofold. First generated LIPSS pattern distracts the formation of the second LIPSS pattern. Also, the formation of the second LIPSS pattern smears the first LIPSS pattern due to the accumulation of energy.

## 4 Conclusion

We have exposed  $5\times(\text{Al}/\text{Ti})$  multilayer thin film metal structures to fs laser beam of various wavelengths and fluences. Due to differences in materials characteristics, the temperatures are higher in Ti layers than in neighboring Al layers, which was illustrated by simulations. The appearance of LIPSS indicates lateral periodical distribution of temperature in second layer (Ti). Two types of LIPSS emerged depending on the beam fluence. For fluence lower than  $\sim 170 \text{ mJ}/\text{cm}^2$ , LIPSS in the form of ridges are generated most probably by the penetration of nitrogen and/or oxygen into the sample material (thermochemical LIPSS), which can be deduced by the decrease in the AFM current indicating the increase in resistivity. For higher fluences (above  $170 \text{ mJ}/\text{cm}^2$ ), LIPSS in the form of grooves are generated by ablation mechanisms (ablative LIPSS). Both types are preserved after 10–15 consecutive beam scanning along the same trajectory. Intersecting perpendicular LIPSS can't be successfully formed because of competing influences of perpendicular patterns causing smearing of LIPSS.

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Article

# Molding Wetting by Laser-Induced Nanostructures

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**Abstract:** The influence of material characteristics—i.e., type or surface texture—to wetting properties is nowadays increased by the implementation of ultrafast lasers for nanostructuring. In this account, we exposed multilayer thin metal film samples of different materials to a femtosecond laser beam at a 1030 nm wavelength. The interaction generated high-quality laser-induced periodic surface structures (LIPSS) of spatial periods between 740 and 790 nm and with maximal average corrugation height below 100 nm. The contact angle (CA) values of the water droplets on the surface were estimated and the values between unmodified and modified samples were compared. Even though the laser interaction changed both the surface morphology and the chemical composition, the wetting properties were predominantly influenced by the small change in morphology causing the increase in the contact angle of ~80%, which could not be explained classically. The influence of both surface corrugation and chemical composition to the wetting properties has been thoroughly investigated, discussed and explained. The presented results clearly confirm that femtosecond patterning can be used to mold wetting properties.

**Keywords:** wetting; nanostructures; LIPSS; nano-optics

## 1. Introduction

Generating periodical sub-wavelength structures on material surfaces by interaction with pulsed laser beams has been known for some time [1–4]. The structures in the form of parallel ripples—laser-induced periodic surface structures (LIPSS)—have been reported on various materials, including dielectric materials, metals, semiconductors, and graphene [5–7]. Being in the nanometer scale, and with the occurrence due to the laser-surface interaction, they are a frequent subject of investigation in nano-optics (nanophotonics).

The causes of LIPSS generation and shaping are seen most probably in the emergence of the surface plasmon polariton (SPP), as well as in the hydrodynamic features [8–10]. If compared to single layer metal films, more regular LIPSS are generated with low-fluence femtosecond (fs) laser beam interaction with multilayer thin metal films, since the existence of the metal sublayer influences the quality and stability of LIPSS [11].

Changing the wetting and tribological properties of the material by LIPSS formation opens new fields of application in nano/microfluidics, optofluidics, fluid microreactors, biomedicine, biochemical sensors, and thermal management [12,13]. The control of wetting properties and achieving super-hydrophobic surfaces by laser interaction have been reported for various materials: stainless

steel, TiAl alloy, Si and materials coated with hydrophobic materials, such as chloroalkylsilane and fluoroalkylsilane [14–16]. In all mentioned cases super-hydrophobicity is achieved by the chemical modification of the surface or micro-structuring.

Laser interaction can induce the change in chemical composition of the surface, forming ultra-thin oxide layers, which contribute to the wetting. Wetting is initially enhanced, but as time passes, it is ultimately reversed by surface chemistry phenomena that take place on the irradiated surfaces—i.e., hydrophobicity is increased [16,17].

Van der Waals and electrostatic forces play an important role in adsorption, adhesion and wetting phenomena [18]. The Casimir force shows application potential in the field of micro- and nano-electromechanical systems—engineered devices, where controlling forces between microscopic bodies or surfaces are crucial for a variety of applications [19,20]. The influence of submicron surface corrugations on Lifshitz–van der Waals forces have been calculated for polyethylene, where surface nano-patterning is responsible for changing the forces from attractive to repulsive [21,22]. For metal surface nano-structuring at scales below the plasma wavelength, the Casimir interaction decreases faster than usual for large inter-surface separation, while at short separations an equivalent pressure is larger [23]. The motivation of our study is to experimentally reveal the link between wetting and nano-corrugation [21,22] and to extend the potential application of nanostructures. Surface nanostructures affect the optical characteristics via refractive index on the one hand, while on the other, they influence the wetting characteristics. Patterning the surface of the lamellar material changes the characteristics of the interface, as well as the characteristic of the bulk to some extent. The nanometer level of the patterns increases the importance of their control and places it in the field of nano-optics/nano-photonics.

The morphology of surface micro/nanostructures obtained by fs laser is evaluated with the aim of determining the influence of morphology on their wetting. By interaction with fs laser beam, we generated LIPSS on the surfaces of several metallic multilayer materials. We estimated the contact angle (CA) difference for different materials and also for materials before and after LIPSS forming, thus examining the change in hydrophobicity.

## 2. Materials and Methods

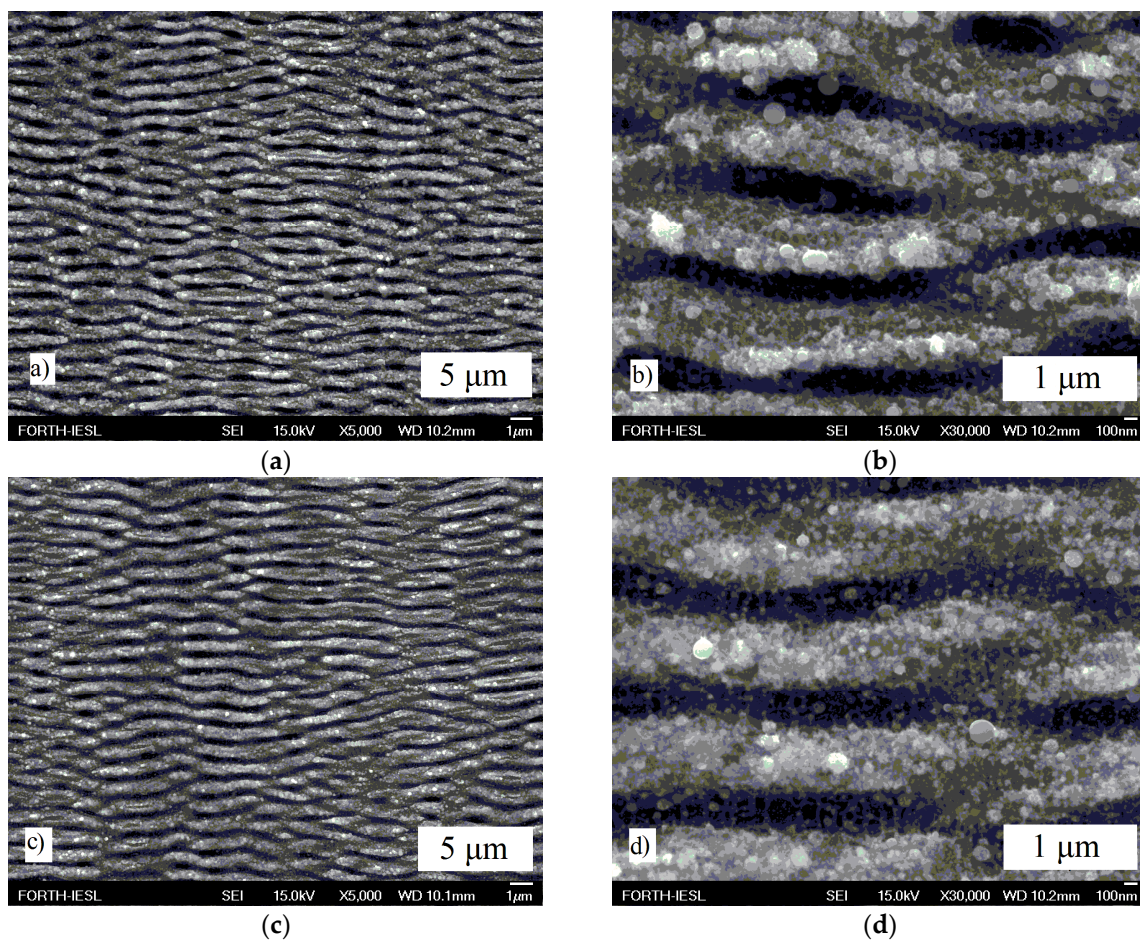
The samples were multilayer thin films on Si substrate: 15×(Zr/Ti), 15×(Ti/Zr), 8×(Zr/Cr/Ti), with the topmost layer being Zr, Ti and Zr, respectively. The total thickness of each multilayer structure was aimed to be ~500 nm, making the thicknesses of individual layers—controlled by the deposition rate—about 17 nm for bi-layer samples and ~21 nm for tri-layer sample. The samples were exposed to femtosecond beam which generated the LIPSS.

The laser source was the Pharos SP Yb:KGW laser system from Light Conversion. The surfaces of the thin films were irradiated at normal incidence in open air by focusing a linearly polarized pulsed beam with a 1 kHz repetition rate, 160 fs pulse duration, and 1030 nm central wavelength to a 43 μm Gaussian spot ( $1/e^2$ ) diameter. The samples were mounted on a motorized, computer-controlled, X-Y-Z translation stage, which enabled scanning (3 mm/s) and positioning out-of-focus to widen the scanning line. For higher precision, the irradiations were conducted at identical conditions, covering a surface of 5 × 5 mm at a pulse power of 2.5 mW (fluence of 0.662 J/cm<sup>2</sup>) and scan velocity of 3 mm/s with a constant distance between the lines of 15 μm.

In order to determine the effects of chemical composition and morphology on wetting, the drops of distilled water were placed onto both the unmodified and laser-processed surface areas in open air. The volume of each droplet was of 3 μL, controlled by a motorized syringe, while the shape was determined by micro-photographing in the horizontal direction by using Data Physics OCA (optical contact angle) Series device. The values of contact angles were estimated from the images by the utilization of the Gwyddion software [24].

### 3. Results and Discussion

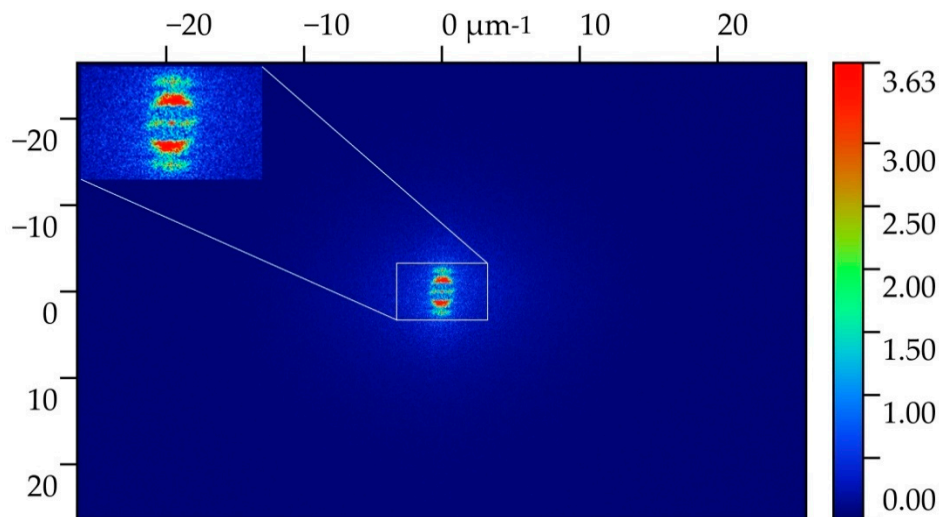
The wetting properties of the surfaces were influenced both by their chemical composition and by their morphology—i.e., corrugation [25,26]. The influence of the different chemical composition is represented by different materials of the topmost layers, while the influence of the morphology is caused by the interaction with the laser. The role of the more complex multilayer structure is represented by using  $8\times(\text{Zr/Cr/Ti})$  samples. Exposing the samples to the fs laser beam generated LIPSS on the surfaces, shown in Figure 1. LIPSS on the surface of  $15\times(\text{Ti/Zr})$  on Si are shown for two magnifications in Figure 1a,b. LIPSS on the  $8\times(\text{Zr/Cr/Ti})$  are presented in Figure 1c,d, and also for two different magnifications. In addition to wavy-patterned LIPSS, the occurrence of nanoparticles with dimensions less than the periods of LIPSS, is also noticeable.



**Figure 1.** SEM micrographs of LIPSS on the surface of the: (a)  $15\times(\text{Ti/Zr})$  on Si, magnification 5000 $\times$ ; (b)  $15\times(\text{Ti/Zr})$  on Si, magnification 30,000 $\times$ ; (c)  $8\times(\text{Zr/Cr/Ti})$  on Si, magnification 5000 $\times$ ; (d)  $8\times(\text{Zr/Cr/Ti})$  on Si, magnification 30,000 $\times$ .

Since the ablation threshold for the multilayer components is lower than the applied fluence, the ablation of the multilayer samples was expected [27]. The experiment with similar conditions (beam, environment, material), created similar ablative LIPSS, while the remaining material in the laser-affected area retained the layered structure (alternating Ti and Zr layers) [28]. Due to the similarity in both conditions and of the outcomes (LIPSS) between the two experiments, it is possible to assume that in the presented experiment the material also retained layered structure in laser affected zones and there were no drastic changes in Ti, Zr, Si and O components among the three zones—the center of, the periphery of, and away from the laser-affected zone.

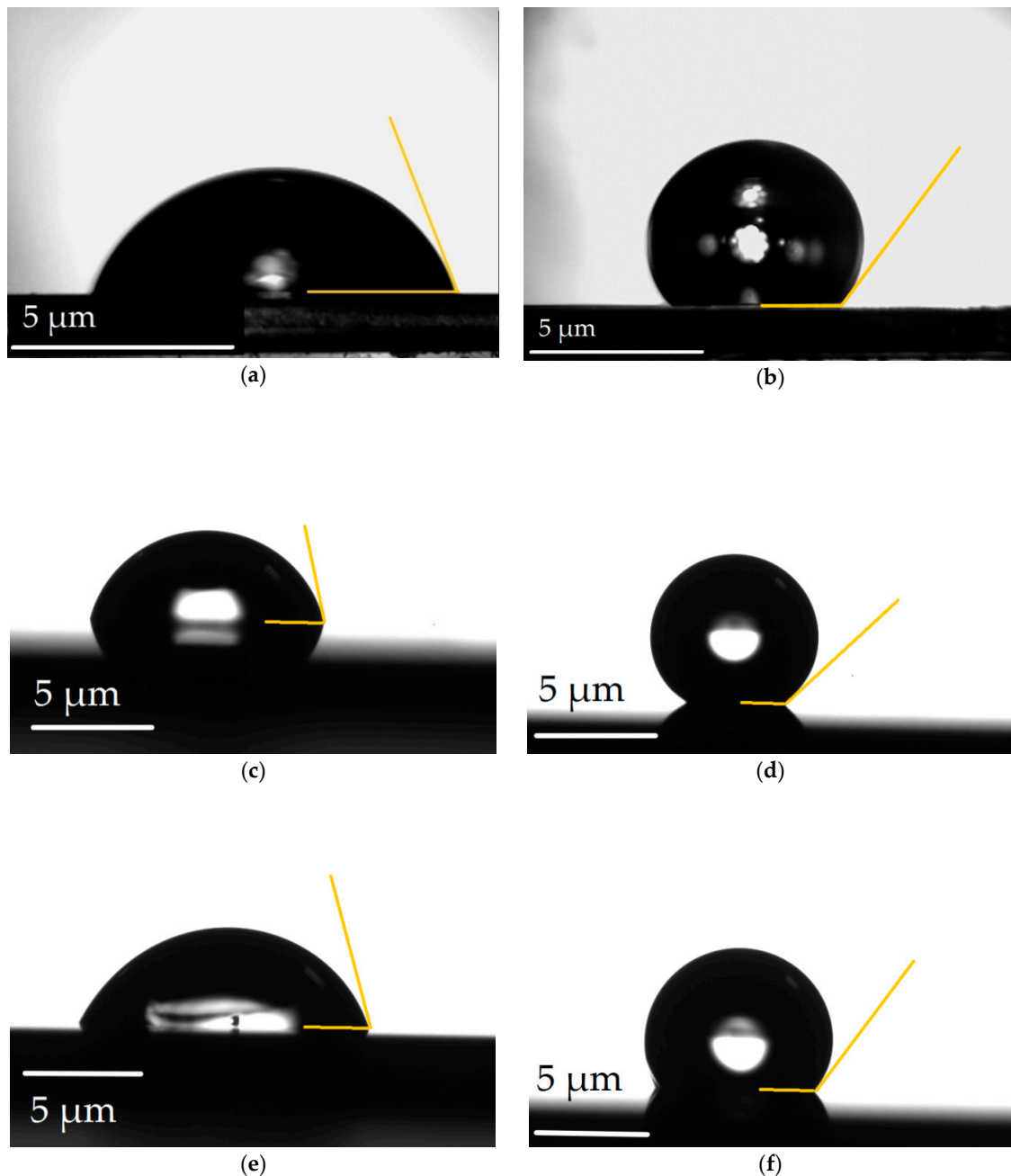
SEM results (Figure 1) reveal that there is a periodicity in the pattern, but the 2DFFT processing of LIPSS micrograph from Figure 1a presented in Figure 2 provides better insight. The appearance of the maxima (inset in Figure 2) shows that the structures are periodic. A clear distinction in the maxima indicates that LIPSS are highly regular, due to the multi-layer structure. By the convenient combination of the materials, the existence of the underneath layer produces a steep change in the layers' temperatures and enables the transfer of thermal energy through the interface and away from the interaction zone [11].



**Figure 2.** Representative 2D-FT of the image shown in Figure 1a. Inset: detailed view of the central part. Intensity scale (bar on the right) is in arbitrary units.

The interaction of fs beam with multilayer thin film materials induces the intermixing of initially separated layers, which could be attributed to the alloying between the components [29]. Due to no drastic changes in the components, not much of the oxides were produced, suggesting that their contribution to the CA value was not significant.

The contact angle of a surface processed by laser beam evolves after the processing. It was shown for some alloys that CA increases to the equilibrium level with a higher rate for lower implemented laser fluence: for alloys containing Ti and fluence of  $0.78 \text{ J/cm}^2$ , it takes a couple of days to be close enough to the equilibrium [16]. The images of water droplets, placed on the surfaces both unexposed and exposed to fs beams, are presented in Figure 3. For  $15\times(\text{Ti/Zr})$ , the droplets are shown in Figure 3a (on unexposed surface) and Figure 3b (on exposed surface). Different shapes indicate the influence of the laser-induced change of the surface, which is twofold: both in the morphology and in the chemical composition. Similar holds for the droplets on the  $15\times(\text{Zr/Ti})$  unexposed (Figure 3c) and exposed (Figure 3d) surfaces, as well as on the  $8\times(\text{Zr/Cr/Ti})$  unexposed (Figure 3e) and exposed (Figure 3f) surfaces. On exposed surfaces, the droplets were placed 180 days after the exposition ( $15\times(\text{Ti/Zr})$ ) and 2 days after the exposition ( $15\times(\text{Zr/Ti})$  and  $8\times(\text{Zr/Cr/Ti})$ ).



**Figure 3.** The image of the droplet of distilled water on the surface of: (a) 15×(Ti/Zr) on Si (Ti is the topmost layer), as-deposited, the CA = 72.11°; (b) 15×(Ti/Zr) on Si (Ti is the topmost layer), laser-modified, the CA = 137.15°; (c) 15×(Zr/Ti) on Si (Zr is the topmost layer), as-deposited, the CA = 77.29°; (d) 15×(Zr/Ti) on Si (Zr is the topmost layer), laser-modified, the CA = 144.49°; (e) 8×(Zr/Cr/Ti) on Si (Zr is the topmost layer), as-deposited, the CA = 68.10°; (f) 8×(Zr/Cr/Ti) on Si (Zr is the topmost layer), laser-modified, the CA = 123.45°. White bar denotes 5 μm. Golden lines are added for the visualization.

As a consequence of ultra-short laser beam interaction with metal surfaces in open air, the surface plasmon polariton (SPP) occurred at the interface between the metal and the dielectric (air) surfaces, inducing periodic distribution and accumulation of the energy on the surface. Because fs pulse duration is shorter than the characteristic relaxation time of the material, energy deposition is shock-like and leads to the ablation of the material, leaving parallel trenches—LIPSS. The spatial period of LIPSS is less than the incoming wavelength. The heat load to the surrounding material was minimal. The ablated material was dispersed away in the form of nanoparticles with the dimensions of up to 100 nm. In this

way, the deposited energy was mostly invested in the ablation and not in heating, which implies that, for fs interaction, the creation of a thick oxide layer did not take place.

The values of the LIPSS periods estimated from the micrographs, as well as measured values of contact angles of both surfaces, are summarized in Table 1. The period matches between 72 and 77% of the value of the implemented wavelength. The uniformly distributed LIPSS are oriented in the normal to the polarization direction of the incoming beam and their period is not close to the used laser wavelength for all observed samples. These types of LIPSS originate from the interference of the incident laser beam with surface electromagnetic wave excited during the laser treatment [8]. The CA values indicate that non-corrugated surfaces are hydrophilic. Similar CA values for surfaces unmodified by the laser suggest that they are not much influenced by the difference in the composition. The difference in CA between the untreated samples of Zr/Ti and Zr/Cr/Ti could be explained by long-range wetting transparency [30]. For surfaces modified by laser beam, the values also do not differ much. However, a strong increase after the interaction by the fs laser beam suggests it is dominantly caused by the structural change—the occurrence of LIPSS—and not by the composition change. Different to the case where a 40° rise in hydrophobicity of Ti surface was obtained by  $\mu\text{m}$  corrugations [17], better results in this work were obtained by nm corrugations. In open air ambience, all transition metals (except a few) are covered with a thin native oxide layer [31]. Different values of CA in the literature are the consequence of different preparation methods, which lead to differences in the chemical composition on the interface. Thin zirconium oxide layer (80–90 nm) on the  $\text{SiO}_2$  substrate shows small values of CA (hydrophilicity), but thicker layers show higher values of CA [32,33]. Oxides of titanium have various values of water contact angles [34]. Due to long-range wetting transparency, the water contact angle of the  $\text{MgF}_2$  layer, thinner than 100 nm, becomes highly insensitive of its chemical composition and its value approaches the value of the CA of the underlying materials: glass, Au and Au/ $\text{MgF}_2$  metamaterial [30]. Therefore, it could be assumed that very thin (<100 nm) layers of metal-oxides did not contribute to the water CA value. Due to the similarity in the experiments [28], the change in the components was small and due to the forming of LIPSS by fs ablation (energy invested in ablation, not in heating), not many of the oxides were produced and their contribution to the CA value was not significant. The CA changed mostly due to the corrugations and not the presence of oxides.

**Table 1.** The LIPSS period of the sample materials and the CA values.

Material	LIPSS Period (nm)	CA (°)		$\Delta\text{CA}$ (°)	CA Increase (%)
		untreated surface	corrugated surface		
15×(Ti/Zr)	740	72.11 ± 3.32	137.15 ± 11.63	65.04	90
15×(Zr/Ti)	740	77.29 ± 1.71	144.49 ± 14.97	67.20	87
8×(Zr/Cr/Ti)	790	68.10 ± 6.41	123.45 ± 25.26	55.35	81

In this view, all the samples should have a similar increase in the CA after the interaction. However, there is a small difference which would mean that slightly different amounts of Zr and Ti components are removed by laser treatment depending on the initial order of the layers. Here, a significant mixture of the materials did not take place and spatial distribution retained a layered structure, which resulted in a similar increase in the CA angle.

From the SEM images (Figure 1), the Gwyddion program was used to graphically extract the profiles along the selected lines perpendicular to the direction of the LIPSS, and from them to estimate the filling factors—the ratio between the ripple width and the spatial period. For 15×(Ti/Zr), it is  $\sim 0.6$ , while for 8×(Zr/Cr/Ti), it is  $\sim 0.72$ . It is possible to assume that greater filling factor leads to greater Casimir pressure for the same distance between the surfaces, while the corrugation height does not play a significant role [23]. The topmost surface is covered with a very thin oxide layer, because the

ambience is open air. Laser interaction will enforce the oxidation, but only to the saturation level [35]. However, the results presented in Table 1 show that whichever film structure we have at the beginning, after the interaction with the laser, the creation of the nanoscale corrugation causes an increase in CA. The higher filling factor for the samples with Zr as the topmost layer should cause the CA values' relatively more significant change than for those with Ti on top. This was observed for  $15\times(\text{Zr}/\text{Ti})$ , but in the case of  $15\times(\text{Zr}/\text{Cr}/\text{Ti})$ , the long-range wetting transparency [30] means that CA is also affected by the presence of a thin Cr layer.

While LIPSS are anisotropic, the isotropy of the contact angles was not investigated. The direction of LIPSS (sample orientation) relative to the direction of recording (camera orientation) was arbitrary for each sample and undetermined. No significant difference in the left and right contact angles can be seen in Figure 3b,d,f, meaning either LIPSS and recording directions were set parallel by chance for each sample or the droplets were not significantly anisotropic. To match the directions of LIPSS and camera recording to be parallel by chance is less likely, indicating that the droplets are not significantly anisotropic. Though the anisotropy of the surface structures influences the anisotropy of the CA, with the smaller width of the structures (ripples itself), the influence is smaller [36]. Very small widths of the ripples ( $\sim 700$  nm) in the presented experiment suggest that the influence of the structure anisotropy (LIPSS) to the droplet anisotropy is not significant.

Corrugation is responsible for hydrophobicity (wetting). Classical theory demands the size of corrugation to be above micrometer level in order to achieve super-hydrophobicity. If we assume that a multilayer structure can be imagined as an optical cavity, quantum effects cause the rise of super-hydrophobicity [21,22]. Recently, experimental proof of the impact of nano-corrugation to wetting properties exploiting the Casimir effect has been published [37]. The effect of small corrugations and quantum effects to superhydrophobicity is phenomenologically confirmed. In our work, it has been shown that, for laser generated nano-corrugation, observed wetting properties can be explained only if quantum effects are taken into account. Corrugations of nanoscopic level dramatically affect the Van der Waals interaction energy (through quantum vacuum photon modes) and thus the wetting contact angle is modified. Up to now, to achieve super-hydrophobicity, different coatings and dozens of micro-size structures were described elsewhere [38,39]. For thin film materials, the wetting angle is nearly insensitive to the chemical nature of the immediate substrate and, by the forming of hyperbolic (plasmonic) metamaterials, the effective refractive index changed as well as the contact angle [30].

Here, the authors are the first, to our knowledge, to report significant change in the wetting properties by subwavelength corrugation. The increase in hydrophobicity caused by fs nano-patterning experimentally confirms the model developed by Delliou et al. [21,22].

Furthermore, the presented study goes beyond the proposed model, since super-hydrophobicity is achieved on the metallic surface (not only the molecular solid). In the end, the possibility of attaining super-hydrophobicity using submicron corrugation by controlling quantum vacuum modes opens large almost unlimited fundamental and technological applications. While the responsibility for the change in the dispersion component is doubtful, the corrugation is responsible for all the changes in the CA. However, the magnitude of the effects is too big for such small corrugations, which can be explained with the abovementioned non-classical approach [21,22]. Untreated metal surfaces are generally hydrophilic, but in this experiment they changed to highly hydrophobic by the implementation of sub-micrometer-sized corrugation.

#### 4. Conclusions

We have investigated the wetting of laser modified multilayer thin films of different metal materials by using contact angle measurements. The values do not differ much for different materials (both as-deposited and laser modified) but significantly change after the laser interaction and the formation of LIPSS. Here, due to nanometer-sized structuring, wetting cannot be explained without taking quantum effects into consideration. To our actual knowledge, this is the first example of significant wetting change by nanostructures and it is in good agreement with previous publication [21,22], which explains

wetting by quantum vacuum modes. This suggests that the increase in the hydrophobicity is related to the morphological changes in the cavity. The presented results extend the application of the model previously developed [21,22] for molecular solids, and show that the nanocorrugation of metal surfaces can be used to significantly affect the wetting. While the influence of quantum modes freezing to superhydrophobicity has been phenomenologically confirmed [37], we have shown that observed wetting properties of surfaces with fs laser generated non-corrugations can be explained only if quantum effects are taken into account. The potential applications of the presented research could be numerous.

**Author Contributions:** The contributions are as follows: conceptualization, B.K. and A.G.K.; methodology, B.K. and S.P.; validation, B.K. and S.P.; investigation, A.M. and S.P.; resources, E.S.; data curation, A.M. and S.P.; writing—original draft preparation, A.G.K.; writing—review and editing, A.G.K., S.P., A.M., E.S., D.P. and B.K.; visualization, A.G.K. and S.P.; supervision, E.S.; project administration, D.P., E.S. All authors have read and agreed to the published version of the manuscript.

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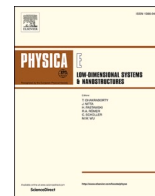


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# Structural properties of femtosecond laser irradiation induced bismuth oxide based nano-objects in Bi<sub>12</sub>SiO<sub>20</sub> (BSO) single crystal

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## ABSTRACT

Single crystal of Bi<sub>12</sub>SiO<sub>20</sub> was grown from the melt by Czochralski technique. The crystal growth was in the [111] direction. The surface of the polished sample was irradiated by a femtosecond pulsed laser beam of various power. The influence of laser power on structural properties of Bi<sub>12</sub>SiO<sub>20</sub> crystal, as well as on its phase composition, was studied. The surface morphology of our samples was investigated by AFM. The surface of unirradiated sample is rather smooth with no cracks observed. In sample modified by pulsed femtosecond beam, we registered the presence of small spherical islands on the surface. The dimensions of the islands and their density depend on the applied power. There were also significant changes in far-infrared spectra of irradiated sample in comparison to non-irradiated sample. Based on these results, the material obtained after femtosecond pulsed laser irradiation consisting of bismuth oxide based nano-objects, formed as nanocrystals (dimensions below 20 nm in diameter), which are arranged in a matrix of Bi<sub>12</sub>SiO<sub>20</sub>.

## 1. Introduction

Sillenites (Bi<sub>12</sub>MO<sub>20</sub>, M = Si, Ge, Ti) are optically active crystals exhibiting a lot of strong effects (optical rotation, electro-optical (Pockels), magneto-optical (Faraday) and photo-induced effects) and interesting properties such as remarkably large values of dielectric, piezo-electric and elasto-optic constants, very high values of the dark electric resistance, the index of refraction [1] etc. These crystals have application as active elements in many devices [2]. For these applications the materials are bulk single crystal samples.

On the other hand, due to their extremely small sizes, nanomaterials (one, two or three dimensions of less than 100 nm) cannot be used in large scale, particularly as long-bearing materials in engineering applications. For this it has long been a desire to develop bulk composites incorporating these nanomaterials (for example nanocomposites) to harness their extraordinary properties in bulk applicable materials. Initial ideas and principles are given in Ref. [3]. The most important fact is that the characteristics of the nanomaterials are fundamentally different in comparison with the bulk materials [4].

Lasers play an ever expanding role in material processing [5], as is the case with surface treatment of single crystals [6] where the energy of a laser beam interacts with a material to transform it in some way in a

thin surface layer. This transformation (or laser process) is controlled by precisely regulating the wavelength, power, duty cycle and repetition rate of the laser beam. All materials have unique characteristics that dictate how the laser beam interacts and consequently modifies the material [7,8].

In our previous papers, we have investigated the influence of locally induced heating with increasing laser power densities on some nano-materials such as stable hexagonal transition oxides ZnO doped with CoO [9] and cubic rock-salt MnO [10]. The influence of femtosecond pulsed laser power on the quality and optical characteristics of Bi<sub>12</sub>GeO<sub>20</sub> single crystal was also studied [11].

The aim of this work is to continue our research with investigation influence of femtosecond pulsed laser irradiation on Bi<sub>12</sub>SiO<sub>20</sub> single crystal using FTIR spectroscopy along with atomic force microscopy (AFM), but this time the focus is on modification of material and its structural characterization.

## 2. Experimental procedure

### 2.1. Preparation of crystal samples

Czochralski technique was applied to grow Bi<sub>12</sub>SiO<sub>20</sub> single crystal,

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where MSR 2 crystal puller controlled by a Eurotherm was used with temperature fluctuations of the experiment lower than 0.2 °C. Additional weighing set was used to monitor the crucible weight in order to keep a crystal diameter constant (absolute deviation was below 0.1 mm).

A platinum crucible was used to contain the melt, which was placed in an alumina vessel on a zircon – oxide wool. This system was constructed in order to stop the excessive radiation heat losses. Also, a cylindrical silica glass after heater was constructed around the system to reduce the thermal gradients in the crystal and in the melt. Crystal growth was occurred in an air atmosphere while iridium wires were used as initial crystal seeds. Later on, seed cuts from the produced  $\text{Bi}_{12}\text{SiO}_{20}$  crystals were used for the growth of other crystals.

$\text{Bi}_2\text{O}_3$  and  $\text{SiO}_2$  were used for synthesis of crystals. Starting materials were mixed in 6:1 stoichiometric ratio. Optimal pull rate was chosen in the range 5 – 6 mm/h. Equations of the melt hydrodynamics were used to calculate critical crystal diameter,  $d_c = 10$  mm and critical rotation,  $\omega_c = 20$  rpm. The crucible was not rotating during crystal growth. The crystal boule was cooled at  $\sim 50$  °C/h down to a room temperature, after the crystal growth. Crystals grew in [111] direction, without core being observed. Finally, crystals were cut and polished.

## 2.2. Crystal irradiation and characterization

Crystal samples were exposed to a pulsed femtosecond laser beam (pulse width 90 fs, repetition rate 76 MHz) from Coherent Mira 900 F laser system pumped by a 532 nm continuous wave Coherent Verdi V-10 laser. The irradiating beam wavelength was monitored by an Ocean Optics HR2000CG UV-NIR spectrometer. The samples were irradiated along their longest axis, z, i.e., along the crystal growth direction. During irradiation, the crystal facet was partially exposed due to the oval shape of the beam profile. A graded filter was used to adjust the beam power on the sample from 50 to 800 mW (measured by Ophir Nova II powermeter with thermal and photometric heads), which corresponds to the fluence range of 75–1200 nJ/cm<sup>2</sup>. Exposure time of each irradiation power was 3s, measured by a stopwatch of 0.2s of accuracy. The total irradiation time and energy were intentionally kept low to avoid significant contribution of an accumulative process caused by repopulation of the traps [12].

Far-infrared reflection spectra were recorded in the wave number range up to 650 cm<sup>-1</sup> utilizing an A BOMEM DA - 8 FTIR spectrometer with a deuterated triglycine sulfate (DTGS) pyroelectric detector.

The surfaces of samples were examined in detail using Atomic Force Microscope (AFM), NTEGRA prima from NT-MDT. NSG01 probes with a typical resonant frequency of 150 kHz and 10 nm tip apex curvature radius were used.

The X-ray diffraction (XRD) data for  $\text{Bi}_{12}\text{SiO}_{20}$  single crystals was measured using X-ray diffractometer (XRD) Rigaku Ultima IV, Japan, with filtered  $\text{CuK}\alpha_1$  radiation ( $\lambda = 0.154178$  nm). The X-ray diffraction data were collected over the  $2\theta$  range from 20° up to 80° with the step of 0.02° and scanning rate of 2°/min. The PDXL2 v2.0.3.0 software [13], with reference to the diffraction patterns available in the International Center for Diffraction Data (ICDD) [14] was used for the phase identification and data analysis.

## 3. Results and discussion

### 3.1. Femtosecond pulsed laser modification

In order to establish the behaviour of the sample material under the influence of femtosecond beam, three wavelengths from the common range of the Mira device (700–900 nm) have been chosen. The samples were exposed to femtosecond beam of 730, 800 and 830 nm, with input powers of 50–700, 50–800, and 50–550 mW, respectively. For each wavelength, the transmitted power vs input power has been monitored. The input power has been gradually increased and in this way the

influence of possible strong modifications by higher power to the results of low power was diminished. Transmitted vs. input power dependency was established (Fig. 1).

Transmitted power dependence on the input power is in fact linear for each chosen wavelengths. The linear dependence shows uniform absorption during the input power change – there were no significant damages in the material of the samples caused by the beam during exposition. If present, strong or large-scale structural changes inside the material would change the absorption coefficient which would lead to the deviation of the  $P_{out}$  vs.  $P_{in}$  (Fig. 1) dependence from the linear one.

Because of that, in the further analysis in this paper, under treated sample we will consider the sample modified with a laser line of 730 nm and a power of 700 mW as a representative one. In Fig. 2, a sample treated with 730 nm and a power of 50 mW was analyzed for comparison.

### 3.2. AFM

Fig. 2 shows the results of AFM measurements of the  $\text{Bi}_{12}\text{SiO}_{20}$  single crystal and the same sample after being irradiated by a femtosecond pulsed laser beam. The surface of unirradiated sample is rather smooth with no cracks observed, and only traces of mechanical polishing can be seen in Fig. 2a. Fig. 2b and c. Show the surface of the sample after irradiated by a femtosecond pulsed laser with 730 nm and power of 50 mW and 700 mW respectively. Full lines at Fig. 2 a-c present directions in which the structure dimensions were determined. Height profile on the surface and in the shine dots is shown in Fig. 2 d. For the treated samples, the nanoobject clearly stands out in the selected direction. It could be said that the height of the nano-object for both samples is in the range of about 10–15 nm, and that the height of the nanoobject increases with the increase in the power of the femtosecond laser. On the other hand, the diameter of these nano-objects is about 20 nm (Figs. 2b) and 15 nm (Fig. 2c). In addition, we note that the density of nano-objects is significantly higher in Fig. 2c.

Average roughness (Ra) for the samples shown in Fig. 2 a, b, c is 1.25 nm, 1.57 nm and 1.6 nm, respectively. Although the value for Ra is relatively small, i.e. the surface of the samples is relatively smooth, we can conclude that as a result of the femtosecond laser treatment, as well as when increasing the laser power, the value for Ra also increases, which is expected.

### 3.3. XRD measurements

Phase analysis using XRD of single crystal BSO samples are presented

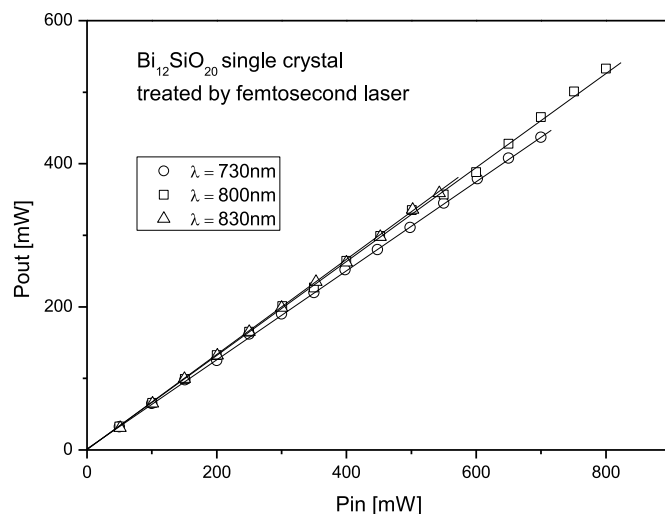


Fig. 1. Transmitted ( $P_{out}$ ) vs. input ( $P_{in}$ ) power for samples exposed to the beam of 730 nm, 800 nm and 830 nm.

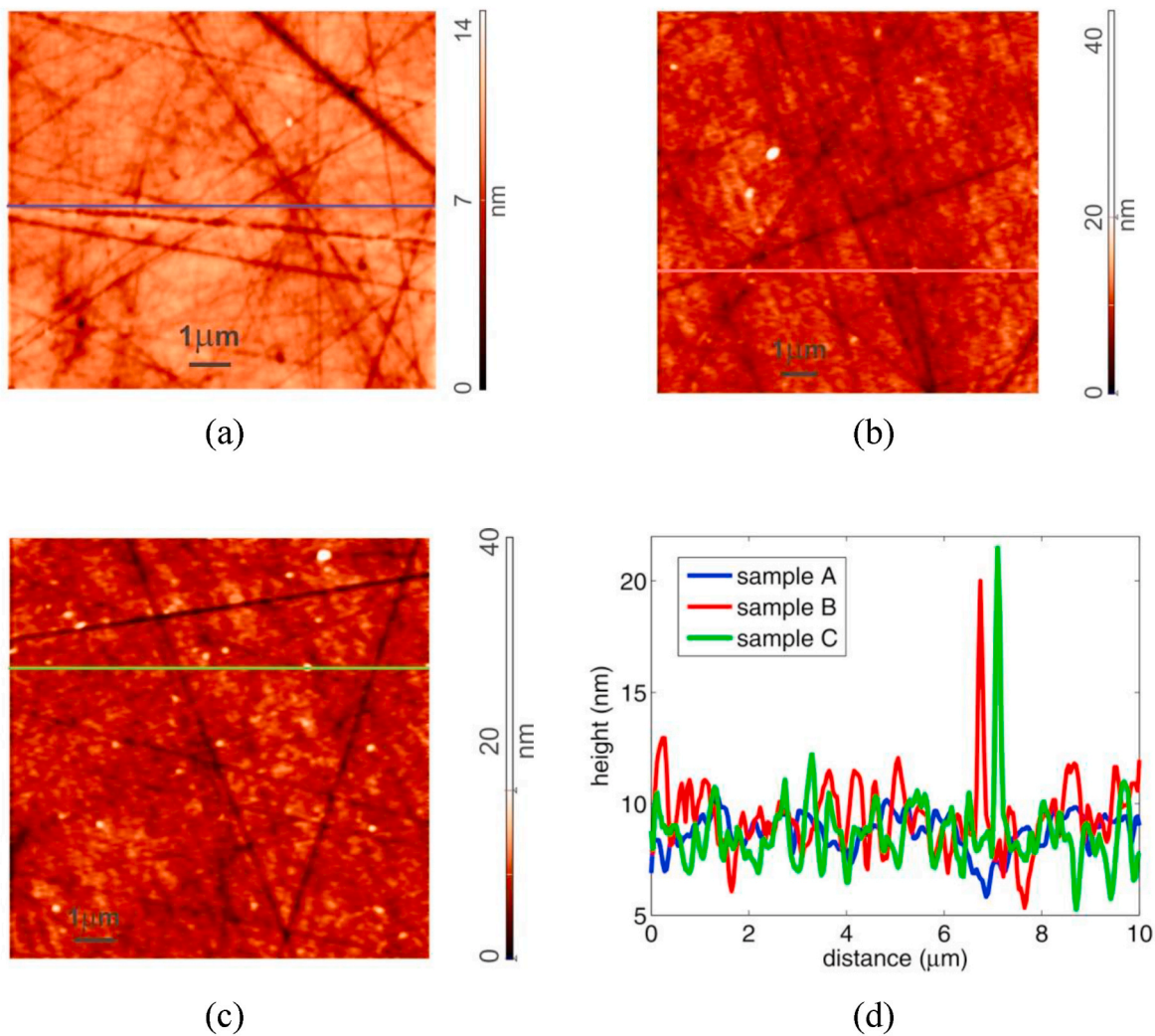


Fig. 2. AFM results of  $\text{Bi}_{12}\text{SiO}_{20}$  single crystal: untreated (a); femtosecond laser treated sample: 730 nm, 50 mW (b) and 730 nm, 700 mW (c). Height profile on the surface (d).

in Fig. 3. Phase analysis indicates that all peaks belong to the  $\text{Bi}_{12}\text{SiO}_{20}$  phase, which is in good agreement with the JCPDF Card No. 37-0485.

The XRD for the treated sample is no different from that for the untreated.

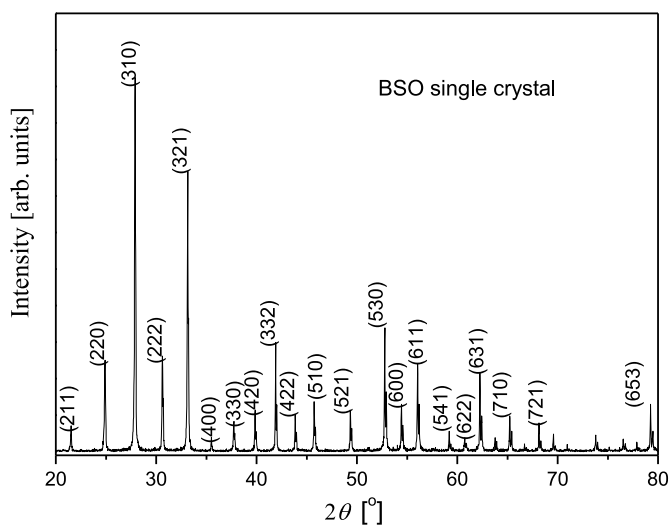


Fig. 3. X-ray diffraction results of untreated  $\text{Bi}_{12}\text{SiO}_{20}$  single crystal, peaks of  $\text{Bi}_{12}\text{SiO}_{20}$  phase marked with hkl.

### 3.4. Far-infrared spectroscopy

The experimental far-infrared spectrum of BSO single crystal was recorded in the spectral range of  $70\text{--}650\text{ cm}^{-1}$  at room temperature and in Fig. 4 is presented as a blue line. The obtained spectrum shows all characteristics described in the literature [15,16]. The far-infrared spectrum of the femtosecond laser treated BSO, recorded in the spectral range of  $70\text{--}650\text{ cm}^{-1}$  at room temperature, is presented in Fig. 4 as a red line. Even though the spectra given in Fig. 4 were recorded under the same conditions, differences in the BSO single crystal and femtosecond laser treated BSO spectra are clearly visible at several places, such as about 130, 180, 280  $\text{cm}^{-1}$  ....

$$\Gamma = 8A + 8E + 25F \tag{1}$$

Among these modes, only the F modes are infrared active.

Fig. 5, lower spectrum, shows the far-infrared spectra of BSO single crystal. The points are given the experimental results, and the solid line is obtained in the standard way by the procedure of fitting parameters [18,19]. Due to the large energy gap ( $E_g = 2.57\text{eV}$ ) and accordingly very low concentrations of free carriers, a dielectric function was used which takes into account only the interaction of electromagnetic radiation with

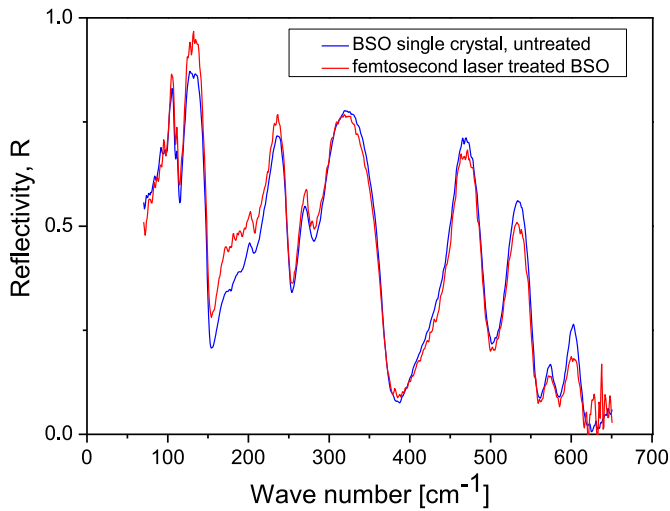


Fig. 4. Experimental far-infrared reflection spectra of  $\text{Bi}_{12}\text{SiO}_{20}$  single crystal untreated (blue line) and treated by femtosecond beam (red line). First, in short about factor group analysis. Crystal BSO has a cubic unit with space group I23 (T3) [17].

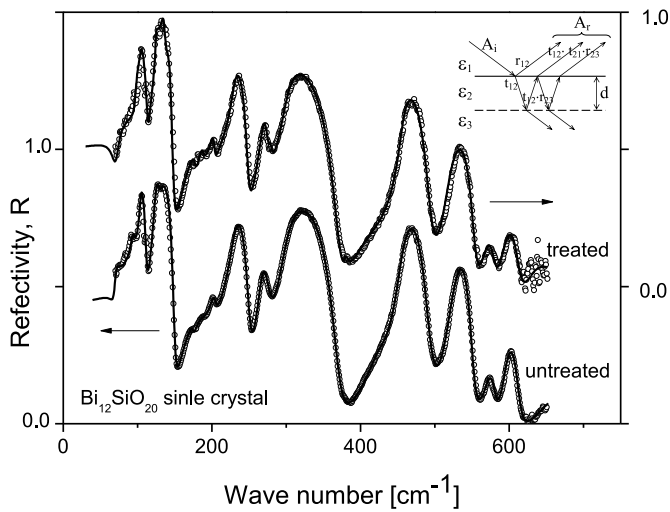


Fig. 5. Far-infrared reflection spectra of  $\text{Bi}_{12}\text{SiO}_{20}$  single crystal and femtosecond laser-treated  $\text{Bi}_{12}\text{SiO}_{20}$  sample. The experimentally obtained data points are depicted as circles. The theoretical spectrum obtained with the model defined by eqs. (2) and (3) and fitting procedure is given as solid line. Insert: Schematic overview of the femtosecond laser-treated  $\text{Bi}_{12}\text{SiO}_{20}$  sample.

phonons:

$$\epsilon(\omega) = \epsilon_{\infty} \prod_{k=1}^s \frac{\omega^2 + i\gamma_{kLO} - \omega_{kLO}^2}{\omega^2 + i\gamma_{kTO} - \omega_{kTO}^2} \quad (2)$$

where  $\epsilon_{\infty}$  is the bound charge contribution and is considered as a constant,  $\omega_{LOk}$  and  $\omega_{TOk}$  are the longitudinal and transverse optical – phonon frequencies, and  $\gamma_{TOk}$  and  $\gamma_{LOk}$  are the phonon dampings.

The results obtained for TO/LO frequencies (in  $\text{cm}^{-1}$ ) are: 69/71, 94/95, 104/112, 123/150, 129.5/130, 142/143, 175.2/175.5, 187.5/192.5, 202.5/204.3, 229/251, 267/276.8, 297.5/371, 424/427, 453/494, 520/551.5, 572/581, 594/613, 647/658. The agreement with the literature data [15,16] is excellent. This result serves as an introduction to the spectrum analysis for the femtosecond laser treated BSO sample, where the situation is somewhat more complex. Fig. 5, upper spectrum, shows the far-infrared spectrum of femtosecond laser treated BSO single crystal. The points are given the experimental results, and the solid line

is obtained in the following way.

Namely, as can be seen from Fig. 2b and c, laser treatment leads to a change in the surface of the samples. It seems that its composition changes in a very thin layer, but also that nanoobjects are formed inside the layer and on its surface. Therefore, we decided to use a model that takes into account the existence of a three-layer structure (see insert of Fig. 5), where.

- (a) medium 1 is air ( $\epsilon_1 = 1$ ),
- (b) medium 2 is a layer with thickness  $d$  present at the sample surface with dielectric constant  $\epsilon_2$  (eq. (2)), and
- (c) lower optically thick layer, medium 3, practically single crystal BSO, described with  $\epsilon_3$  (eq. (2)).

In this case, the reflectivity can be determined as described in Ref. [20]:

$$R_A = \frac{A_r}{A_i} = \frac{r_{12}e^{-i\alpha} + r_{23}e^{i\alpha}}{e^{-i\alpha} + r_{12}r_{23}e^{i\alpha}} \quad (3)$$

where  $r_{ij} = (n_i - n_j) / (n_i + n_j) = (\sqrt{\epsilon_i} - \sqrt{\epsilon_j}) / (\sqrt{\epsilon_i} + \sqrt{\epsilon_j})$  are the Fresnel coefficients,  $A_i$  and  $A_r$  represent amplitudes of the incident and reflection beams, respectively,  $n$  is the complex index of refraction,  $\epsilon$  is the dielectric constant and  $\alpha = 2\pi\omega d (\epsilon_2)^{1/2}$  is the complex phase change related to the absorption in the crystal layer with the thickness  $d$ . Reflectance,  $R$ , is given as  $R = |R_A|^2$ .

The parameters of the treated sample were determined by the fitting procedure. In that manner, the parameters for the single crystal BSO layer (medium 3) remained the same as those determined from untreated sample. The surface layer (medium 2), besides them, has additional modes. The layer thickness is  $d = 1,9 \mu\text{m}$ . Comparison of our result with the values from the literature for the registered additional phonons is given in Table 1.

Some results from literature, for example [22], show that laser-induced oxidation of bismuth can occur, but the degree of oxidation and the formation of the crystalline phase strongly depend on the laser power. We think that in our case, due to laser heating, on the  $\text{Bi}_{12}\text{SiO}_{20}$  single crystal, the formation of starting material phases occurs. It is known that bismuth oxide can exist in several polymorphic forms:  $\alpha\text{-Bi}_2\text{O}_3$ , the only phase stable at room temperature, and three high-temperature phases,  $\beta$ -,  $\delta$ - and  $\gamma\text{-Bi}_2\text{O}_3$ . The orthorhombic phase,  $\alpha\text{-Bi}_2\text{O}_3$ , transforms to cubic  $\delta\text{-Bi}_2\text{O}_3$  at  $729 \text{ }^\circ\text{C}$ , which may transform to tetragonal  $\beta\text{-Bi}_2\text{O}_3$  or body-centered cubic  $\gamma\text{-Bi}_2\text{O}_3$  upon cooling to  $650$  and  $639 \text{ }^\circ\text{C}$ , respectively [24–27]. Both of these forms are metastable,

Table 1

Comparison between additional far-infrared frequencies registered in this paper and experimentally and calculated frequencies from the literature.

Phonon peaks This work [cm <sup>-1</sup> ]	Experimental literature values of phonon frequencies [cm <sup>-1</sup> ]	Calculated phonon frequencies [cm <sup>-1</sup> ]	Description
120	120 [21]	120 [21]	$\text{Bi}_4\text{O}_7$ [21]
	118 [22]		$\alpha\text{-Bi}_2\text{O}_3$ [22]
161	157 [21]	124 [21]	$\beta\text{-Bi}_2\text{O}_3$ [21]
			$\gamma\text{-Bi}_2\text{O}_3$ [21]
278	166 [23]	279 [22]	$\gamma\text{-Bi}_2\text{O}_3$ [23]
	281 [21]		$\gamma\text{-Bi}_2\text{O}_3$ [22]
380	381 [21]	388 [21]	$\gamma\text{-Bi}_2\text{O}_3$ [21]
			$\text{Bi}_4\text{O}_7$ [21]

but may be stabilized at room temperature by the addition of impurities [25].

Another metastable phase, which was also registered by our measurements, is  $\text{Bi}_4\text{O}_7$ . This phase is a fully chargeordered pseudo-binary bismuth ( $\text{Bi}^{3+}$ ,  $\text{Bi}^{5+}$ ) oxide [28,29]. This mixed valence and the optical gap within the visible range (1.9eV) turns the  $\text{Bi}_4\text{O}_7$  interesting for applications in photocatalysis. Also, because of strong luminescence at about 420 nm  $\text{Bi}_4\text{O}_7$  is a candidate as for purplish-blue light emitter [30]. One of the following directions of research can be dedicated to this topic as well.

It seems to us that in this way it is clearly shown that femtosecond laser treating produces nano-objects consisting of different phases based on bismuth oxide in a matrix of  $\text{Bi}_{12}\text{SiO}_{20}$  single crystal. In the future, we will search for new functionalities, which would open up new topics and areas.

#### 4. Conclusions

We used a femtosecond pulsed laser to modify the surface on a  $\text{Bi}_{12}\text{SiO}_{20}$  single crystal growth by Czochralski technique. The treatment led to the formation of bismuth oxide based nanoobjects in the  $\text{Bi}_{12}\text{SiO}_{20}$  matrix. These nanoobjects are formed as nanocrystals with dimensions below 20 nm in diameter and about 15 nm in height. By composition, they are  $\alpha$ -,  $\beta$ -, and  $\gamma$ - $\text{Bi}_2\text{O}_3$  and  $\text{Bi}_4\text{O}_7$ . The concentration of nanoobjects increases when the power of the femtosecond laser increases. Application in optoelectronics and optical sensor industry is expected.

#### Author contributions

**N. Romcevic:** Conceptualization, Methodology, Formal analysis, Writing—Original draft preparation. **N. Paunovic, M. Lekic, A. Kovacevic, B. Vasic:** Investigation, Formal analysis. **M. Romcevic:** Investigation, Formal analysis, Writing—review and editing. All authors have read and agreed to the published version of the manuscript.

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#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper

#### Data availability

Data will be made available on request.

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# Effects of static and dynamic femtosecond laser modifications of Ti/Zr multilayer thin films

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**Abstract.** The experimental study of the static and dynamic femtosecond laser ablation of the multilayer 15x(Ti/Zr)/Si system is reported. The layer-by-layer selective laser ablation mechanism was studied by analysis of the surface morphology and elemental composition in static single pulse irradiation in a range of pulse energy from 10 to 17  $\mu\text{J}$ . The selective ablations, as number of concentric circles in modified spots are increased with the pulse energy. The boundary between the circles was shown a change in the depth, comparable to the thickness of the individual layers. Changes in the elemental composition at the edges are associated with the removal of the layer by layer. The dynamic multipulse irradiation was observed via the production of lines with laser-induced periodic surface structures (LIPSS) at different laser parameters (scan velocities and laser polarization). The spatial periodicity of the formed LIPSS depends on changes in the effective number of pulses and laser polarization, as well as the nature of the material. For better interpretation of the experimental results, simulations have been conducted to explore the thermal response of the multiple layered structure 15x(Ti/Zr) after static single pulse irradiation.

## 1 Introduction

Micron and nano-scaled surface structuring confer additional functionalities to the material in terms of mechanical improving, bioactivation and photonic selectivity. Ultrafast laser surface modification has become a powerful tool in high quality surface texturing of a wide range of materials including metals, ceramics, semi-conductors and plastics [1–4]. The material processing such as ultrafast laser-surface structuring can enable specific features of the materials including extraordinary surface wettability, reduction of friction and wear, improve corrosion resistance, colorization of metallic surface, and also improve solar cell performance and activation of biomaterials [5–8]. Simply by irradiating different materials with ultrashort laser pulses in the various ambient conditions, the following surface structures can be formed: ripples, grooves, spikes, bumps, cavities, nanoparticles and cellular structures. The creation of these structures during the laser processing is caused by thermal and non-thermal processes such as plasma formation, interference effects, Coulomb’s explosion, surface plasmon generation, surface tension gradients, as well as hydrodynamical effects [9–11].

Laser ablation of solids is a complex process of removing materials in combination by evaporation, melting, explosion and exfoliation of materials, while the interaction mechanism is determined by the nature of the material and applied laser parameters. Laser pulse duration plays a critical role in the ablation of materials. The ablation process induced by nanosecond laser pulses generates huge heat-affected zones (HAZ) with a wide molten area of metallic materials due to their large thermal diffusivities [12]. However, femtosecond laser ablation results in precise modification without collateral damages, due to suppress heat diffusion to the surroundings of irradiated regions, which significantly reduces the generation of a heat-affected zone (HAZ). In femtosecond time domain the excitation of multiphoton and avalanche ionization together with free electron heating are occurred, but without significant changing the lattice temperature [13–15].

In addition, after multiple-pulse laser ablation it is possible to generate laser-induced periodic surface structures (LIPSS) on numerous types of materials attractive for many applications. Irradiation of metallic surfaces by linearly polarised ultrashort laser pulses can induce shallow periodic corrugations with sub-wavelength spatial periods [16,17]. When the spatial periodicity of LIPSS is almost equal to the laser wave-

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length, they are classified as low spatial frequency LIPSS (LSFL). The creation of LSFL is explained by the interference between the incidence laser beam and the surface-scattered electromagnetic wave, which induces a periodic variation of laser intensity along the surface. On the other hand, high spatial frequency LIPSS (HSFL) has much smaller spatial periodicity. Several interpretations have been proposed for their creation, such as self-organization, second harmonic generation, excitation of surface plasmon polaritons, and Coulomb explosion [18–20]. Lately, the progress in the field of ultrafast laser processing based on ablation is very important, including surface micro- and nanopatterning as well as 2D-surface and 3D-volume processing.

An unusual form of the material, like nano-scaled metallic multilayer thin films, attracts attention to specific applications in areas where superficial materials are decisive factor. Mostly, metallic thin films possess specific physico-chemical and mechanical properties as high corrosion resistance, good radiation stability, satisfactory hardness and porosity. Multilayer thin films are suitable material for a wide range of application, as protective coatings, catalytic components, optical devices, photovoltaic gas sensors, dye sensitized solar cells, in biomedicine as implants and tools [21–23]. Multilayer thin film is inherently metastable state and susceptible to various surface modifications, especially laser processing on the micro- and nanoscale by direct patterning in a very fast and cost-effective manner [24].

In this work, we study the possibility to achieving the selective ablation as well as the formation of LIPSS on the multilayer Ti/Zr thin films [25]. Layer-by-layer selective ablation would be predicted from experimental results obtained after a static single fs pulse irradiation. The depth and elemental composition of the ablated circular spots are comparable to the thicknesses of individual layers as a part of the multilayer structure. The ablation study of the multilayer systems is included a detail analysis of the influence of scan speed and laser beam polarization on the spatial periodicity of the formed LIPSS in ablative regime during the dynamic fs irradiation.

## 2 Experimental

The multilayer structures composed of titanium and zirconium layers were deposited in a Balzers Sputtrion II system, using 1.3 keV argon ions and 99.9% pure Ti and Zr targets. Before deposition the chamber was evacuated to the base pressure of  $1 \times 10^{-6}$  mbar, while the Ar partial pressure during deposition was  $1 \times 10^{-3}$  mbar. For substrate has selected a silicon wafer Si (100), which was cleaned by etching in HF and immersion in deionized water before mounting in the chamber. The deposition of multilayers was performed in a single vacuum run, at deposition rate of  $0.17 \text{ nm s}^{-1}$  for both Ti and Zr components, without heating of the substrates. The total thickness of the complete multilayer structure

consisted of fifteen (Ti/Zr) bilayers was 500 nm, where thickness of individual Ti and Zr layers were about 17 nm.

Laser processing of the multilayer 15x(Ti/Zr) thin films was performed by two laser systems. One is the Yb:KGW laser source Pharos SP from Light Conversion. The surface of thin films was irradiated by focused linearly p-polarised pulses with follow characteristics: repetition rate of 1 kHz, pulse duration equal to 160 fs, central wavelength of 1030 nm and  $40 \mu\text{m}$  Gaussian beam diameter. The other is Ti:S laser system Mira 900 by Coherent, a source of linearly polarised  $\sim 130$  fs pulses with 76 MHz repetition, wavelength of 860 nm and 600 nm Gaussian beam diameter. Mounted on a motorized, computer-controlled X-Y-Z translation stage, samples were processed by laser beam at normal incidence in open air environment. By irradiation of Ti/Zr samples with different pulse energy/fluence and scan velocities, i.e. number of pulses, respectively, formation of spots and lines were included. In each line, energy per pulse was assumed to be constant, since the pulse energy deviation was less than 1%.

Detailed surface morphology after irradiation was examined firstly by optical microscopy, and then by scanning electron microscopy (JEOL JSM-7500F, equipped with energy dispersive X-ray spectroscopy—EDS—by Oxford Instruments INCA, and Tescan MIRA3 SEM). The laser-modified and ablated surface profiles were studied in 2D- and 3D-modes using an optical profiler 7300 SWLI (Zygo).

## 3 Results and discussion

The effects of morphological changes induced during the static single fs pulse irradiation performed on the 15x(Ti/Zr)/Si system were examined through the spots made at the different pulse energies (Fig 1). For all applied pulse energies, a circular ablated spot with a distinct sharp boundary between unmodified and ablated areas occurred. The lowest absorbed pulse energy ( $10 \mu\text{J}$ ) exceeds the kinetic energy of the removed material, whereby the ablation occurs in the form of a very shallow and flat crater (Fig. 1a). With an increase in the pulse energy, the number of concentric circles in observed individual spots increased (Fig. 1b–d), which can be attributed to the selective ablation of the multilayer 15x(Ti/Zr) /Si system. However, the number of circles (four) is same for pulse energies of  $15 \mu\text{J}$  and  $17 \mu\text{J}$ , and with further increasing of pulse energy the number of circles remains constant, whereby for sufficiently high pulse energies the circles disappear. For the given range of pulse energies, the spot diameters had values from 27 to  $47 \mu\text{m}$ . According to the established procedure, the ablation threshold fluence  $F_{th}$  can be experimentally determined by representing squared diameter of the ablated areas  $D^2$  as a function of the logarithm of the applied pulse energies  $E_p$ , for the fs laser pulse and Gaussian distribution [26,27]. The cal-

culated ablation threshold fluence for the multilayer 15x(Ti/Zr)/Si system is  $F_{th} = (220 \pm 40) \text{ mJ cm}^{-2}$ .

Simultaneously, the selective ablation of multilayer system can be recognized in the corresponding depth profiles displayed with SEM images (Fig. 1). Maximum depth in the centre of spots is gradually rising with pulse energy. The height/depth of the removed material almost match with the thickness of as-deposited layers (thicknesses of individual layer were 17 nm), these insignificant deviations can be attributed to measurement errors ( $\sim 5\%$  deviations in the profilometric measurement). The differences in dimension (height and width) of the ablated steps originated from a different distribution of energy in Gaussian profile for given pulse energy. The boundaries between the ablated steps closer to the central part of the spot indicate a partial melting of Ti and/or Zr layers (Fig. 1c, d), also can be consequence of Gaussian energy distribution. The arrangement of concentric circles corresponding to selective ablation can be related to Gaussian spatial beam fluence profile, which is schematically presented in (Fig. 2.) [28]. The fluence of each ablated area was estimated from Gaussian profile (Fig. 2) and calculated by  $F = F_0 \exp(-2r^2/\omega_0^2)$  equation for surface modification obtained at a pulse energy of  $17 \mu\text{J}$  and the fluence at the centre of  $597 \text{ mJ cm}^{-2}$  [28]. The estimated values of ablated steps from periphery to the centre are  $F_1 = 342 \text{ mJ cm}^{-2}$ ,  $F_2 = 418 \text{ mJ cm}^{-2}$  and  $F_3 = 503 \text{ mJ cm}^{-2}$ .

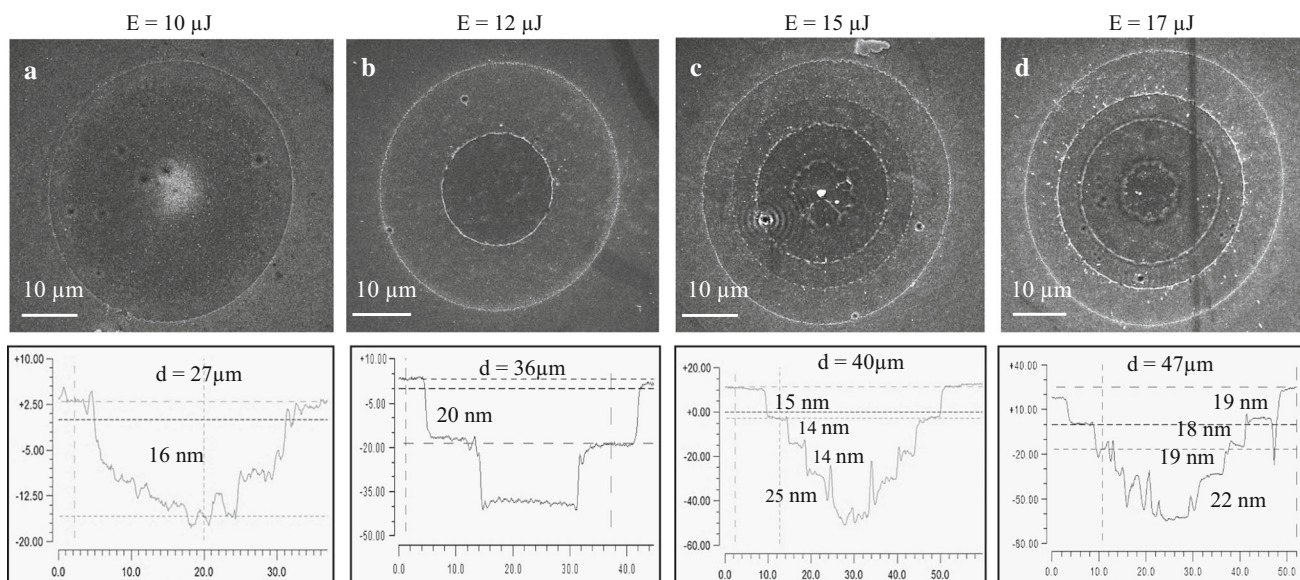
The EDS analysis of the 15x(Ti/Zr) /Si multilayer system was performed in order to compare the differences in the elemental composition of unmodified multilayer thin film and the ablated steps for the spot made at the average pulse energy of  $17 \mu\text{J}$ . The EDS spectra recorded at particular points in the different steps are given in Fig. 3. Obviously, the ablation effects observed from periphery to the centre of the spot are confirmed by gradually increasing of the relative concentration of silicon [in spectrum 2, peak for Si (Fig. 3)], is attributed to the contribution of the substrate in regard to unmodified area of the 15x(Ti/Zr) /Si multilayer system. However, the relative concentrations of Ti and Zr change quite differently, in the ablated steps where the concentration of Ti decreases, the concentration Zr remains unchanged and vice versa. The concentration of Ti decreases in first and third steps, while in the second step is recorded the reduction of the Zr concentration. These distribution of the Ti and Zr contents through the ablated steps indicate that the layer-by-layer selective ablation are occurred during the static single fs irradiation of multilayer 15x(Ti/Zr) /Si thin film.

In the fourth central step, the concentrations of Ti and Zr simultaneously decrease, indicating the peak fluence is high enough to cause the mixing of components between the layers. Additionally, with decreasing concentrations of the main components (Ti and Zr) from periphery to centre of spot, the quantity of bonded oxygen is increased, which is also associated with Gaussian energy distribution for given pulse energy. Due to the intermixing of components and higher content of oxy-

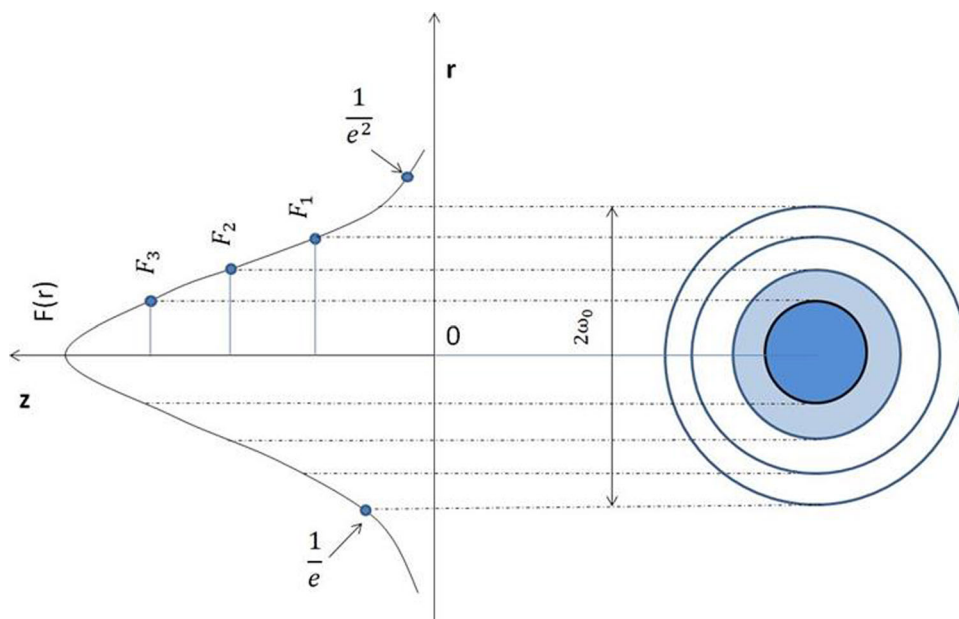
gen in the central area of ablated spots, it is expected that an ultra-thin layer composed of Ti and Zr oxide phases is formed at the bottom of the ablated centre [29].

Effects of the dynamic fs modification of multilayer 15x(Ti/Zr) /Si system is considered during the formation of lines with different scan velocities at the constant pulse energy ( $2.5 \mu\text{J}$ ) and with laser beam polarisation parallel to scan direction. For the selected scan velocities range ( $0.5\text{--}3 \text{ mm s}^{-1}$ ), in all cases, the creation of the laser-induced periodic surface structures (LIPSS), oriented normally to the direction of laser polarization have occurred (Fig. 4). In the midline, the well-defined LIPSS (LSFL) are created, originating from an interference of the incident laser beam with surface electromagnetic wave excited during the laser irradiation [30]. Created ripples are oriented parallel to the scan direction and their length almost coincides with the width of the lines for lower scan velocities. The formation of ripple surface structures is followed with the significant ablation of multilayer 15x(Ti/Zr) /Si as well as hydrodynamical effects system, but ripples are somewhere covered with nanoparticles with dimensions of up to 100 nm. The ripples mainly are formed on the surface of Ti/Zr thin film although the ablation of materials is significant, at the highest scan velocity with the effective number of pulses was  $N_{\text{eff}} = 266$  (Fig. 4a). The concentration for Ti and Zr components recorded by EDX method, are reduced (for 40 % wt.) in compare to the unmodified surface of the multilayer 15x(Ti/Zr) /Si thin film. With a reduction of the scan velocity, the ablation of the material is enhanced, whereby the Ti/Zr thin film for the lowest velocity and the highest effective number of pulses ( $0.5 \text{ mms}^{-1}$  and  $N_{\text{eff}} = 1600$ ) is completely removed and the ripples are formed on a silicon substrate (Fig. 4d). Furthermore, in the midline, made with the highest pulse number, a starting stage of the regularity failure of the ripple structure and/or formation of the grooves can be recognized as appearance of clusters on the surface of silicon. In this case, the EDS analysis was shown that the concentrations of Ti and Zr components dropped to zero, while the concentration of Si increased with the presence of oxygen in a relatively low concentration (about 5% wt.).

The fs modification in the form of lines, obtained with another laser polarisation (changed for  $90^\circ$ ) and with all laser parameters same as in the previous case, has shown a very similar morphological characteristics and composition changes (Fig. 5). The ripples as LSFL are oriented along the scan direction, but perpendicular to the laser polarization. For higher number of applied pulses, the ablation becomes more intense, e.g. with an effective 1600 pulses for a scan velocity of  $0.5 \text{ mm s}^{-1}$ , the thin film is completely ablated and the ripple structure is formed on silicon. For the given laser polarisation, the modulation of ripples can be recognized in the sense that their length changes for the different scan velocities. This modulation can be associated with the number of overlapped pulses for various scan velocities, which would mean that the ripple lengths would be shortened with the scan velocities due to the



**Fig. 1** SEM images and corresponding profiles at the surface of 15x(Ti/Zr)/Si multilayer system for spots made by femtosecond laser pulses at different pulse energies in follow range **a** 10 μJ, **b** 12 μJ, **c** 15 μJ and **d** 17 μJ

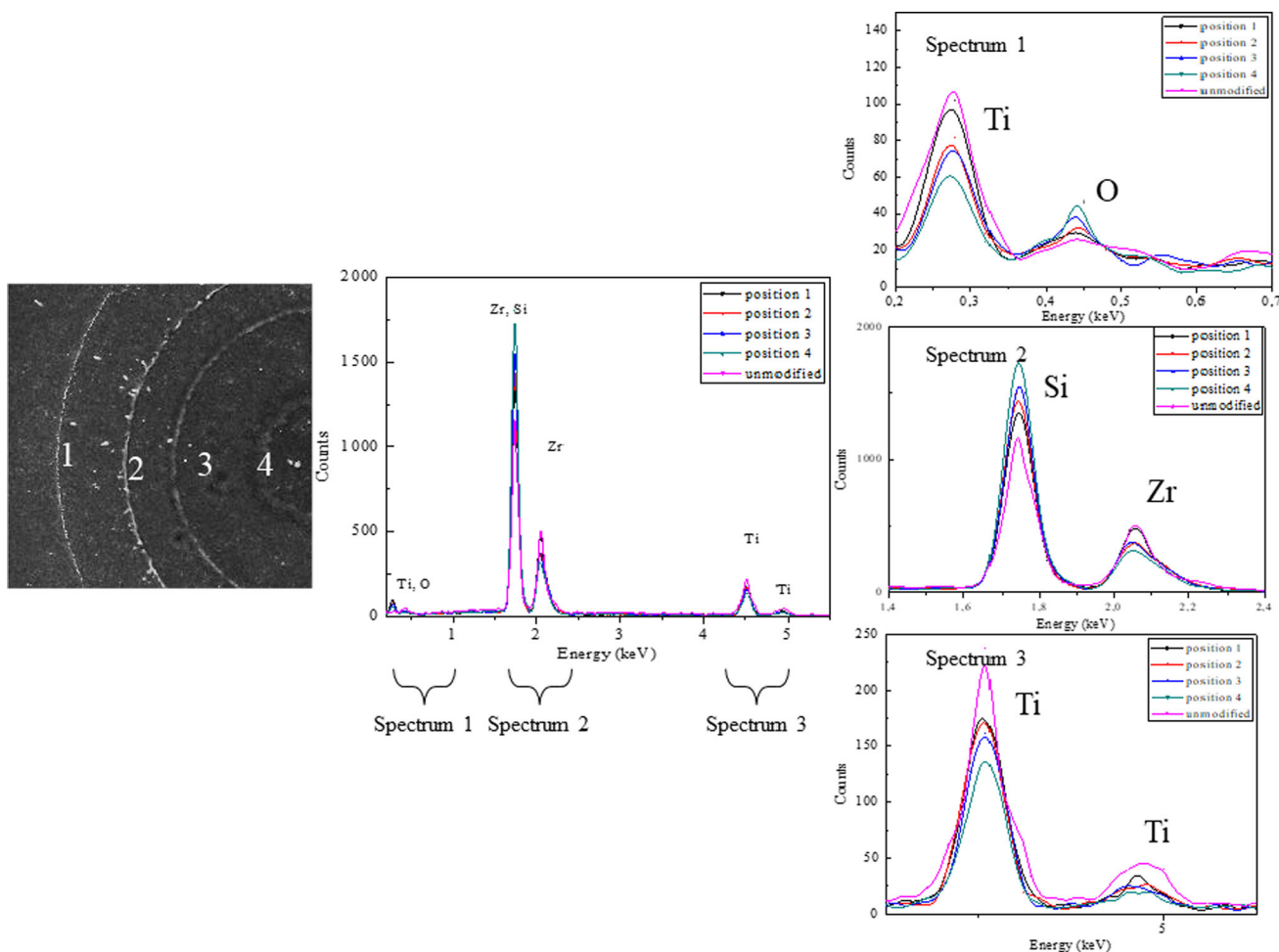


**Fig. 2** Illustration of Gaussian fluence profile for laser ablation at pulse energy of 17 μJ

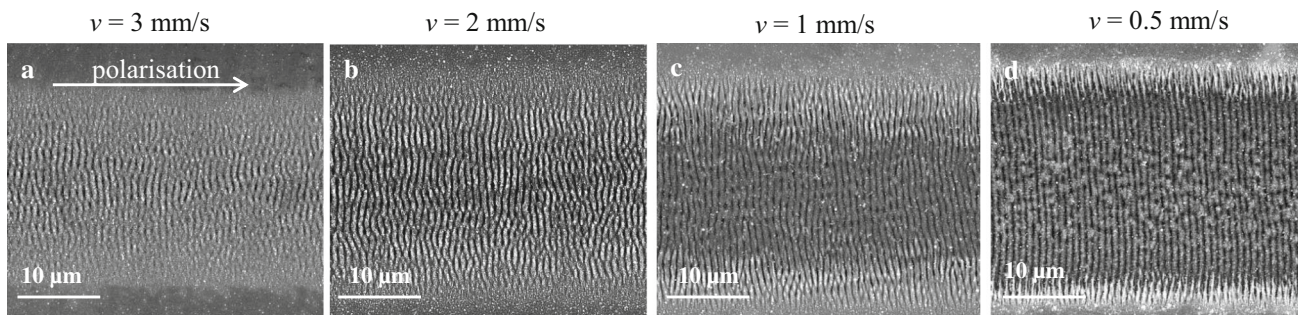
increase of the overlapping factor. The ripple lengths are in the range of 4–9 μm, where one number of ripples is ended or other can be continued from bifurcation points. These values of ripple length coincide with the mean free path of excited surface plasmon polaritons (SPP) for Ti irradiated with laser pulses at wavelength of 1030 nm [30]. It has been found that titanium has small SPP mean free path, which supports good coherence between excited SPP and incident laser radiation, favouring the formation of high regularity LIPSS [31].

The lines drawn by laser beam at different scan velocities, which is actually dynamic multipulse irradiation with different effective number of pulses, induce the

creation of the laser-induced periodic surface structures (LIPSS) with different spatial periodicity. The spatial periodicities of the created LSFL at the given range of effective number of pulses have shown tendency to reduction with their increasing, for both used laser polarizations (Fig. 6). The reduction in the spatial period is observed up to 800 pulses, afterwards for the highest applied pulses (1600), in fact at the smallest scan velocity (0.5 mm s<sup>-1</sup>), the spatial period in both cases is increased. The sudden increase of the spatial periods can be associated with the fact that the ripple structures for a given scanning speed of 0.5 mm s<sup>-1</sup> were created on a silicon substrate due to inten-



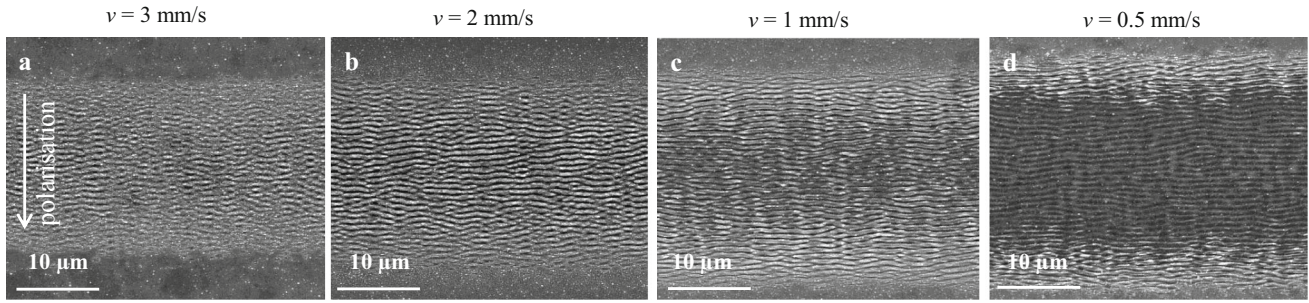
**Fig. 3** EDS spectra for the marked positions of the ablated steps at the surface of the multilayer 15x(Ti/Zr) /Si system in the spot made by fs modification at 17 μJ average pulse energy



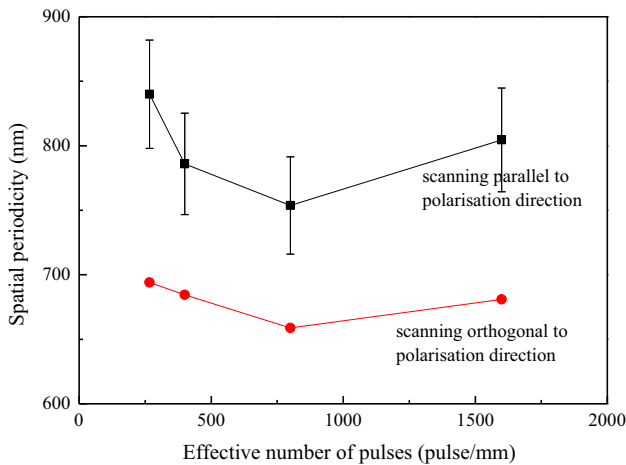
**Fig. 4** SEM images for the created lines during the fs modification at the pulse energy of 2.5 μJ with different scan velocities: **a** 3 mm s<sup>-1</sup>, **b** 2 mm s<sup>-1</sup>, **c** 1 mm s<sup>-1</sup> and **d** 0.5 mm s<sup>-1</sup>

sive ablation of the multilayer 15x(Ti/Zr) /Si thin film. On the other hand, in the case when the scan was performed parallel to the polarization direction, the periodicity has a value higher approximately 100–130 nm compared to the orthogonal scan in the polarization direction (Fig. 6). Based on this experimental observation, it can be established that the spatial periodicity of the formed LIPSS (LSFL) is quite sensitive to changes in laser parameters (the effective number of

pulses and laser polarization), as well as the nature of the material. The spatial periods varied in a wide range, which depended on the number of generated carriers in the conduction band of the laser excited materials, and the applied laser pulses [32]. The density of generated carriers for excited multilayer 15x(Ti/Zr)/Si thin film increases with increasing number of pulses, consequently the ripples with lower periods occurred.



**Fig. 5** SEM images for the created lines during the fs modification at the pulse energy of 2.5 μ J with changed laser polarisation for 90° at the different scan velocities: **a** 3 mm s<sup>-1</sup>, **b** 2 mm s<sup>-1</sup>, **c** 1 mm s<sup>-1</sup> and **d** 0.5 mm s<sup>-1</sup>



**Fig. 6** Spatial periodicities of LIPSS in function of effective number of pulses, after fs modification with different laser polarisation

### 4 Theoretical model-simulation procedure

To interpret the experimental observations, simulations have been conducted to explore the thermal response of the multiple layered structure 15x(Ti/Zr) after irradiation with single laser pulses of pulse duration  $\tau_p = 160$  fs and wavelength  $\lambda_L = 1030$  nm. It is also noted that the multilayered structure is placed on a silicon substrate (in which  $T_L^{(Si)}$  corresponds to its lattice temperature). Due to the fact that the laser spot radius is substantially larger than the film thickness, an 1D-Two Temperature Model (TTM) [33] can sufficiently describe the [34] relaxation process following electron excitation due to laser heating through the following equations:

$$\begin{aligned}
 C_e^{(i)} \frac{\partial T_e^{(i)}}{\partial t} &= \frac{\partial}{\partial z} \left( k_e^{(i)} \frac{\partial T_e^{(i)}}{\partial z} \right) - G_{eL}^{(i)} (T_e^{(i)} - T_L^{(i)}) \\
 &+ S^{(i)}(z, t) \quad [S^{(i)} = 0, \text{ for } i > 1] \\
 C_L^{(i)} \frac{\partial T_L^{(i)}}{\partial t} &= \frac{\partial}{\partial z} \left( k_L^{(i)} \frac{\partial T_L^{(i)}}{\partial z} \right) + G_{eL}^{(i)} (T_e^{(i)} - T_L^{(i)}) \quad (1)
 \end{aligned}$$

$$\begin{aligned}
 S^{(1)}(z, t) &= \frac{\alpha(1 - R - T)\sqrt{4 \log 2} F}{\sqrt{\pi} \tau_p} \\
 &\exp \left( -4 \log 2 \left( \frac{t - 3\tau_p}{\tau_p} \right)^2 \right) \exp(-\alpha z) \quad (2)
 \end{aligned}$$

while  $C_L^{(S)} \frac{\partial T_L^{(S)}}{\partial t} = \frac{\partial}{\partial z} \left( k_L^{(S)} \frac{\partial T_L^{(S)}}{\partial z} \right)$  describes the heat diffusion in the Si substrate. In Eqs. 1–2,  $T_e^{(i)}(T_L^{(i)})$  stands for the electron (lattice) temperature of layer  $i$  ( $i = 1, 3, 5, \dots, 2n - 1$  for Ti layer,  $i = 2, 4, 6, \dots, 2n$  for Zr layer, for  $n = 15$  Ti/Zr multilayer system). The thermo-physical properties of the material such as electron and lattice heat capacity,  $(C_e^{(i)}, C_L^{(i)})$ , electron and lattice heat conductivity  $(k_e^{(i)} \equiv k_{e0}^{(i)} \left( B^{(i)} T_L^{(i)} / \left( A^{(i)} (T_e^{(i)})^2 + B^{(i)} T_L^{(i)} \right) \right), k_L^{(i)} \sim .01 k_e^{(i)})$ , electron–phonon coupling strengths  $(G_{eL}^{(i)})$  and model parameters used in the simulations are listed in Table 1. For Si,  $C_L^{(S)} = 10^6(1.978 + 3.54 \times 10^{-4} T_L^{(S)} - 3.68 (T_L^{(S)})^{-2})$  [Jm<sup>-3</sup>K<sup>-1</sup>] and  $k_L^{(S)} = 158500 (T_L^{(S)})^{-1.23}$  [Jm<sup>-1</sup>s<sup>-1</sup>K<sup>-1</sup>] [17]. Equations 1, 2 are solved by using an iterative Crank–Nicolson scheme based on a finite-difference method. For initial conditions, we choose thermal equilibrium at  $T_e(z, t = 0) = T_L(z, t = 0) = 300$  K. Adiabatic boundary conditions are considered on the surface (at  $z = 0, k_e^{(Ti)} \frac{\partial T_e^{(Ti)}}{\partial z} = k_L^{(Ti)} \frac{\partial T_L^{(Ti)}}{\partial z} = 0$ ). Furthermore, at the interface between two layers, the following conditions are applied:  $T_L^{(Ti)} = T_L^{(Zr)}$ ,  $T_e^{(Ti)} = T_e^{(Zr)}$ ,  $k_L^{(Ti)} \frac{\partial T_L^{(Ti)}}{\partial z} = k_L^{(Zr)} \frac{\partial T_L^{(Zr)}}{\partial z}$ ,  $k_e^{(Ti)} \frac{\partial T_e^{(Ti)}}{\partial z} = k_e^{(Zr)} \frac{\partial T_e^{(Zr)}}{\partial z}$  while on the interface between the last layer (Zr) and the substrate (Si), the following boundary conditions are used:  $T_L^{(Si)} = T_L^{(Zr)}$  and  $k_L^{(Si)} \frac{\partial T_L^{(Si)}}{\partial z} = k_L^{(Zr)} \frac{\partial T_L^{(Zr)}}{\partial z}$ . We note that in the above formulation, for the sake of simplicity, the inclusion of latent heat for evaporation or melting has been neglected [17, 35].

While Eq. 2 provides the general expression of the form of the source term due to material heating with a pulsed laser that includes the absorption coefficient  $\alpha$ , the reflectivity  $R$  and the transmission coefficient  $T$  of the material, the Transfer Matrix Method [34] is used

**Table 1** Simulation parameters chosen for Ti and Zr

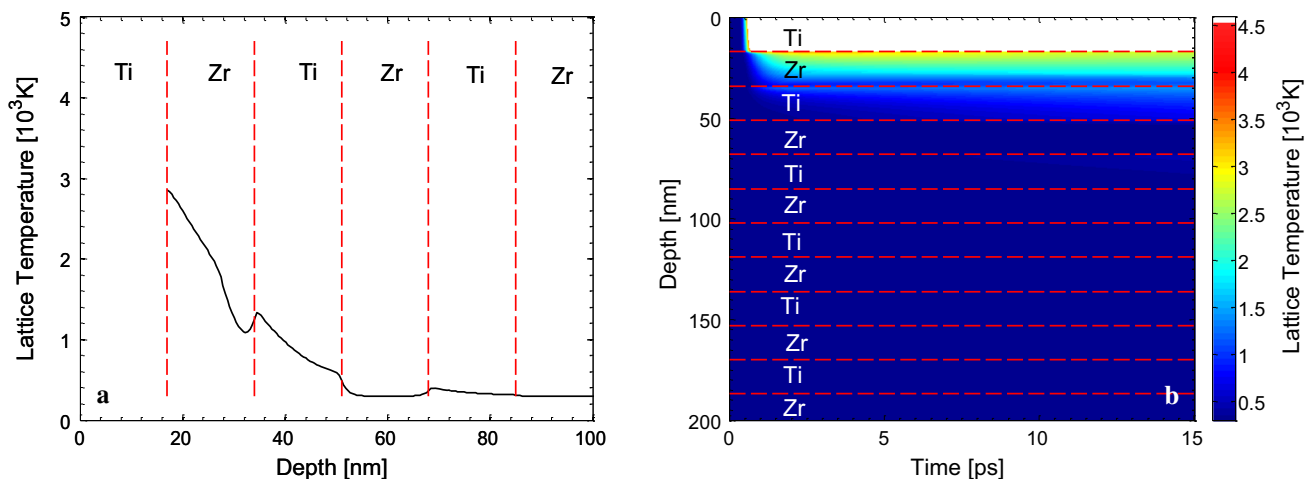
Parameter	Ti	Zr
$G_{eL}$ ( $\text{Wm}^{-3}\text{K}^{-1}$ )	Fitting [36,37]	Fitting [38]
$C_e$ ( $\text{Jm}^{-3}\text{K}^{-1}$ )	Fitting [36,37]	$2.78 T_e^{(\text{Zr})}$ [39]
$C_L$ ( $\text{Jm}^{-3}\text{K}^{-1}$ )	$2.3521 \times 10^6$ [40]	$1.6952 \times 10^6$ [40]
$k_{e0}$ ( $\text{Jm}^{-1}\text{s}^{-1}\text{K}^{-1}$ )	21.9 [40]	22.6 [40]
$T_{\text{melting}}$ (K)	1941 [40]	2128 [40]
$T_{\text{boiling}}$ (K)	3560 [40]	4650 [40]
$T_{\text{critical}}$ (K)	15500 [41]	8650 [41]
$A$ ( $\text{s}^{-1}\text{K}^{-2}$ )	Fitting [36,37]	$3.41 \times 10^5$ [38]
$B$ ( $\text{s}^{-1}\text{K}^{-1}$ )	Fitting [36,37]	$5.16 \times 10^{12}$ [38]

to compute the optical properties of the top layer (Ti) after irradiation with pulsed laser of 1030 nm by taking into account the presence of the rest of the thin layers. Calculations yield  $\alpha = 4.89 \times 10^5 \text{cm}^{-1}$  [42],  $T \cong 0$ ,  $R = 0.43$ , that indicate that  $\sim 57\%$  of the energy will be absorbed in the first layer, while the transmitted part of the laser energy into the second layer (Zr) is very small and it is not sufficiently high to excite the electrons in the rest of the layers (especially the second layer) and produce meaningful results. This argument justifies the use of a source term to describe laser heating only of the first layer and it is assumed that laser energy is not transmitted into the next layers.

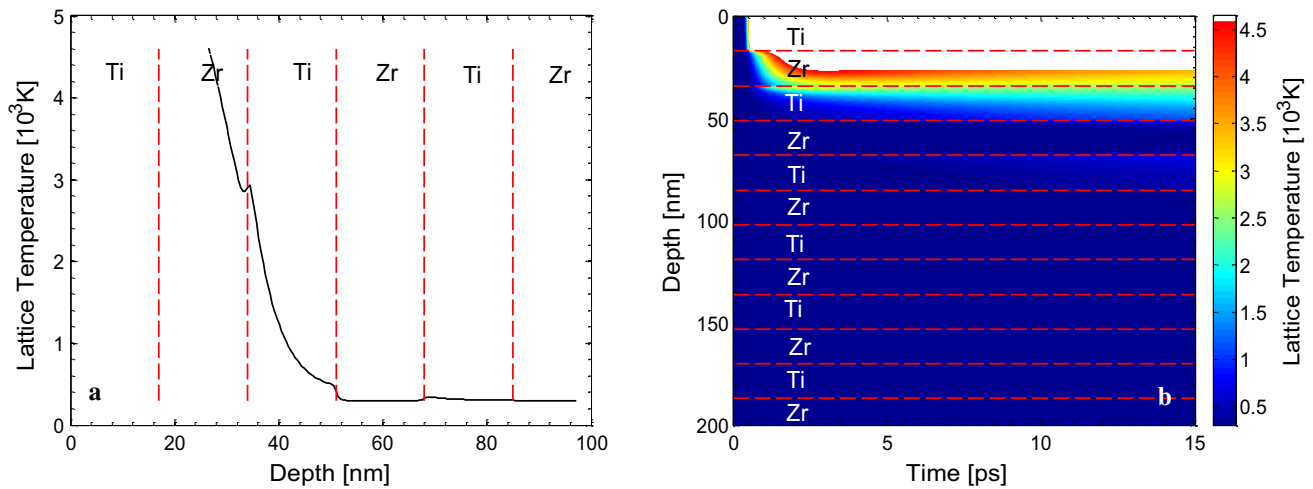
The evaluation of the thermal response of the material following irradiation with single pulses is performed through the correlation of the simulation results with the measured ablation. As noted in previous reports, ablation may be associated to the lattice temperature exceeding the condition ( $\sim 0.90T_{\text{critical}}$ , where  $T_{\text{critical}}$  is the critical point temperature [17,35,43] which is the temperature at which boundaries for gas and liquid phase vanish). Another criterion also usually

employed is the boiling temperature of the material,  $T_{\text{boiling}}$ , (i.e. the region of the material that is characterised with lattice temperatures higher than  $T_{\text{boiling}}$  is removed [44]). Finally, non-thermal mechanical stress-related processes (i.e. spallation) have also been used to describe ablation [45,46]. Our simulations and comparison with experimental observations indicate that the first layer (Ti) is totally removed at fluence  $F = 220 \text{mJcm}^{-2}$ . This value corresponds to fluence that is sufficient to raise the temperature of the upper layer above  $T_{\text{boiling}}$ ; therefore, the boiling temperature is regarded as a reliable ablation threshold criterion. Theoretical calculations of the lattice temperatures based on the scheme described above yield a spatio-temporal evolution that is illustrated in (Fig. 7a, b). The jump of lattice temperatures at the interfaces is related to the differences in the thermophysical properties of the materials (i.e. heat conductivities, heat capacities) and the electron-phonon coupling constants. It is noted that only the first three Ti/Zr layers thermally respond to the heat transfer (Fig. 7a). It is noted that to take into account ablation, all lattice points with temperatures higher than the boiling point are removed and they do not continue to heat up.

It is important to note that due to the fact that lattice temperatures on the second layer (Zr) are lower than the boiling temperature for Ti, no material is predicted to be removed from the second layer. On the other hand, it is evident that the lattice temperature attained from a large part of the second layer (Zr) for  $F = 220 \text{mJcm}^{-2}$  is above the melting point of the material (2128 K). This indicates that fluid dynamics and re-solidification processes are expected to modify further the surface profile of the assembly. Therefore, appropriate phase changes-related corrections need to be incorporated into the model for a more accurate description of the surface modifica-



**Fig. 7** **a** Spatial lattice temperature profile at  $t = 15$  ps, vertical dashed lines indicate the border of each layer. **b** Lattice temperature field evolution in depth, perpendicular to the surface of the sample (white region indicates material removal; horizontal dashed lines indicate the borders of each layer). ( $F = 220 \text{mJcm}^{-2}$ ,  $\tau_p = 160$  fs, laser beam wavelength is 1030 nm). Only six (three) of the fifteen Ti/Zr layers for the lattice temperature field (spatial temperature distribution across the depth) are presented as thermal response at greater depths is minimal



**Fig. 8** **a** Spatial lattice temperature profile at  $t = 3$  ps, vertical dashed lines indicate the border of each layer). **b** Lattice temperature field evolution in depth, perpendicular to the surface of the sample (white region indicates material removal; horizontal dashed lines indicate the borders of each layer). ( $F = 600 \text{ mJ cm}^{-2}$ ,  $\tau_p = 160$  fs, Laser beam wavelength is 1030 nm). Only six (three) of the fifteen Ti/Zr layers for the lattice temperature field (spatial temperature distribution across the depth) are presented as thermal response at greater depths is minimal

tion processes and determination of the morphological changes. A thorough approach requires the inclusion of Navier–Stokes equations (to describe fluid dynamics) and relevant equations to account for evaporation [17, 47–49]. Furthermore, in order to describe effects due to scanning an appropriate modification to the model and the intensity profile should be incorporated [50]. Certainly, a more rigorous description of the thermalization processes, a microscopic analysis of nonequilibrium phase-transition mechanisms through the use of hybrid molecular-dynamics-TTM models [51] should be considered towards providing a complete picture of the ultrafast processes.

Nevertheless, as the predominant objective of the work is to demonstrate that the laser energy used in the experiment is sufficient to remove the upper layer, simulations are performed, to first approximation, by ignoring hydrodynamics-generated effects.

The thermal response of the irradiated material is also explored for a higher value of fluence,  $F = 600 \text{ mJ cm}^{-2}$  to evaluate structural effects on the second layer (Zr). Simulation results demonstrate that apart from the upper Ti layer that is removed, a small portion of the second layer (Zr) of thickness equal to 10 nm is also ablated as the attained temperature exceeds the boiling point (Fig. 8).

## 5 Conclusion

The static and dynamic femtosecond laser ablation of the multilayer  $15x(\text{Ti}/\text{Zr})/\text{Si}$  system were experimental and theoretical analysed based on morphological and composition changes. Selective layer-by-layer ablation was recorded during the static femtosecond irradiation, applying pulse energy in range of 10–17  $\mu\text{J}$ . The circu-

lar ablated spot with a distinct sharp boundary between unmodified and ablated areas occurred in the form of shallow and flat crater. The tendency of increasing the number of ablated concentric circles continues up to pulse energies of 15  $\mu\text{J}$  and 17  $\mu\text{J}$ , whereby for sufficiently high pulse energies the circles disappeared. In the theoretical simulation of the static fs modification, it is established that the maximum achieved temperature in the first top Ti layer was comparable to  $T_{\text{boiling}}$  and the experimental observation that the Ti layer is completely removed was confirmed at the fluence of ablation threshold.

The dynamic femtosecond irradiation of multilayer  $15x(\text{Ti}/\text{Zr})/\text{Si}$  structure is considered during the formation of lines with different scan velocities at the constant pulse energy (2.5  $\mu\text{J}$ ) and with both laser beam polarisation (parallel and normal to scan direction). For the selected scan velocities range (0.5–3  $\text{mm s}^{-1}$ ), in all cases, the creation of the laser-induced periodic surface structure (LIPSS) was recorded, but with different spatial periodicity. The reduction in the LIPSS periodicity was continued up to 800 pulses, when the periodicities in both cases are increased due to the LIPSS generation on Si substrate.

The selective layer-by-layer ablation at static fs modification and LIPSS forming conditioned by the dynamic fs irradiation regime may provide an additional direction for controlling and optimizing of the laser texturing of the complex systems.

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**Data Availability Statement** This manuscript has no associated data or the data will not be deposited. [Authors' comment: All results and data are presented in this manuscript.]

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# IZABRANI PROBLEMI DANAŠNJICE U OBLASTI KOHERENTNE OPTIKE, FOTOFIZIKE I INTERAKCIJE SA ELIJSKIM ZRAČENJEM

## CHOSEN CONTEMPORARY PROBLEMS IN FIELDS OF COHERENT OPTICS, PHOTOPHYSICS AND INTERACTIONS WITH ELION RADIATION

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*Mnogobrojni metodi transformacije energije, koji se danas koriste praktično u mass media i istraživanjima u raznim savremenim disciplinama zaslužuju analize sa nekoliko aspekata. Jedan od aspekata praktične prirode je poboljšanje karakteristika konstruisanih/ostvarenih senzora i komponentata u praksi, a drugi pitanje transformacije energije i filozofski posmatranog „pozitivnog ili negativnog pravca”. U radu se analiziraju odabrani problemi i problematike, sa teorijske i praktične tačke gledišta, potkrepljene i eksperimentalnim pristupom.*

*Ključne reči: konverzija energije, metodi, otpornost materijala na zračenje, modifikacije, elion*

*Versatile methods of energy transformation, among which some of them currently are used even in mass media and in researches at different contemporary disciplines, deserve further analyzing from few aspects. One of the practical approaches is improving the characteristics of designed sensors or components which are present in praxis, while the other is the question of transforming of energy, as some would to say "from the philosophical view as positive or negative direction". In this paper are analyzing the chosen problems from the theoretical and practical view points.*

**Key words:** energy transformation, methods, material hardness, modification, elion

### 1 INTRODUCTION

The topics, which have nowadays an impact on our planet, historically are interlinking for millenia. However, this need not influence our approach to begin from the *first days* of the Earth.

On the level of fundamental subjects at technical faculties, some assessments looked futuristic at the beginning, but after 50-100-150 years; the problems commonly evaluated for the educational purposes became a main basis for investigations in a lot of branches through many terms. Some questions as like: a) what would happen to the level of hydrosphere after melting of glacier ice?, b) rate of human hair growing, c) the Earth's temperature increasing due to human activity, arose at the end of previous centuries, set down from the people dealing with black body radiation. Today they became the objects of discussion in Space investigation, for energy resources, and the *scenario* for the future is accelerating.

In Tabs 1. And 2, the parallel between some of the main energies is drawn in contemporary sources – renewable or not – and the discussions are about the question of the place of the geo-thermal energy whether it could be approximate to solar energy or not. Which of them is most acceptable? There are many parameters used for the comparison of the energy (kind, resources, etc.); which energy is more positive, which transformations are appropriate, logical, practical, technically feasible, expensive, cheap and all of those should be considered dynamically. Looking at the energy scale, the coherent energy is the most sophisticated, than follows nuclear, while chemical is somewhere in the middle, and thermal is behind them for about two levels. The direction of development

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is going from the lower to a higher, and such approach is considered as a positive, while in opposite direction logically is not recommended.

Some parallels are drawn for three energy types; the basis is to consider the processes of final energy through chain processes, beginning from the starting acts, selected attributes (or technically defined parameters) and transformations, to the final results of quantified energy. The definitions of the coefficient of merit are not included in this consideration [1, 2].

Table 1. Comparative analysis of branched chain processes.

Comparison parameter	Chemical processes	Nuclear processes	Electromagnetic processes	Comment
Super-critical processes	Self-accelerating reaction	Self-accelerating reaction of the fission processes of heavy nuclei	Self-amplification of coherent induced radiation	Term <i>Critical</i> has many interpretations
Active center (AC)	Free atom or radical	<i>Moderate neutron</i>	Quant of induced radiation	
Act responsible for multiplication	Reaction of obtaining new ACs	Fission of heavy nucleus with „neutron evaporation“	Transition of particles (↓) with emission of quant of stimulated emission	
AC quantity during one act	3	2.5 (mean value for <sup>235</sup> U)	2	Nonlinear and multi photon processes have to be discussed
Cause of chain break	AC loosing during interaction per volume on vessel walls	Parasite absorption AC through collisions and leaving the system	Parasite absorption AC through collisions in resonant system	
Conditions of super-criticality	Rate of generation of free radicals or atoms of higher velocities	Primary fission excited in mean more that one <i>post acts</i>	Number of acts of induced radiation exceeds the number of acts in no inverse milieu	
Energetic reservoir	Energy of chemical bonding	Energy of nuclear forces	Energy of excited particles in no inverse milieu	
Final result of PCP disintegration in energy reservoir	Molecular transformation with release of energy of chemical bonding	Fission of heavy nuclei with release of energy of nuclear forces	Transition of inverted milieu in equilibrium state with coherent radiation emission	

Table 2. Processes with high power extraction

Science	Type of the process	Field
Chemistry	Thermal inflammation	Branching
Physics	Thermonuclear synthesis	Chemical RCP realizes in atomic reactor and atomic explosion processes
Physics	Thermal radiation at high temperatures	Electromagnetic RCP realizes by lasing of various wavelength by transitions

## 2 APPLICATION OF COHERENT/NON-COHERENT ELION BEAMS RADIATION FOR INCREASING PERFORMANCES OF COMPONENTS (DETECTORS, SOLAR CELLS, FIBERS) AND SYSTEMS

Many experiments have dealt with the facts related to doses (exposition, absorption) of elion techniques and amelioration of components (detector, fiber, contact, integrated circuits,) characteristics. It means that various beams (of accelerated/no accelerated particles: nuclear particles, radiation, laser, maser/microwave) and their actions are evaluated through different parameters. It is important to distinguish whether the beam is coherent or not [3-18].

As stimulated processes are related to the whole electromagnetic spectrum, the parallels between spontaneous and stimulated processes/irradiations could be drawn always, because features can't be viewed only as „quantitative“ but as joint actions of electric and magnetic fields associated to the EM wave of ideal coherent (or often polarized) characteristics. The actions lead to the modulation of the organic and inorganic materials and most important, they influence the biosphere.

### 2.1 Solar cell irradiation/processed/modified by laser cells

The influence of power laser which irradiates photovoltaic cell is mostly focused to enhance their performances. Here, only few examples are presented, where modulation or laser damage are useful for laser operation. Irradiation by laser beams influences the characteristics of sensors, solar cells, fibers, etc. In Figs. 1-4, are presented 7 steps of solar cell tailored by laser beams, and chosen characteristics of laser action, some details of the processes (etching and other processing methods).

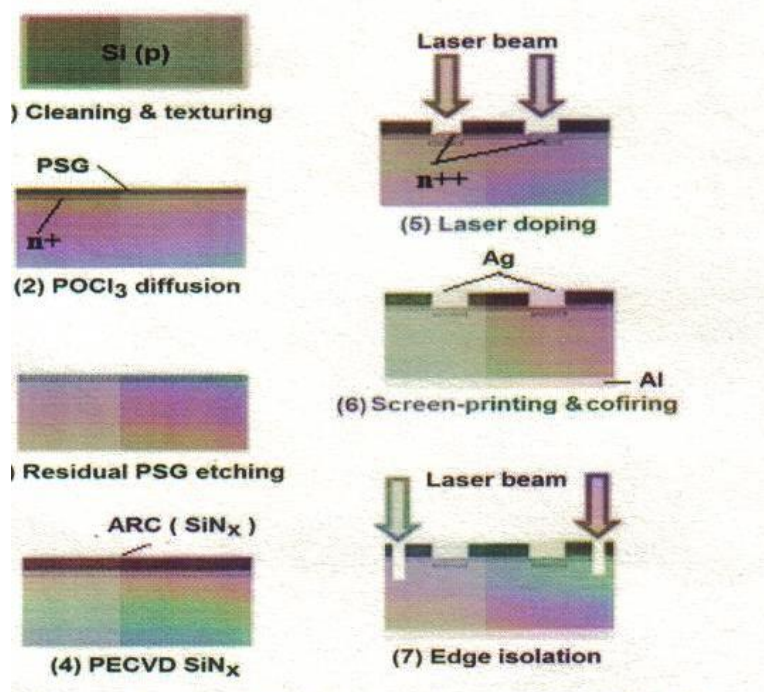


Figure 1. 7-steps processes in laser tailored emitter solar cells [14,15]

The seven steps of the process are:1:Surface cleaning and texturing, 2. Shallow doped  $POCl_3$  emitter,3. Phosphosilicate glass PSG removing, 4.  $SiN_x$  antireflection ARC deposition, 5. Laser doping, 6. Screen printing,7. Edge isolation.

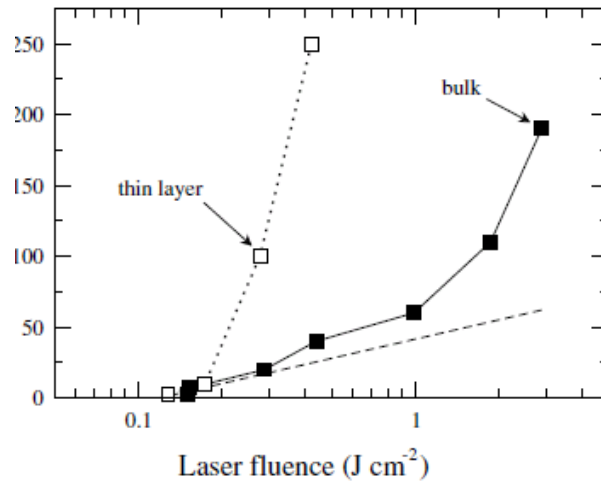


Figure 2. Ablation rate versus  $F_{las}$  laser beam fluence for femto-second laser regime [14,15 ].

Bulk Mo and a 500nm Mo layer on a glass substrate were specimens for solar cells treatment.

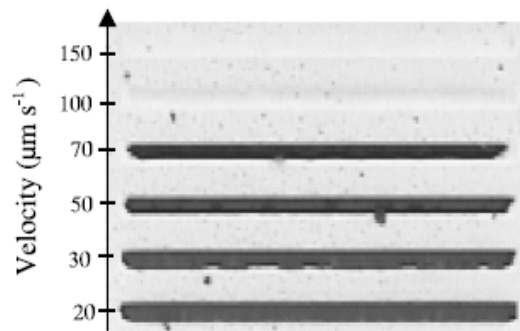


Fig. 3. Ablation channel on a Mo/glass sample machined by femto-second laser beam for velocities for  $f=50Hz$  and  $F_{las}=0.17Jcm^{-2}$  [14].

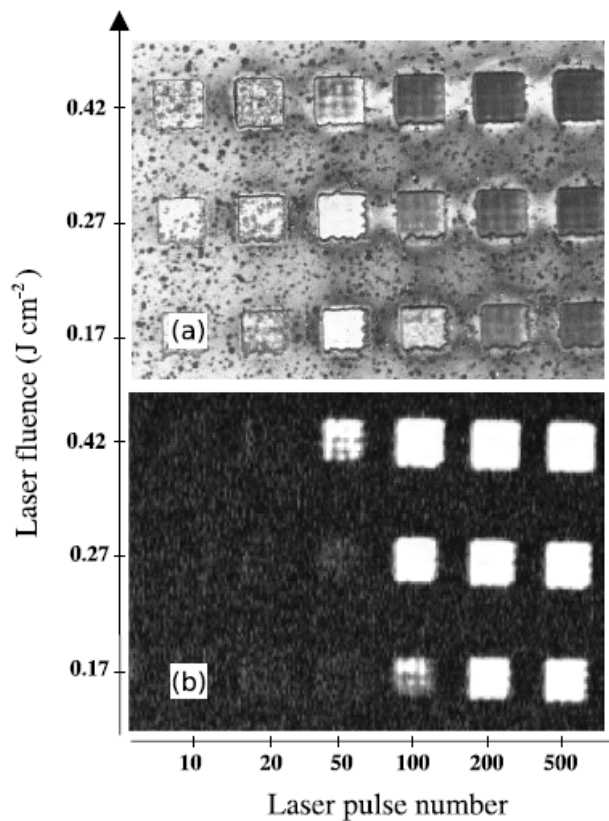


Figure 4. [14]. Morphology of material depending on laser fluence and laser pulse number.

( $Z_{max}$  etching depth is presented as the efficiency potential of SE compared to standard cells by reducing the finger with  $w_f$ . Some differences exist between simulations and measured data. The fine line metallization could be provoked efficiency increase for 0.7%, i. e. to be in the range  $\approx 18.8\%$ . [14]

Industrial application understands waver processing time as 1s, laser doping of 15.6x15.6cm wavers require pulse repetitions rates  $\approx 30$  kHz.

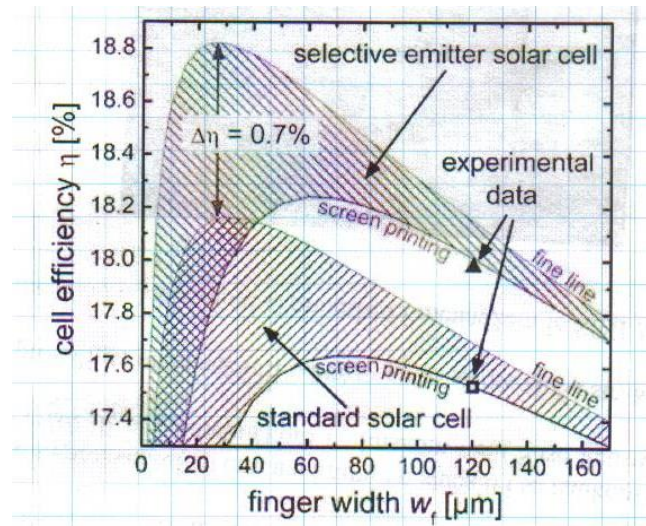


Figure 5. Simulated cell performance/ efficiency cell versus finger width  $w_f$ . [14].

## 2.2 Implementation of coherent radiation in the producing of fiber structures

**a) Dimension control** of optical fibers, textiles yarn, metal or dielectric wires, resistors, new complex materials, etc. is performing for a long time by laser techniques. By the implementation of diffraction effects in automated processes, diameter, hairiness and other industrial parameters are controlled. Faults in products can be controlled in 1-3 D geometries. It is interesting that the algorithms linked to the search of airborne missiles in Space are similar to the algorithms of the searching of the faults in weaving by implementation of similar basic estimations.

**b) Material and system dispersion.** Laser techniques use determine the dispersive characteristics of the materials, including optical fibers links.

**c) Failure development** Failure growth in systems and components is also monitored by using optical nondestructive techniques in real time. The title optical NDT techniques widely replace other methods; but they use all modern nonlinear methods and couplings: electro-optics, photo-elasticity, magneto-optics, piezo-optics. Note that, on the other hand, laser processing of magnetic material performances is the most acceptable. LAMMA techniques in combination with electron microscope and time of flight spectrometry, gas chromatography, FTIR are unavoidable today in all technological standard investigations from ecology up to polymerizations, and metal bending, ozone hole problems, etc.

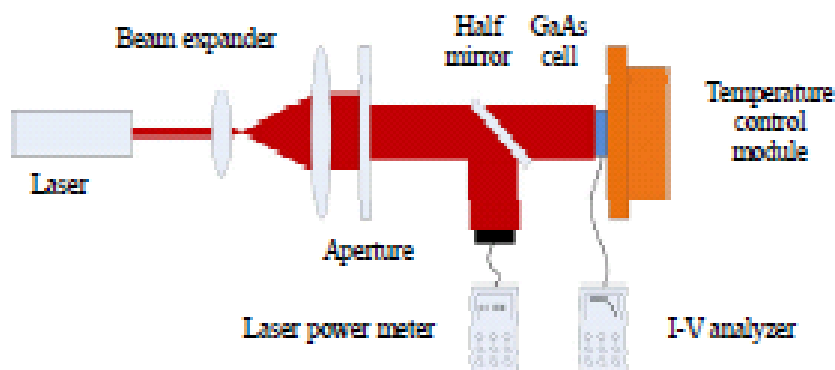


Figure 6a System setup for laser irradiations of components/systems/fiber links.

This is set up in general, and many details are in experiments different and more complex (thermal control, half mirror /beam splitter to monitor the input laser energy/power ,oscilloscope, laser type etc.)In Fig. 6a, b are presented geometry of the experimental equipment layout and output characteristics of GaAs cell irradiated by laser [14, 15, 24, 25 ].

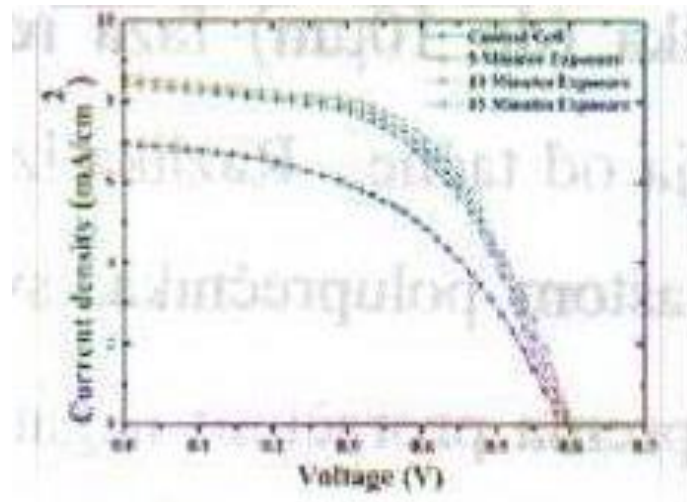


Fig. 6b Currents density versus voltage .

Some facts of laser action to solar cell and other components are following:

- Thermal effects and electrical properties of nano-crystalline and polycrystalline Si solar cells due to laser irradiation with 850nm were found.
- Photovoltaic receivers versus laser beam power in NTRS are changed after irradiation; dependance on laser wavelength in given power range is weak for the response of solar cells (at 850nm for GaAs cells and cell without radiation damage were not so remarkable distinction in response.
- Effects of laser patterning on properties of crystalline Si: Laser irradiation and ablation decreased the lifetime of photo-generated charge.
- Enhanced efficiency of the dye sensitized solar cells by is found for solar cell based on hydrothermally deposited laser irradiated carbon nanotubes.

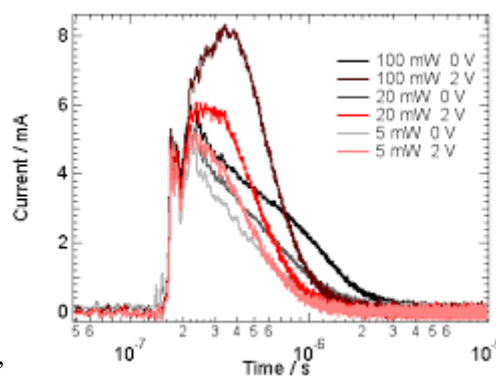


Figure 6c.

In Figs. 8 are presented some performances of GaAs cells after laser irradiations. Damages during material breakdown or during the occurrence of the effects of birefringence, drilling and cutting of glass/plastic optical elements (windows, lenses, active materials, klorite, composites with fillers, carbon components which laser beam can cut or drill).

The influence of ELION techniques and studying the influence of nuclear radiation and particles to optical fibers, devices and sensors within is always topic, but with time, more and more detailed quantitative descriptions are formed.

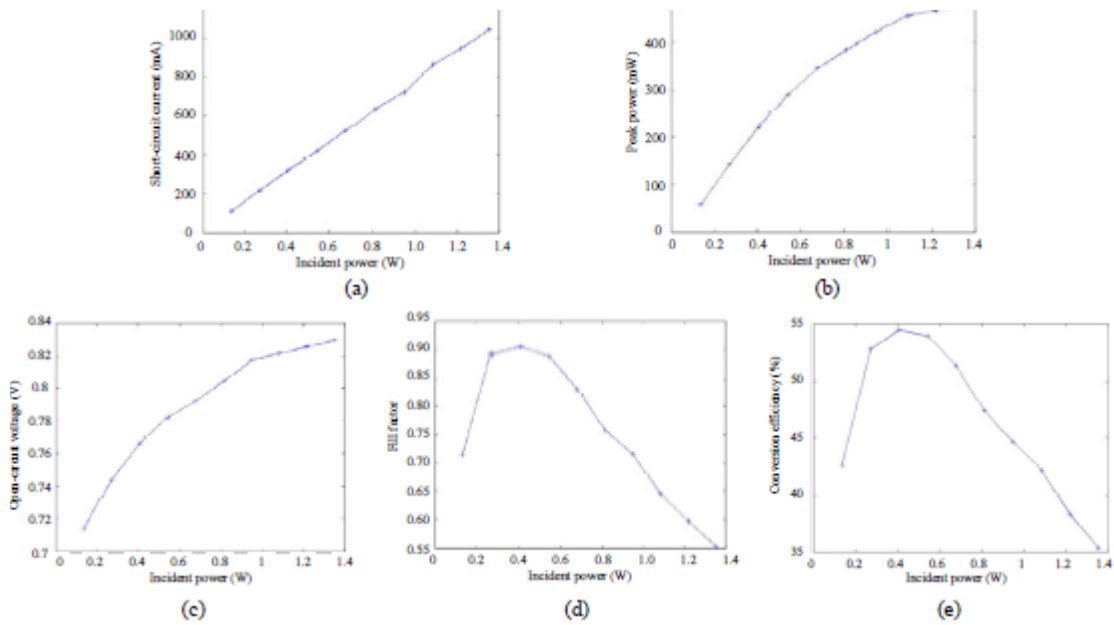


Figure 7 Performances of GaAs cells versus incident power [ ], :a) short –circuit current b) Open – circuit voltage, c) Peak power. d) factor and e) conversion factor efficiency.

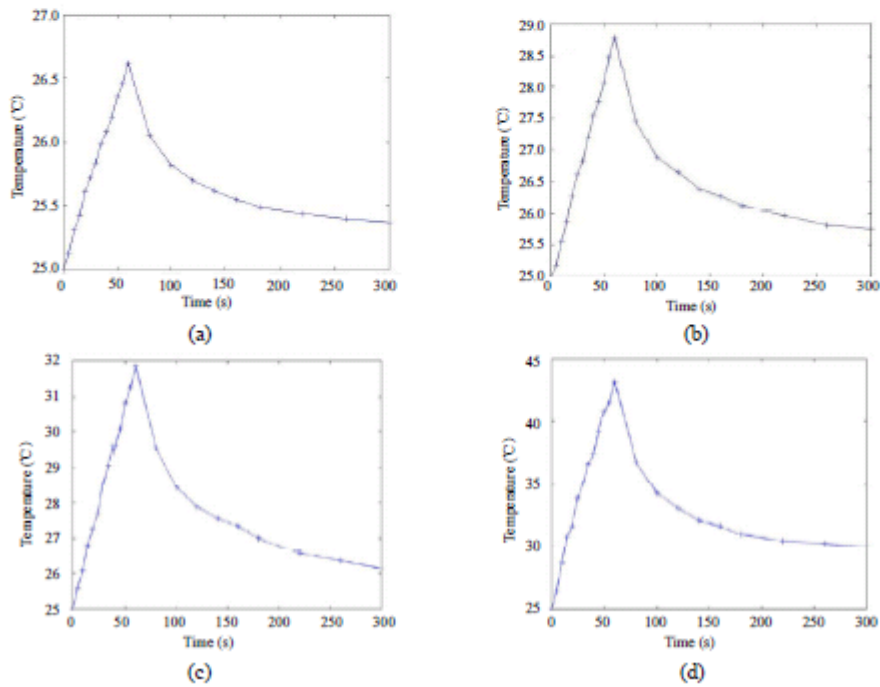


Figure.8 Temperature of GaAs cell versus time and incident power: 0.1435W (a), 0.405W (b) ,0.675 Wc) and 1.35W(d)[ ]

The number of descriptions of nuclear radiations together with high power laser facilities is increasing. In those places the radiation and particles appear as the result of the interactions with various targets. On-going repeated question of what is the most powerful laser is answered with the fact that Japan is the owner of the most powerful laser facility. Standard division of scientific disciplines (nuclear, atomic) and is visible with the concept of optical chains and nonlinear effects [], and metrology.

The other group of studies continues fundamental research and estimations by Monte Carlo and similar approaches (4,) and begins with descriptions of optical defects provoked by radiation, which is the research subject for a decade. PSD techniques, which divide nuclear radiations, here would have something to say because the fields in which the fibers might be found frequently contain neutron, gamma, beta , ..., beams [ 6, 45,46,]





of systems for trace reading. Some are commercially available and greater number is specific for laboratories. Fig.10 is one of the oldest but today there is many other experience of positive issue [2,3,48]

**Optical systems.** Optical systems, linked to the following the traces of nuclear particles, mostly is used for reading. These systems are numerous, from optical microscopes for manual reading to systems which contain images. Fig. 3. Image and camera control, video adapter, video signal adapter, camera control, video camera, focus, stopping motor and encoder, stage movement control, manual control board, pixel addresses, three manual control boards, storage movement control and step motor and encode, and encoder.

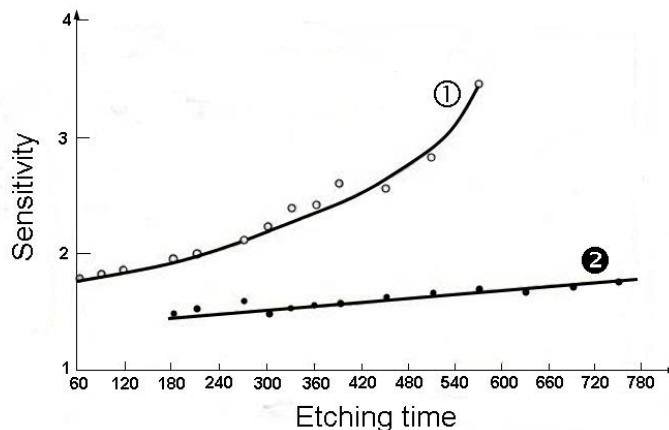


Figure 10.

**LIS, Rydberg, Atoms, Lidar,** Fibers for ecology and Space mineral and raw material processing, especially flotation of metals, principally are based on classical chemical enrichment technology. Laser technology has found application in mining and geology not only for mineral detection or characterization but enrichment, especially when chemical treating represent on dangerous process in regard to environment. Some specific methods are developed based on the nature of a laser beam and provoked processes. (LIS, Rydberg atoms, LIDAR or fiber in solving many problems, as well as to powering sensors in explosive environment can be in some kind of group of problems that are solving by laser techniques-). They are based on multi photon processes for ionization, isotope separation, actinides treating. The improving laser methods change the classical chemical methods in enrichments [23] and coefficients of efficiency.

**Efficiency and processess.** In Table 3 is presented the efficiency of some lasing systems of other choice of commercial lasers (Terrestrial) using data from various references and in Fig.11 diode pumped solid state lasers of higher powers..

Table 3 Efficiency of commercial lasers.

Type	Efficiency %	Comments/Applications
Ar <sup>+</sup> :ion	0.0001-0.01	Medicine, surgery, metrology, scattering
CO <sub>2</sub>	5-20	Material processing
Cu vapour	0.2-0.8	Surgery, spectroscopy
Excimer ( noble gas VII groupe)	1.5-2	
GaAlAs semiconductor	1-10	Microelectronics, Mass media
HeCd (hole cathode)	0.002-0.014	White light
He-Ne	0.01-0.1	Metrology, interferometry, medicine
Nd <sup>3+</sup> :Yag, Nd <sup>3+</sup> :glass	0.1-1	
Al <sub>2</sub> O <sub>3</sub> :Cr <sup>3+</sup>	0.1-1	

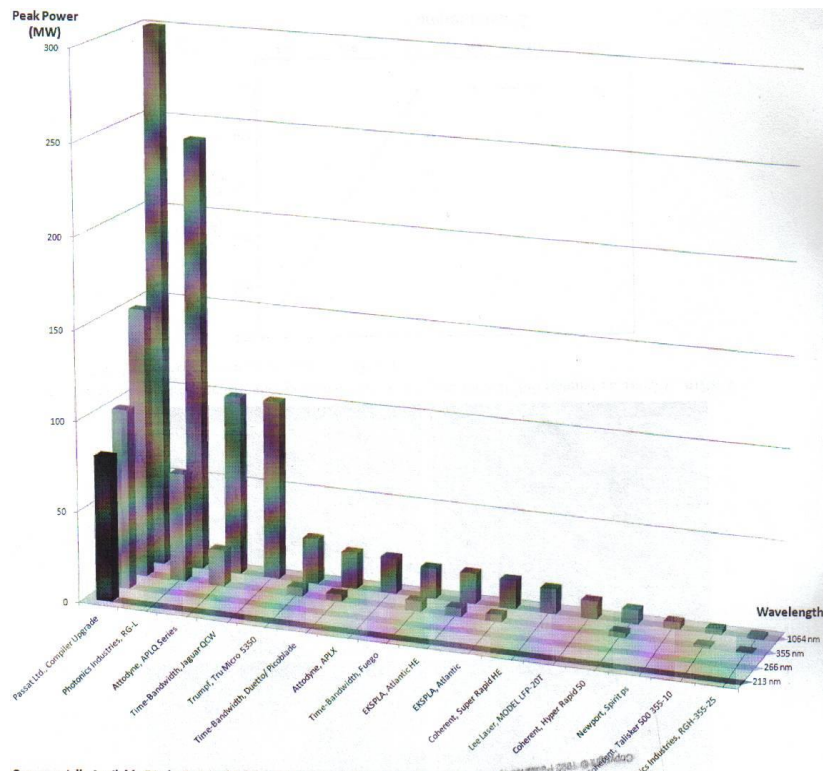
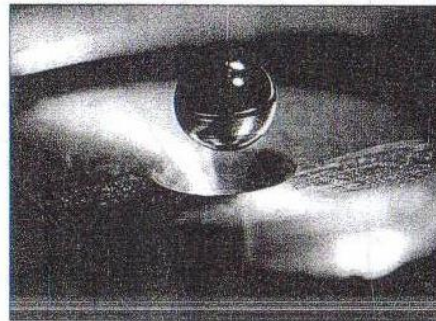


Fig.11. Commercially Diode Pumped Solid State ps- Lasers with peak powered ( $>1\text{MW}$ )[].

Under the title „the most powerful laser“, different data appear. According to some data, it is the laser in Lawrence Livermore National Laboratory in California, where parameters were 2 MJ per pulse. This laser was designed for 1.875MJ (2.03MJ-with different lens). Obtained blast damaged optics less than was predicted, Figs.12. Second pulse was obtained after 36 h of recovering (4-step amplifications were used). Its energy was 1000 times higher than energy/power system in USA. It was a part of investigation for fusion ignition.



a)



b)

Figure 12

**Tactical lasers** Next important operation was obtaining a powerful beam of X rays, and the expression of Ed Moses was that this was a significant demonstration from the point of view of energy, precision and availability. The experiments are considered as the endeavour for obtaining the clean energy and decreasing the dependence on coal, oil and fission power plants. The nowadays concepts of the development of sources of renewable energy up to 2050 is presented in Fig.13.

**Point of view tactical lasers.** After prediction of Fig.13, follow return to tactical lasers that are in the areas from new conceptions as *laser reactor*, nuclear pumped lasers, the most powerful lasers and laser processes applied in military conceptions. The shortest pulses dynamics of development up to near 2010y could be followed in Fig.14.

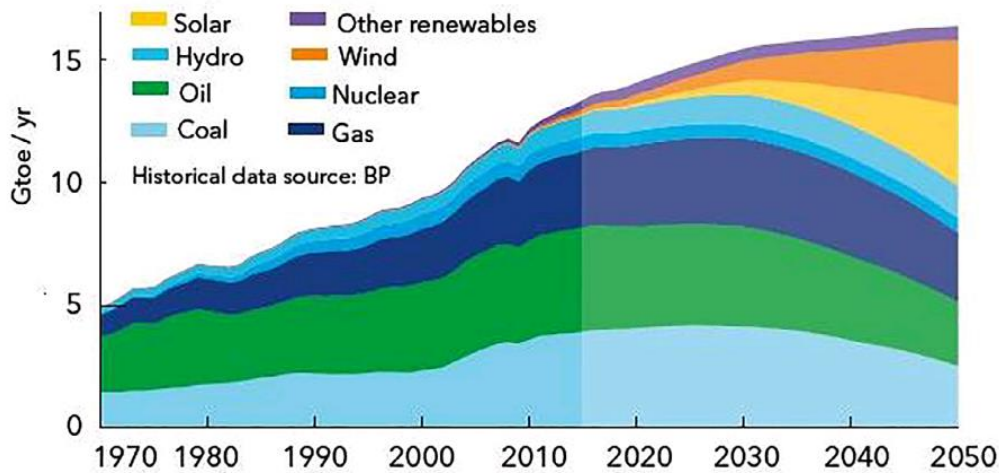


Fig.13 Future of sustainable energy transmission.

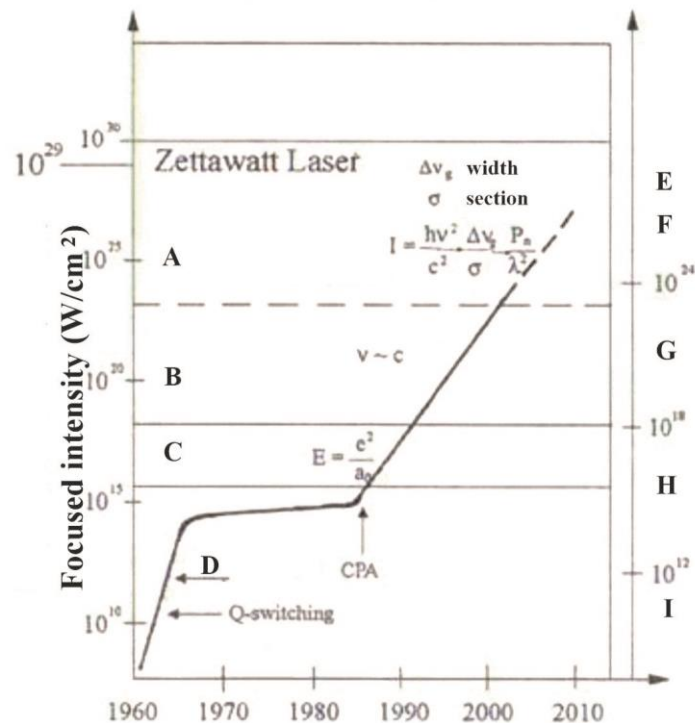


Figure 14. Development of focused laser intensity: A- limits of laser intensity,calculated to  $\text{cm}^2$  of active materials; B-Relativistic optics, C-Bonded electrons, D-mode synchronization, E-Era of soft electrons, F-quark era, G-Positron- electron era, D-Plasma era, I- Atomic era.

Consideration of qualitative and quantitative parameters is related to the laser weapons, too. It is not a singular area of human activity but concerning that the research is related to high energies (energy density) the researches had to be in connection to high energy plants, of the lower kind followed by couplings according to technical possibilities. In the early years of second half of the 20<sup>th</sup> century (1965), the development of laser weapons emerged. Successful realization of laser cannon (1972) and a successful testing have shown the possibility of destruction of airborne targets. Fever induced by racket systems and defense in the world resulted in one of the most replicated scenario with photos, with laser in systems of coarse and fine tracking, computer solutions of optical computers and firing defense pulses for the protection of sky. From then more than 60 years have passed. It would take long to reconsider and question what happened to the predictions Platform A-60 followed and the first flights (1981) and in the beginning just the race between the east and the west and then throughout the world. ‘‘Star Wars’’ (1983). USA introduced the new project and the

number of space related movies increase in the world. That meant the accelerated unprecedented arms races of the superpowers. In 1984 the A-60 was first to use laser for destruction of the airborne targets. The triple dagger laser weapon follows with high power pulses for attack photoconductive guidance systems. CsF chemical laser comes to the fore. It is still in some answers to the question about the most powerful laser in the world.

The era of the development of laser weapons in the world is connected to the year 1970 and the years when certain issues were resolved concerning the enriching of uranium isotope, etc. This enabled the issue switch of fission and fusion. The stand stating that this is only of interest for the future faded. The future was approaching. In tab 5. commercial approach to lasers in industry, illustrated in Fig. x and in Tab. 2 were given as differences example efficiencies of laser processes depending on the pump used in semiconductor lasers.

Laser efficiency is defined in the number of ways and systematically it could be considered for the efficiency of the pump, amplifications, lamp,..[ ], microscopically or coarse macroscopically, etc. It varies from below 1% and theoretically trend towards 100%. When tens of % were achieved it was considered as a success. The books of science fiction, Hyperboloid engineer Garin the predictions of reality appeared and sometimes the order is as novel of movie and sometimes the reverse. But it is becoming reality.

In 1965 on the screens of the country was a fantasy movie "Hyperboloid engineer Gavin" based on the novel by Alexei Tolstoy. Not to follow the history era of the quantum electronics development, but to commence with experiments and first masers around 1959, with Ruby laser 1961 and with Theodor Mayman. Awards followed and interaction of Russian and American scientist on begetting them. The special design bureau Vympel was involved in the development, which by the end of the 1960s was separated into an independent organization of the "alser provile" – TsKB "Lunc" (later – NPO "Astrophysics"). After previous went the "Terra" and "Omega" for the destruction of missiles, aimed at aircraft, Sary-Shgan (Kazakhstan) tests on polygon Tetra – works for NGO 'Alamy') with regard to air defence.

To hold here for a moment, but as the area is still in development, a conclusion is inevitable that energetically the higher controlled energy densities on relatively lower frequencies are achieved compared to the possibilities of the lasing transitions. The years 2016 and 2018 passed and at the middle of the 2019 to remind that the data will be late. Most of the energy beams was eaten by evaporation of moisture from the sea surface, because of which the efficiency was only 5 percent for some processes is just an illustration.

Historical events of various breakages in the world will be left aside, but the ribbon remains.

For application in Space one pulse melts the part of the components of the systems and the system is preheated.

Supposed stock in Russian army until 1995. Systems as the "Soviet 1K17 mobile laser complex" could 'blind' the enemy optics and personnel, but not much more. Even quarrels began that armed lasers are a real psychological area given that it threatens the enemy in scouting units, aircraft [operators#, artillery gunners, snipers and other personnel who use optical systems).

Discussion about coefficient of laser/laser system efficiency, has many approaches and is expressed on various manner. Laser efficiency as defined, can be studied as partial, as in efficiency of some subsystems: pump, amplification, excitation bulb, microscopic, or macroscopic, output / input, etc. [ ]. It can be smaller than percent, and theoretically to tends  $\approx 100\%$ . When lasing was obtained with 30%, it is considered as very successful. As for lasing pump and active material are the most important, for chosen theoretical approach, in Table 4 some data is presented that illustrate the problems/issues in semiconductor lasers; Table 5 present data for some most known laser types based on various active materials.

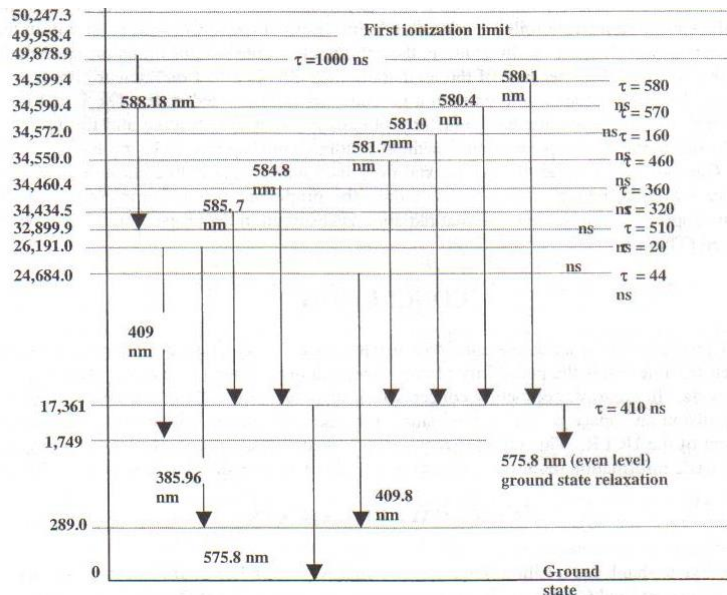


Fig.15 Energy level diagram of U atoms. (Odd parity transitions  $J=5$  and  $7$ ).

Tab. 4 Energy conversion- Efficiency of semiconductor laser depending on pump

Pump	Max-im.output	Pulse width	Max.density pump power	Max output power	Active layer thickness	Spec. demands/Comments
Elektron.excitation	$\frac{\hbar\omega}{3E_g} \approx 0.3$	$10^{-8}-10^{-6}$	$10^1-10^8$	$10^0$	$10^{-4}-10^{-3}$	Sizes of semiconductor laser depends on the pumping type
Optical excitation 1.photon excitation	$\hbar\omega < \hbar\omega_p$	Cw generation $10^{-8}-10^{-7}$	$10^1$	$10^3$		$E_g' < \hbar\omega_p$
Optical excitation:two-photon excitation	$\hbar\omega < 2\hbar\omega_p$	$10^{-8}-10^{-7}$	$10^2$		$10^{-3}-10^{-3}$	$E_g' < 2\hbar\omega_p$
Injection	$\frac{\hbar\omega}{eU} \approx 1$	Cw generation $10^{-8}-10^{-7}$	$10^4-10^3$	$10, 10^2$	$10^{-3}-10^{-3}, 10^{-2}$	1) Elektroconductivity 2) Inject. contact 3. Electrical contact
breakdown	$\frac{\hbar\omega}{3E_g} \approx 0.3$	$10^{-8}-10^{-7}$	$10^0$	$10^4$	$10^{-4}-10^{-3}$	1). Electrical contact 2) problems with risetimes 3) nonstationary conditions

In Table 6 are presented cyclotron based masers and some previous facilities.

Apparent success was achieved in equipping laser weapons with self-propelled tracked vehicles. "laser tank" could detect and attack the target, which has optoelectronic devices. The laser strike was supposed to disable the enemy's guidance systems, making it unfit for use, and to blind the gunner, damaging the retina of the eye.

New state armament programs are in course. Laser systems in 2018 are highly developed from 1965, but still problems are heat excess, beam dispersion, dust, fume, (scattering centres are formed). (Only first pulse can provoke melting of components of the systems and system is preheated)

Some systems have mobile reconnaissance units using a laser locator to detect low-level atmospheric chemical contamination and non-lethal systems. It means temporarily blinding of enemies.

For the future, problems of the theoretical point of view should analyze photonic framework in Handle Physical and Chemical Processes, Quantum Entanglement, Coherence, De-coherence, RE-coherence and the roles of Multipartite Base Stones. [32, 41]

Tab.5 Chosen laser types and performances

Features	CO <sub>2</sub> laser	Nd <sup>3+</sup> :YAG laser	Nd <sup>3+</sup> :YAG disc laser	Fiber laser
Active medium	CO <sub>2</sub> :N <sub>2</sub> :He	Crystal rod	Crystal disc	Doped Silica Fiber
Pump	Gas discharge	Lamp, Diode laser	Diode laser	Diode laser
Wavelength, nm	10 600	1060	1030	1070
Max cw regime P, kW	15	6	16	50
Spot dia, mm	0.15	0.3	0.15	0.15
Power density MWcm <sup>-2</sup>	84.9	8.5	90.5	113.2
Efficiency %	10	2 (lamp) 6 (diode)	18	30*
Delivery	Free space	Fiber	Fiber	Fiber

Table 6 Quantum generators

Laboratorijs	Frequency, GHz	Power, kW	Coefficient of efficiency	Pulse width
Varnan	28	340	45	Cw regime
Varnan	60	120	38	Cw regime
Lab.VMS USA	35	340	54	1 μs
MTI	140	180	30	1 μs
Hudges	60	240	30	100ms

Prognosis to the 2050y. presenting the fractions/types of energies/power in the world and state in our state [28,29 ] could be discussed in regard to laser processes of energy transformation. Considering chemical lasers 2 possibilities are for investigation :theoretical calculations/simulations for needed transition probability, searching of efficiency/cross-section [1,30, 36,- 40.] and the work in large existing laboratory/facilities where strong experimental support, i.e. tactical lasers under consideration could investigated.

Laser application for initiation of atmospheric falls. lightning , protection measures developed with microwaves ranges contra natural falls, belong to these activities, but in all activities specific sophisticated technical supports [ 42] are needed. Either there are a lot of activities with few real data, for a long time will be seen the thunders on Evia island; when for a 24 hours 5000 thunder attacks were recorded, damaged/destroyed solar panels and gasoline stations.

On the other hand all conceptions with stimulated radiations, conceptions via fusion, fission and involved **aser** processes including parametric, and exotic pumping, have to be paid attentions. Solar laser excitations, seem that will have further effects including in Space investigations. But, concentrator design should be further investigate as well as theoretical evaluation, calculations of probabilities rate [19,20 ].

**Nuclear pumped lasers** deserve attention from the experiments with setting He Ne tube in neutron field up to exotic excitation /pumping with gamma radiation, alpha and beta particles. In particular are succesfully processes where preionisation is applied (with gamma and X radiations) and the laser threshold decreases

**Gammaraser.** Especially place is admitted to gammarasers, found under various names, besides incorrect *gamma lasers*, gasers. Not that graser exist also, but the discussion with gravitation

waves will be let from. Models are based on: Mössbauer's lasers, *nonmössbauer's* lasers, laser without threshold, electrodynamic lasers in Röntgen and gamma ranges. Special attention should be paid to pumps and there is a difference if short living or long term living nuclear isotopes are.

### 3.2 Discussion and Conclusion

Problem of electromagnetic pulses of nuclear explosions to electrical and power systems from electro plants was actual at the end of 20 century. By initiation of nuclear explosion beside of shock, heat and radioactive action electromagnetic pulse that was responsible for electronic and power electrical facilities and equipment and systems. Discovering of quantum generators in the middle of 20 century, has bring up the new conjunctions and more energetic processes. Now was question in the field of human application of coherent energy. The question is also will be systems installed into medical institution or in great laboratories will be installed medical equipment. The questions about laser reactor and its conception of nuclear excitation of quantum generators, and also sensitivity of nuclear sensors, have introduced a lot of new investigations, but it seems that there are many unanswered questions, either the experiments have rich tradition and history, and shown positive conjunctions. The energetic analysis is monitored for the future and for planning of new types of energy transformations. EMINA and simulators are presented many evaluation with parallels of strikes, as natural processes. There are investigations in atmospheric discharging by using a laser, but this time the archeological finds are damaged. These analysis of processes and trends could be expressed that the pulse of the most intensive laser systems are comparable with our Đerdap, but also with whole powering in USA. Don't forget that is only in the regions of femtosecond. Considering discussion of laser possible for nuclear fusion, discussion start with possibility of Nd<sup>3+</sup>: YAG /glass, CO<sub>2</sub>, etc, but the smaller quant energy the higher number of beams should be unified in irradiations of target of very small size; in this case, phase conjugated optics is unavoidable. Reactor laser conceptions are more in simulations than in *productions*, but the future will give the answers.

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## SCIENTIFIC RATIONALE

Spectroscopy is a powerful tool for the analysis of radiation from different plasmas in astronomy, laboratory, fusion research, atmospheric research and industry. Effective theoretical analysis, synthesis and modelling of stellar spectra as well as the spectra from other plasma sources, depends on atomic data and their sources. In particular, for the modelling of stellar atmospheres and opacity calculations a large amount of atomic data is needed, since we do not know *a priori* the chemical composition of a stellar atmosphere. Consequently, the development of databases with atomic data and astroinformatics is important for stellar spectroscopy.

The Conference is planned as an opportunity to consider above mentioned aspects of spectroscopic research on plenary sessions and then to work on the special mini-projects, which will result in common papers to be published in international astronomical journals after the Conference.

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## CONTENTS

Zlatko Majlinger, Milan S. Dimitrijević and Vladimir A. Srećković: <i>Curve fitting method of Stark width determination – example of H I line in G191-B2B spectrum</i>	9
Slavica Malinović-Milićević: <i>Clear-sky spectral UV radiation modeling</i> (Invited lecture)	10
Željka D. Nikitović and Zoran M. Raspopović: <i>Rate coefficients of He<sup>+</sup> ions in CF<sub>4</sub> gas</i>	16
Milan S. Dimitrijević, Magdalena D. Christova, Nenad Milovanović and Sylvie Sahal-Bréchet: <i>Stark broadening of Zn II spectral lines in stellar atmospheres</i> (Invited lecture)	17
Maja S. Rabasovic, Bratislav P. Marinkovic and Dragutin Sevic: <i>Analysis of laser induced plasma plume in atmosphere: artificial neural network approach</i>	19
Maja Kuzmanoski, Luka Ilić and Slobodan Ničković: <i>Spatial variability of mineral dust single scattering albedo based on DREAM model</i> (Invited lecture)	21
Aleksandra Kolarski and Vladimir Srećković: <i>Lower Ionosphere perturbations due to Solar X-ray flares simultaneously monitored on two VLF signals with close GCPs</i>	23
Aleksandra Kolarski: <i>Monitoring effects of low intensity X-ray Solar flares from 23/24 Solar cycle minimum on VLF signals recorded in Belgrade</i> (Progress report)	24
Aleksandra Kolarski: <i>Numerical simulations of subionospheric VLF propagation under influence of moderate Solar X-ray flare events</i> (Invited lecture)	25
Aleksandra Nina, Vladimir Čadež, Luka Č. Popović and Milan Radovanović: <i>Periodic variations of ionospheric Wait's parameters caused by changes in the intensity of incoming solar hydrogen Ly radiation</i>	32
Oleg R. Odalović: <i>GNSS signals as a tool for detection of the influence of solar radiation of terrestrial ionosphere</i> (Invited lecture)	37
Dušan S. Petković, Oleg R. Odalović and Aleksandra Nina: <i>Influence of the periodic changes in the incoming solar hydrogen Ly-<math>\alpha</math> radiation intensity on the total electron content in the ionospheric D-region</i>	39

Z. Mijatović, S. Djurović, T. Gajo and I. Savić: <i>Approximation of the shape of hydrogen <math>H_b</math> spectral line with Voigt profiles</i>	44
Mihailo Savić, Nikola Veselinović, Aleksandar Dragić, Dimitrije Maletić, Dejan Joković, Vladimir Udovičić, Radomir Banjanac and David Knežević: <i>The study of atmospheric effects on cosmic ray muons in the Low Background Laboratory for Nuclear Physics at the Institute of Physics Belgrade</i> (Invited lecture)	48
Bratislav P. Marinković, Stefan Ivanović and Zoran Mijić: <i>Data analysis on Serbian participation in COST Actions: Celebrating 50 years of research networks</i> (Progress report)	49
Vladimir Srećković, Ljubinko Ignjatović, Milan Dimitrijević, Nikolai Bezuglov and Andrey Klyucharev: <i>Rate coefficients and cross-sections for some collisional processes involving Rydberg atoms</i>	58
Vladimir Srećković, Veljko Vujčić, Aleksandra Kolarski, Jelena Barović and Ognyan Kounchev: <i>Low ionosphere modeling: new data and models</i>	59
Veljko Vujčić and Vladimir A. Srećković: <i>A&amp;M Data: Processing and Modeling in Real Time</i>	60
Vladimir Srećković, Jelena Barović and Gordana Jovanović: <i>Ionosphere modeling and intense radiation</i>	62
Vladimir A. Srećković and Sanja Tošić: <i>Collisional and radiative processes involving some small molecules: A&amp;M data</i>	63
Lamia Abu El Maati, Mahmoud Ahmad, I. S. Mahmoud, Sahar G. Tawfik, Najah Alwadii, Nabil Ben Nessib and Milan S. Dimitrijević: <i>Atomic structure and transition parameters of the V XVIII carbon-like ion</i> (Invited lecture)	64
Violeta M. Petrović, Sanja D. Tošić, Hristina Delibašić Marković and Ivan D. Petrović: <i>Investigation of Laser Induced Breakdown Threshold</i>	66
Ljiljana M. Brajović and Miodrag Malović: <i>Gravity satellite missions measurement data for atmospheric density estimation</i> (Invited lecture)	68
Jelena B. Maljković, Jelena Vukalović, Zoran Pešić, F. Blanco, G. García and Bratislav P. Marinković: <i>Absolute differential cross section for elastic electron scattering from halothane molecule at 150eV</i> (Invited lecture)	70
Zoran R. Mijić and Bratislav P. Marinković: <i>Statistics of Management Committee Members from Serbia in COST Actions</i> (Progress report)	74



Saša Dujko, Danko Bošnjaković, Ilija Simonović and Zoran Lj. Petrović: <i>Collisional and transport processes of low-energy positrons in molecular hydrogen</i> (Invited lecture)	81
N. M. Sakan, I. Traparić, V. A. Srečković and M. Ivković: <i>The usage of perception, feed and deep feed forward artificial neural networks on the spectroscopy data</i>	83
Nikola Cvetanović and Bratislav M. Obradović: <i>Stark broadening method of Hydrogen Balmer beta for low-density atmospheric pressure discharges</i>	85
Darko Jevremovic: <i>History of Serbian involvement in LSST</i> (Invited lecture)	87
Zoran Simić and Nenad Sakan: <i>On the Stark broadening parameters of Ir II spectral lines</i>	88
Zoran Mijić, Maja Kuzmanoski, Luka Ilić, Aleksander Kovačević and Darko Vasiljević: <i>Review of atmospheric aerosol optical properties profiling and lidar station activities in Serbia</i> (Invited lecture)	89
Saša Topić and Zoran D. Grujić: <i>Laser spectroscopy and magnetic resonance atomic magnetometry in search for dark mater: New bounds on Axion like dark matter from GNOME network of OPM's</i> (Invited lecture)	97
SECTIONS (MINI PROJECTS)	99
AUTHORS' INDEX	101

## **Review of atmospheric aerosol optical properties profiling and lidar station activities in Serbia**

**Zoran Mijić<sup>1</sup>, Maja Kuzmanoski<sup>1</sup>, Luka Ilić<sup>1,2</sup>, Aleksander Kovačević<sup>1</sup> and  
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### **Abstract**

An advanced laser remote sensing technique – LIDAR (Light Detection And Ranging) is the most appropriate tool for providing range-resolved atmospheric aerosol vertical distribution. LIDAR measurements of aerosol optical properties with high spatial and temporal resolution give detailed information on the occurrence and development of aerosol structures. In this study a brief introduction of a lidar system developed at the Institute of Physics Belgrade in the past and the new system currently operating, is presented together with several activities conducted within European lidar network. The capacity and the experience from measurement campaigns aiming to provide near real time data products and study the changes in the atmosphere is also discussed.

### **Introduction**

Clouds and atmospheric aerosols play an important role in the Earth's radiation budget, thus quantifying the role of aerosols in climate system is crucial for better weather forecasting and understanding climate change. The amount of scattered and absorbed radiation (both incoming solar and outgoing terrestrial) varies according to aerosol composition, size and shape distributions. The short lifetime and large variability in space and time further contribute to the identification of aerosol radiative forcing as one of the significant unknowns in our understanding of climate change (Stocker et al., 2013). The complexity of the aerosol interaction with the climate system makes it necessary to estimate its impact through the integrated use of ground-level and airborne *in-situ* measurements, ground-based remote sensing, and space-borne observations in combination with advanced numerical modelling. LIDAR (Light Detection And Ranging), an active remote sensing technique, has proved itself to be the optimal tool for profiling height-

resolved atmospheric aerosol optical parameters. Various aerosol lidar techniques have been developed during the last several decades like backscatter lidar, Raman lidar, depolarization lidar, and high spectral resolution lidar. Each type of lidar can operate at one or multiple wavelengths. The LIDAR principle is based on laser emission of short-duration light pulses into the receiver field of view. The intensity of the light backscattered by atmospheric molecules and particles is measured versus time (through the telescope receiver, collimating optics, a bandpass filter for daylight suppression) by an appropriate detector. The signal profile is recorded by an analog-to-digital converter or by a photon-counting device and accumulated for a selected integration period, which may range from a few to thousands of individual laser shots – spanning time intervals from seconds to minutes. In order to establish a comprehensive and quantitative statistical data base of the horizontal and vertical distribution of aerosols at European scale the lidar network called EARLINET (the European Aerosol Research LIdar Network) was founded in 2000 (Pappalardo et al., 2014). The development of the quality assurance strategy, the optimization of instruments and data processing, and the dissemination of data have contributed to significant improvement of the network towards a more sustainable observing system. Currently, EARLINET contributes to the Aerosol, Clouds and Trace gases Research Infrastructure (ACTRIS), the pan-European research infrastructure producing high-quality data and information on short-lived atmospheric constituents. In this paper a brief review of atmospheric aerosol remote sensing capacity over the past period at the Institute of Physics Belgrade (IPB), Serbia, is presented together with short introduction on the methodology of elastic backscatter and Raman lidar systems. In addition, experience from several activities of IPB lidar station from dedicated measurement campaigns is described.

## Methodology

The lidar equation for return signal  $P(\lambda)$  elastically backscattered by air molecules and aerosol particles is found to be

$$P(\lambda, r) = P_0(\lambda) C \frac{O(r)}{r^2} \beta(\lambda, r) \exp \left[ -2 \int_0^r \alpha(r') dr' \right] \quad (1)$$

where  $P_0(\lambda)$  is the laser pulse power;  $C$  is a system constant (taking into account the optics and electronics used);  $O(r)$  denotes the unitless correction function that corrects the lack of coincidence of the laser beam and the receiver field of view for ranges below the complete overlap height;  $r$  is the distance between the laser exit and the point of scattering in the atmosphere;  $\alpha(\lambda, r)$  and  $\beta(\lambda, r)$  denote the height (distance) and wavelength ( $\lambda$ ) dependent extinction and backscatter coefficients respectively. The extinction coefficient describes the ability of particles to scatter or absorb light at a given wavelength while the backscatter coefficient (scattering coefficient at  $180^\circ$ , normalized to the unit solid angle) refers only to scattering events. Backscattering and extinction are both caused by particles and molecules.

While the molecular scattering properties can be determined with sufficient accuracy from the available measurements of temperature and pressure profiles, the aerosol backscatter  $\beta_a(\lambda, r)$  and extinction  $\alpha_a(\lambda, r)$  coefficients remain to be retrieved. In lidar profiling, the most significant errors occur during signal inversion, when the optical parameters of the atmospheric aerosols are extracted from the lidar signals using a number of implicit premises and *a priori* assumptions. Two *a priori* assumptions are necessary to allow the retrieval of  $\alpha_a(\lambda, r)$  and  $\beta_a(\lambda, r)$  profiles from the elastic lidar measurement: an assumed value of the lidar ratio (aerosol extinction/backscatter value) and the reference range chosen such that the particle backscatter coefficient is negligible compared to the known molecular backscatter coefficient value. The main drawback of this method is the fact that the extinction profile is estimated from the determined backscatter coefficient profile.

The first elastic backscatter lidar system at the IPB was developed in 2008 as bi-axial lidar system with transmitter unit based on a water-cooled, pulsed Nd:YAG laser, emitting pulses of 100 mJ and 50 mJ output energy at 1064 and 532 nm respectively, with a 20 Hz repetition rate (Fig. 1). The optical receiver was the Schmidt–Cassegrain telescope with a primary mirror of 304.8 mm diameter. Si PIN photodiode FD5N was used to detect elastic backscatter lidar signal at 532 nm. An interference filter with 3 nm bandwidth was used to select the lidar wavelengths and to reject the atmospheric background radiation during daytime operation. For analog detection, the signal was amplified according to the input range selected and digitized by an A/D converter NI5124.



Fig. 1. Elastic backscatter lidar system developed at IPB.

The described system was the first lidar system of that kind used for aerosol profiling in Serbia. To overcome the limitation of elastic backscatter lidar technique the so-called Raman lidar technique can be used and the profile of particle extinction coefficient can directly be determined. Raman lidar measures lidar return signals elastically backscattered by air molecules and particles and inelastically (Raman) backscattered usually by nitrogen molecules. Whereas the

elastic backscatter lidar is operational both at day and night, the Raman lidar is mainly used during nighttime, due to the strong daylight sky background. The determination of the particle extinction coefficient from molecular backscatter signals is rather straightforward since neither lidar ratio nor other critical assumptions are needed (Kovalev, 2015).



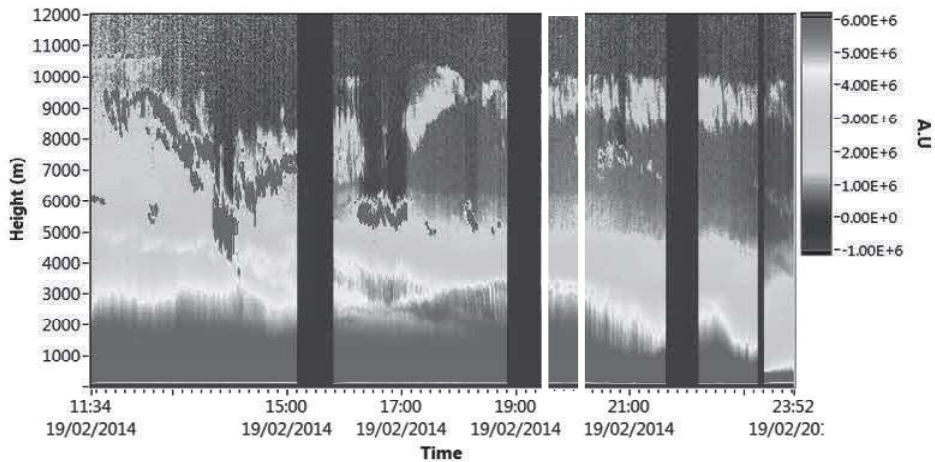
Fig. 2. IPB Raman EARLINET joining lidar station.

In 2014 Raman lidar operating at 355 nm and 387 nm ( $N_2$ ) was set up at IPB, establishing Serbian EARLINET joining lidar station (Fig. 2). The basic characteristics of IPB Raman lidar are summarized in Table 1.

Table 1. IPB Raman lidar system components

Emitter		Receiver	Detection Unit
Pulse laser source:	Nd:YAG (Quantel CFR200)	Telescope type: Cassegrain, model Raymetrics DK250	LICEL TR20-160 (12 bit at 20 MS/s), 250 MHz fast photon
Wavelength	1064, 532, 355 nm	Telescope aperture: diameter: 250 mm	Detectors: Photomultiplier Tubes (Licel-Hamamatsu-R9880U-110)
Energy/pulse	105/45/65 mJ	Field of view: 0.5- 3 mrad (variable)	Detection mode: Analog and photon counting
Pulse duration and repetition	5 ns 20 Hz	Fieldstop type: Circular - Iris Diaphragm, 3mm user selectable	Spatial resolution (raw): 7.5 m
Laser beam diameter:	15 mm (expanded)	Elastic wavelength 355 nm	Full overlap: 250 m
Laser beam divergence:	0.33 mrad	Raman wavelength: 387 nm ( $N_2$ )	Effective range: 0.05 – 16 km

As a joining lidar station, systematic aerosol profiling has started in 2014 mostly for providing data for potential climatological studies as well as conducting dedicated measurements during Saharan dust intrusions or assessment of planetary boundary layer evolution (PBL) (Ilić et al., 2018). An example of such measurement performed on February 19, 2014 is presented in Fig 3. together with aerosol backscatter coefficient profile retrieved and dust load simulated by Dust Regional Atmospheric Model (DREAM) (Ničković et. al, 2011).



Emission wavelength: 355 nm Detection wavelength: 355 nm  
 Detection mode: an+pc, Temporal resolution: 60 s, Spatial resolution:

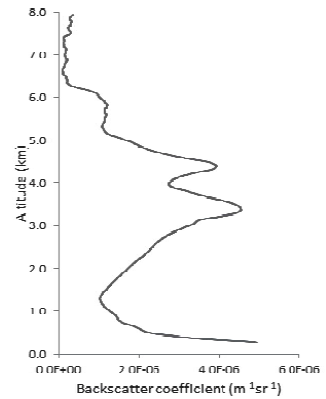
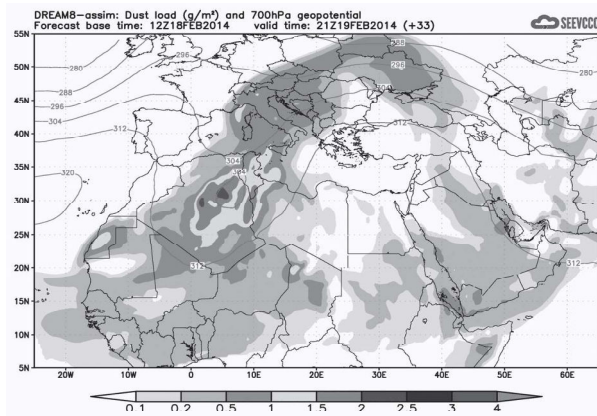


Fig. 3. LIDAR range corrected signal (above) and dust load over South Europe estimated by the DREAM model (below, left) on February 19, 2014; backscatter coefficient profile (below, right) retrieved for the selected time period (white rectangle).

## **Activities of the IPB lidar station**

In addition to regular aerosol profiling mostly performed at the very beginning after official joining the lidar network, several studies were conducted related to the application of gradient method for the identification of aerosol layer, as well as the evolution of PBL height (Ilić et al., 2018). The IPB lidar station also actively participated in several measurement campaigns organized through the EARLINET network.

## **The EUNADICS-AV experiment for NRT alerts**

Following the existing needs, the methodology providing a tailored aerosol products for aviation hazards based on high-resolution lidar data was developed with the aim to provide the EARLINET early warning system (EWS) for the fast alerting of airborne hazards (Papagiannopoulos et al., 2020). The application of the EWS and the timely delivery of the EARLINET data were tested in real time during the EUNADICS-AV exercise in March 2019. Each station submitted raw lidar data to the Single Calculus Chain (SCC) server every hour, which were automatically available on the EARLINET Quicklook Interface (<https://quicklooks.earlinet.org/>, last access: May 2022). The SCC is a tool created inside the EARLINET network for the automatic analysis of aerosol lidar observations (D'Amico et al., 2015). The primary goal of SCC is to offer a data processing chain that allows all EARLINET stations to retrieve aerosol products like backscatter and extinction profiles (measures of aerosol load) completely automatically. The raw lidar data were processed in less than 30 min after the measurement, enabling the timely delivery of the lidar data and the tailored product. When the raw data was submitted to the SCC server, it was instantly processed and made publicly available through the EARLINET portal in order to launch the alert distribution. The exercise revealed the network's strength, which, if activated immediately, can permit measurements in the event of natural threats for aviation.

## **COVID-19 Campaign**

A dedicated EARLINET measurement campaign was organized as part of the ACTRIS initiative to study the changes in the atmosphere during the COVID-19 lockdown period in May 2020, in order to monitor the atmosphere's structure during the lockdown and early relaxation period in Europe, and to identify possible changes due to decreased emissions by comparison to the aerosol climatology in Europe. The EARLINET near real time functioning was proven throughout the campaign, based on earlier experience from the EUNADICS-AV exercise. The IPB lidar station, along with 21 EARLINET stations, participated in the campaign by providing vertical aerosol profiles twice per day (minimum two hours

measurements at noon, and minimum two hours after sunset). The measurements were submitted and analyzed in near-real time by SCC. The first analysis was based on the data processed by the SCC and directly published on the THREDDS server in NRT. The preliminary analysis made on aerosol lidar data shows that simple comparison of the observed backscatter values with the climatological values from 2000-2015 is not sufficient to extract a clear conclusion on how much the COVID-19 lock-down has impacted the aerosols in the atmosphere, but a certain effect in the lower troposphere can be seen.

## Conclusions

Lidar systems are optimal tools for providing range-resolved aerosol optical parameters and information on the atmospheric structure. The IPB lidar station is the only lidar station for aerosol profiling in Western Balkans matching EARLINET quality control and quality assurance requirements. A brief description of the station capacity and activities in a few measurement campaigns are presented. Beyond the scientific goals of these campaigns, the actions organized by EARLINET/ACTRIS (NRT delivery of the data and fast analysis of the data products) proved that aerosol lidars are useful for providing information not only for climatological purposes, but also in emergency situations. Although the IPB lidar station is able to provide valuable data, automatization of the measurement process and the upgrade to the multiwavelength lidar system are required.

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# 11<sup>th</sup> International Conference on Renewable Electrical Power Sources



# PROCEEDINGS

Editor Dr Milica Vlahović

Belgrade, November 02-03, 2023



# PROCEEDINGS

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## 11th International Conference on Renewable Electrical Power Sources



2023

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# INTERAKCIJE LASERA OD INTERESA ZA MATERIJALE U SISTEMIMA I KOMPONENTAMA U TRANSFORMACIJI ENERGIJE U LINEARNOM I NELINEARNOM OPSEGU

## LASER INTERACTION OF INTEREST FOR MATERIALS IN SYSTEMS AND COMPONENTS IN ENERGY TRANSFORMATION IN LINEAR AND NONLINEAR RANGES

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### *Apstrakt*

*Materijali, sistemi i komponente za transformaciju energije su veoma različiti po dimenzijama i principima funkcionisanja. Nekoliko generacija u mnoštvu sistema i transformacija, proširilo se, čak generišući nova imena od nekadašnjih samo pretvarača, do aktuatora, senzora. Među savremenim sistemima i dalje su u upotrebi merni uređaji i komponente, koji se koriste u naučnim institutima, kompanijama, aplikacijama u mass-media primenama, ali i dugogodišnji fotomultiplikatori sa organskim i neorganskim kristalima. Tanki filmovi doživljavaju širenje aplikacija i oblika. Pored rada nekih sistema, u ovom radu se razmatraju vreme života za razmatrane slučajeve, snopne tehnike i različite kombinacije tehnika. Razmatrani su neki režimi rada, kao što su rad u Q-switch režimu, režimu slobodne generacije, i cw, kao i izloženost kratkim impulsima elemenata na bazi Si / solarnih ćelija i drugih savremenih materijala, koji se tiču transformacija energije. Razmatraju se povrede nastale određenim vrstama lasera.*

**Ključne reči:** Interakcija; transformacija energije; laserska oštećenja; modulacija



## Abstract

Material, systems and components for energy transformation are very different in dimensions and principles of operation. Several generations in a multitude of systems and transformations, have expanded, even generating new names, from former to actuators and sensors. Among contemporary systems, measuring devices and components, used in scientific institutes, companies, mass-media applications etc., but also long-standing photomultipliers with organic and inorganic crystals, are still in use. Thin films are experiencing expansion of applications and forms. Besides operation of some systems, in this paper are considered the time of life for considered cases, beam techniques and various technique combinations. Some operation regimes, as operation in - switch mode, free generation regime, as well as cw, are considered, as well as exposures to short pulses of elements based on Si / solar cells and other contemporary materials, concerning energy transformations. The damages caused with certain types of lasers are considered.

**Key words:** Interaction; energy transformation; laser damages; modulation

## 1 Introduction

From a historical point of view, it would be difficult to clearly present the state-of-the art of the first acts of energy transformation made by human beings. One could begin with the expression that people have started using solar energy even without being conscious about the closest star to our planet. The next important second way to transform energy was related to the photo effect and its explanation. It could be listed without selection whether it regards to a realized transformation, proposed from the point of view of the theory, experiment and reality. In the part of the paper that puts coherent light in the foreground, we will immediately have to consider non-linear effects in addition to linear ones. This has repercussion that the basic laws which appear in education, from the first years in school to the final university courses, have to be corrected or amended with terms of various intensities and exponents. This means that the basic laws of optics appear along with deviations, that is, relativism must be included somewhere, or quadratic (or even higher exponents) along with linear terms, or a completely different modeling should be used. In some parts, catastrophe theory, chaos modeling, disintegrations and modeling where chaos transforms into coherent states can be included [1-5].

For detailed consideration of such concept, the approach to the interaction and materials should be presumed with chosen principles of final outcome, of process efficiency and the definitions of existing standard transformations, and respective materials, adequate to selected profiles of interactions both with natural and artificial radiation, should be chosen. From this point, the methods for improving the emissivity of solar panels and photo-electric targets could be theoretically and practically established.

## 2 Experiment

Two solar components were exposed to lasers in the IR and visible range in multiple modes of operation in various dynamical regimes from cw to single- and multi-pulse in the same point (target) (Tabs. 1, 2).

Table 1. Series of experiments with femto second lasers.

Laser type	Coherent Mira 900
Possible parameters depending on the samples	
Min power	1.5 W
Pulse repetition	76 MHz
Wavelength	720 nm, 800 nm, 860 nm
Unfocused beam, linear polarization (horizontally)	
Beam diameter	1 mm
Possible time of exposition in this experiment	1 s, 3 s, 5 s
For conclusion: the highest effects and damage occurred at 720 nm	

Table 2. Series of experiments with  $Nd^{3+}$ : YAG laser; the case of solar cell materials with thin layer and ms regime.

Laser of interest for medicine, e.g. Dermatology, Dentistry	
Pulse duration	$\tau = 20 \text{ ms}$
Energy density:	$300 \text{ J/cm}^2$
Pulse repetition:	1 Hz
Time of interaction/exposition:	10 pulses in the same location
The damage is marked by black arrows	

One group of the components that were related to the work of the laboratory (“Vinča”) were exposed to:  $Cr^{3+}:Al_2O_3$  and  $Nd^{3+}:YAG$  lasers. The macroscopic appearances of other group of the components are given in Figs. 1 and 5, and partial analyses of the resulting damages after the exposure was performed (Figs. 2-4, 6).

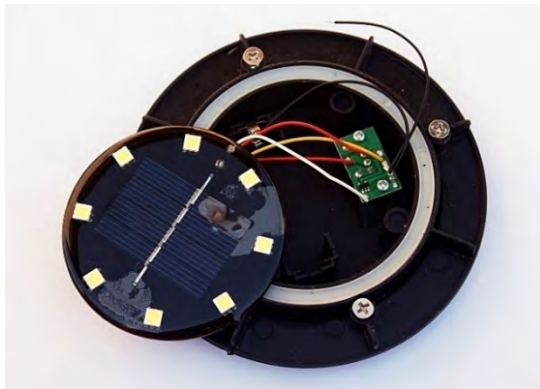


Figure 1. Device with a solar cell, macroscopic view.



Figure 2. Solar cell, damage appearance, magnification 10x, industrial light microscope.

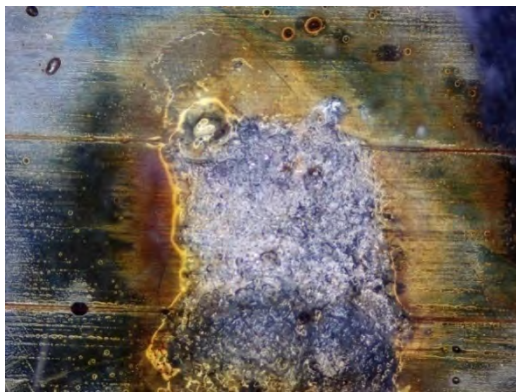


Figure 3. Solar cell, detail of damage appearance, magnification 30x, industrial light microscope.



Figure 4. Solar cell, detail of damage appearance, magnification 30x, industrial light microscope.

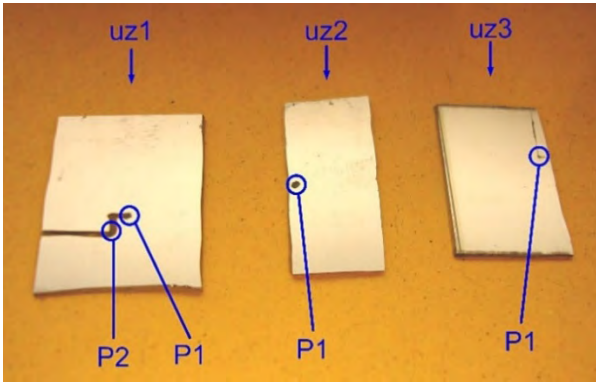


Figure 5. Pure Si, 8066, macroscopic view of samples.



Figure 6. Pure Si, Point P1 (uz3), damage appearance, magnification 10x, light microscope.

### 3 Lumped circuits

Using program packages, like SPICE and MATLAB is an efficient way for modeling of processes which include said devices (components) represented by their equivalent circuits[6]. In [7] the rate equations for the quantum well laser were modeled by corresponding equivalent circuit satisfying the same equations.

Many basic educationally courses cover the equivalent circuit theory as in [10-14]. A part of basic theory for educational detailed modeling is presented.

Transient analysis was performed in SPICE for the step power supply. Some of the models used for representing PV cell are shown in the Fig.7.

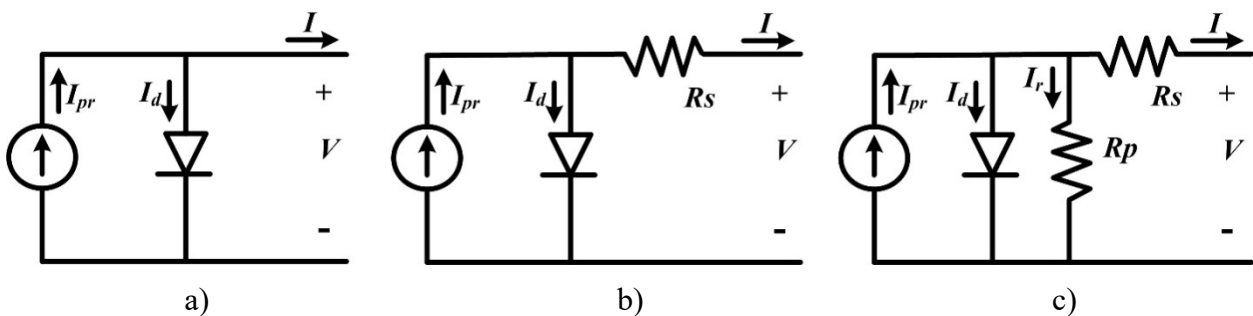


Figure 7. Three variants of PV cell model: a) ideal single diode model; b) practical diode model with serial resistance ( $R_s$ ); c) practical model with serial and parallel resistance ( $R_s$  and  $R_p$ ) [6]

Before testing the PV cell for power characteristics open circuit voltage and short circuit current need to be established[8, 9], which is a good practice in educative environments.

When measuring short-circuit current PV cell is short-circuited. From the model, it is clear that the diode voltage is 0V so its current is zero. Thus, current  $I_L$  is also a short-circuit current.

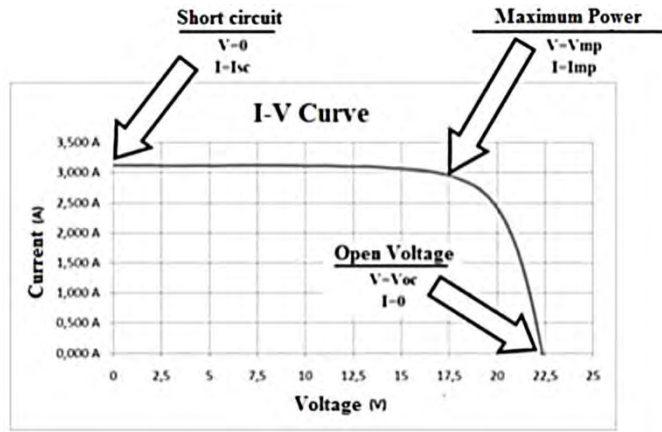


Figure 8. Current-voltage (I-V) curve of a solar cell with specific points (short circuit, maximum power and open voltage)

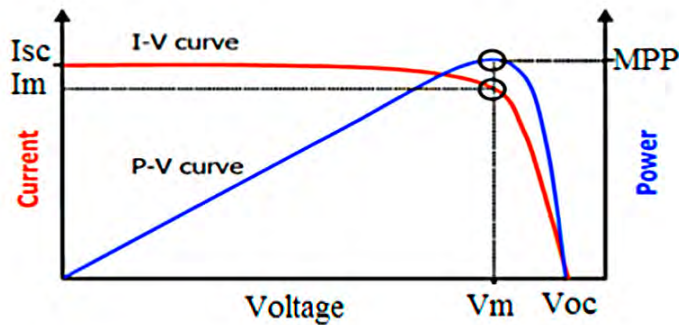


Figure 9.

Open-circuit voltage is measured with the opened circuit (resistance  $\infty$ ). Then the PV cell is connected to load. Measured current depends on the load value. On small loads, small voltage drop leads to the conclusion that the diode is not operating, but the current equals to short circuit current (or slightly differs). For large loads, the current decreases to zero. On medium loads, the current is less than short-circuit current and the curve in I-V diagram shows a downward slope. In P-V diagram (power vs. voltage), maximum value of P is unambiguously shown.

The short circuit current (i.e.  $I_L$  current, *light current* or *photocurrent*) depends on solar irradiance. These values could be representing as a family of curves.

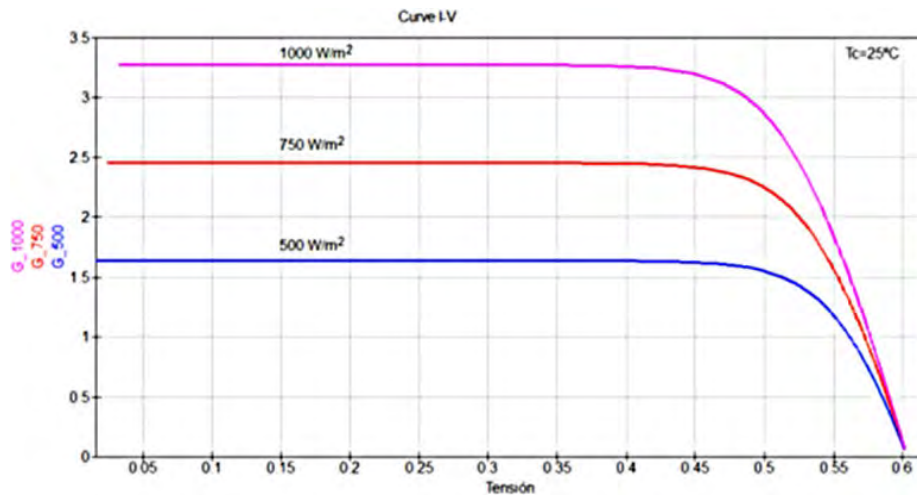


Figure 10. The short circuit current, as a family of curves, depends on solar irradiance.

### **Determining the characteristics of the PV cell.**

- a) Measurement of short circuit current  $I$ , for different solar irradiance. Determining the dependence of this current from solar irradiance  $G$ .
- b) Measurement of open circuit voltage
- c) Curve fitting for different  $G$  – movement of  $P_{\max}$  point.

Contemporary references cover many tasks in civil engineering, power engineering, sustainable sources of energy i.e. many practical problems are solved where physical or chemical type of processes can be recognized [18-23].

## **4 Interaction and simulation draft**

In the early works, near the end of the last century, there was a search for the relief caused by a certain type of laser and short pulses, which at that time were of the order of a few nanoseconds, created a flat area around the damage, practically without accumulating ejected materials around the edge of the crater. Comparing the same samples of various types of laboratory silicon, for regimes of free generations (on the order of  $ms$ , with approximately 100 to 150 spikes), typical images with accumulated layers of material around the crater were obtained. It was of interest to look for the profile of the crater, which was neither a circle nor an ellipse. At that time, the relationship between the incident injury and the theoretical spot in various materials was sought, and the ideal position for focusing the coherent beam was sought. It is often suggested that the beam be concentrated at  $1/3$  of the focal length below the surface. Since the application of lasers in connection with semiconductors and their interaction with resistive components was in its infancy, and along the way trimming of resistors, capacitors (and micro motors) were developed, many specific problems had to be solved for the industrial use of a certain type of quantum generator, related to material with mass production and dimensions prescribed tolerances. At that time, a large group of results appeared which showed the dependence of the lattice and the shape of the laser injury, macroscopically.

Many references show characteristic cases, where we had the influence of beam polarization, differences in material exposure at atmospheric pressure and in vacuum and cases in which there was an accompanying plasma, which depended on the intensity of the beam and on the target material (valence of the main of atoms in the target). Laser damage or materials cleaning are the subject of many experiments as well as modeling (solved by analytics or with computer support). Various principal description interactions with lasers are principally based on laser damage, laser cleaning processes and the cases of joining or drilling. Interaction with recoil modeling could be connected to corrosion processes. Mechanical stresses provoked by laser beam transportation are also interesting area of investigation [24-29].

### **4.1 Life time, luminescence, line shape and application**

Several of the listed concepts / processes / areas of application could cover several theoretical and practical activities, including various branches from the field of physics, metrology, different areas of the electromagnetic spectrum, with the motto of our work, energy transformation. Theoretically, one of the most common lines, from which the other two end lines are obtained by limes, with developed formalisms, are Voigt, Lorentz and Gauss lines. They are derived, on the other hand, according to the dynamics of the process in some ensemble of selected micro particles, in the broadest sense. Indicators are also related to the cosmos (astronomy), dynamics of solutions in liquids, with small or macromolecules. Developed formalisms in the field of critical phenomena, where measurements are much more difficult, in the case of magnets or neutrons, with developed theories, along with measurements in the "easier areas" of metrology, enables many practical answers and confirmations, through the rejection of hypotheses in the field of critical phenomena (BL, Stanly). The main mathematical formula for the description of the line is related to different areas of the electromagnetic spectrum, which extend from the order of kHz, MHz, to widths that are practically expressed in Kaisers. Detailed study of changes in the shape and main

characteristics of the line, for various fields, in spectroscopes of different types (atomic, molecular, etc.), has been practically mastered, with modern packages of programs and apparatus, with direct constants of certain types of materials and used for many temperature sensors, dirtiness, coagulation process, etc. On the other hand, according to the type of micro particles that participates in luminescent phenomena, Fig. 11, in the broadest sense, through PSD techniques (Pulse Shape Discrimination) [5, 15-17, 29]

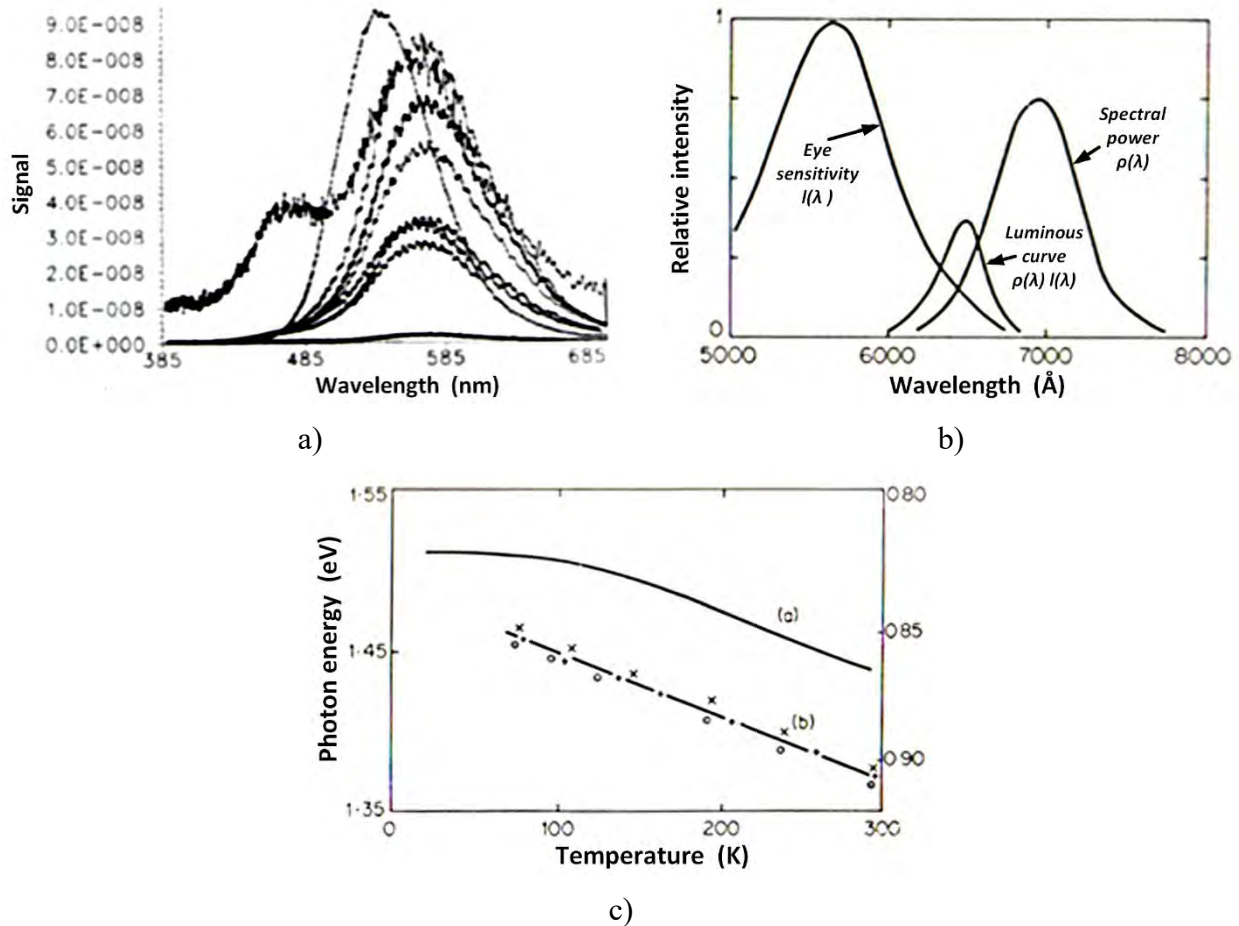


Figure 11 a) Luminescence spectra, relative intensity after irradiation with various gamma radiations intensity b) Luminous equivalent of radiation for GaP-red range. c) Emission  $\lambda$  and photon energy of GaAs lasers and laser emission with  $\lambda$ -variation among devices [15-17]

## 5 Conclusion

In the last few decades, a great deal of activity has been invested regarding the search for new energy/power sources, as well as the transformation of energy, aiming to preserve the existing civilization standard/benefits. For each type of energy and its transformation, purely for *energy purposes*, sensor roles or mass media applications, there are many different aspects of approaching, modeling, and experimental solutions, but it seems that the issues of efficiency, ecology and impact on the biosphere are fundamental. The educational aspect, in search for established paths and the selection orientation of the necessary foundations, includes many requirements for an optimal understanding of the problem. The role of coherent energy, according to the degree of quality in the broad sense and density, along with power density and dynamic mode of operation, has slowly found and continues to establish its place.

## 6 Acknowledgements

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# Some engineering methods used in biophotonics as support in the investigation of insects

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**Abstract**— Joining efforts from different fields of research is an interesting way of easing solving problems or helping developing various applications which would be more difficult the common way. Using insects studying in other-than biology areas is a good alternative and it can also help develop various engineering applications. In the other way round, studying of insects can be boosted by implementing various engineering and biophotonics methods. Studying the structure of cocoons and other parts of insects by using laser microscopy, laser induced fluorescence and femtosecond laser interaction, is selected and described. Ovaj dokument predstavlja šablon za pripremu rada sa već definisanim stilovima u samom dokumentu za određene delove rada [naslov, tekst rada, naslovi poglavlja i potpoglavlja, označavanje tabela i slika, navođenje referenci, itd].

**Keywords**—two-photon absorption, laser induced fluorescence, solitary bees, cocoons, hairs

## I. INTRODUCTION

There are many definitions and approaches to the term of interdisciplinary science, but it could be understood as the research mode where theories, concepts, tools/techniques and perspectives from multiple scientific disciplines are integrated for better understanding of issues and problems in a single discipline [1]. Although it evolved in the 20th century in response to the institutionalization and segmentation of academic research and major transitions in society, it could be traced back to the endeavors in ancient Greek philosophy or Roman engineering.

Many scientific disciplines used biology as a source, mostly medicine and pharmacology, in modern times ecology and social sciences, but also very important influence is in mathematics and computer science - informatics (artificial intelligence, neural networks, genetic algorithms, evolutionary strategies, ...), criminalistics (forensics), cultural heritage protection, food, civil engineering, ... For instance, the earliest record of entomology use in criminal investigation is the discussion in a book *The Washing Away of Wrongs* (by Sung Tzu, 1295 AD), where during the investigation of a homicide flies landed on a sickle which indicated the murder weapon and resulted in a confession by the murderer [2].

In biology, concepts and techniques from other scientific disciplines are used, like mathematics, chemistry, astronomy,

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physics, photonics, ... Here, some techniques and methods from physics, optics and photonics used in entomology research will be shown.

## II. MATERIALS AND METHODS

### A. Improving Microscopy by Two-photon Absorption, Confocal Laser Beam Scanning and Fluorescence

Microscopy, as one of the imaging techniques which enables seeing small objects commonly not visible by human eye, has significantly improved for last several decades by employing several physical concepts. In confocal scanning microscopy, point illumination is used and a pinhole is placed in front of the detector to eliminate the out-of focus signal, and in this way the optical resolution and contrast is increased. In order to obtain the information from all parts of the object, the illumination point is scanned across the object. Modern techniques use laser beam for the illumination. Two-photon absorption, though of low probability if compared to one-photon absorption, is a process used in microscopy with advantages like preserving objects and increasing the penetration depth. Low probability of the absorption is overcome by using high intensity illumination, like laser pulsed beams. In two-photon absorption, two photons of lower wavelength are simultaneously absorbed to excite an atom or a molecule to an excited state via a virtual energy level. This concept is used in two-photon excitation fluorescence microscopy, where the atom or molecule fluoresces after the two-photon excitation and the fluorescence wavelength is shorter than the excitation wavelength. Using lower excitation wavelengths (mainly infrared) preserves the materials of biological origin, e.g. tissues. Using confocal laser beam scanning microscopy together with two-photon excitation fluorescence the imaging is significantly improved. The advantages are optical sectioning (small volume excited), longer wavelength excitation (preserving tissues), increasing the penetration depth, no need for dyeing, while the disadvantage is the higher equipment cost, since mostly ultrafast lasers are used for illumination (excitation).

### B. Nonlinear Laser Microscopy in Entomology Research

The microscopy that uses laser beam scanning, two-photon excitation fluorescence and confocal scanning would have a long and a complex term. For this reason, it is in many

occasions called nonlinear laser microscopy. It is an innovative method in entomology research, which offers the simplicity in sample preparation and enables the monitoring of tiny and hidden structures, whereas standard light microscopy poses some difficulties. Fluorescence is exploited in the sense that chitine autofluorescence enables the investigation of insects structures [3], [4], [5].

### C. Setup

The apparatus used in the experiments was a standard light microscope (Carl Zeiss – Jena), modified, reconstructed and upgraded in the Institute of Physics Belgrade (see acknowledgments section), Fig. 1. The source of the illumination beam is Coherent laser system consisted of Mira 900F with femtosecond beam as an output (739 nm, pulsed, ~150 fs, 76 MHz) and Verdi V12 (532nm, CW, 9.3W) as a pump laser. A beam splitter with a detector is used to use small part of the beam for the power measurement. Laser beam scanner and a home-made telescope (beam expander) were added to the microscope body in order to scan and introduce the beam. Dichroic and fluorescence filters separate IR (illumination) part from the mid-range visible (light microscopy) and violet (photomultiplier detection).

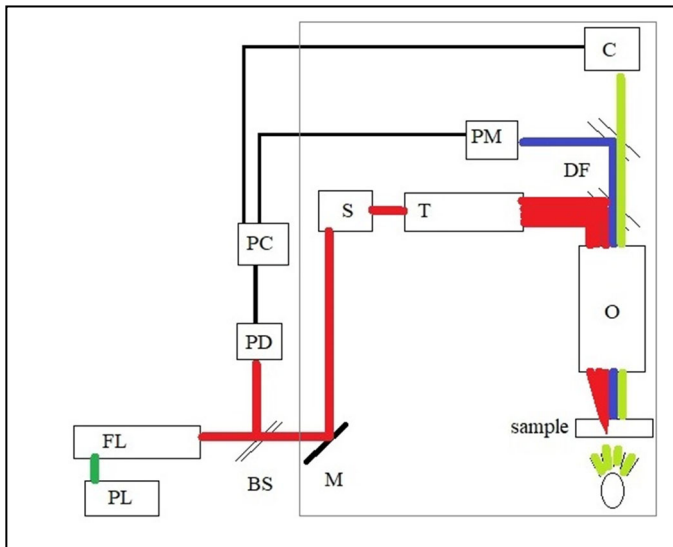


Fig. 1 A schematic representation of the setup used in the experiments. PL – pump laser, FL – femtosecond laser, BS – beam splitter, PD – photodetector, M – mirror, S – XY scanner, T – beam expander, DF – dichroic filter, O – objective, C – camera, PM – photomultiplier, PC – computer.

Added devices for observation were Canon EOS 100D camera and a photomultiplier. A PC, equipped with home-made software for the integration of the software for the control of the devices (photodetector, scanner, photomultiplier, camera), for imager analysis and for the system control, was a control part of the system.

The beam from the pump laser (green) excites the femtosecond (fs) laser to emit the NIR beam (red). One, small, part of the fs beam is used to measure the power, while the greatest part is introduced into the microscope. The scanner (S) enables the beam to scan across the surface of the sample. The beam expander (T) widens the beam in order to be more tightly focused by the objective (O). The fluorescence signal (blue), occurred due to the two-photon absorption, passes through the

dichroic filter (DF) and is directed to the photo-multiplier (PM). A camera (C) is used to monitor the sample in visible light (green).

### D. Solitary Bees

Solitary bees are in the family of Megachilidae, and are one of the best pollinating agents. The objects of this investigation are several examples of the genus *Osmia* (mason bees): *O. caerulescens*, *O. cornuta* and *O. bicornis*.

Characteristic of *Osmia* are: use hollow small tubular spaces for eggs, like hollow branches or common reed straws. They have common four-stage development (egg, larva, pupa, adult); larva spins cocoon around itself and enters a pupal stage. An adult matures either during autumn or winter by hibernating inside its insulatory cocoon. The anatomy shows head (three small ocelli, two large compound eyes, antennae and mouth), thorax (six legs and four wings) and abdomen (scopa for collecting pollen – females only; scopa is a cluster of hairs). Pollination is very efficient due to both the anatomy and the behavior. A bee is almost completely covered with hairs for pollen collecting [6] and it also “dances” in a flower in order to collect as much pollen as it could. Foraging occurs in early spring mainly on apples, pears, almonds, strawberries, even in bad weather.

A bee uses its hairs to collect pollen and oil, as well as for the thermo-regulation. A cocoon is made of silk (a protein polymer excreted by labial glands), and is consisted of two to three layers with the roles of protection, diffusion of gases and waterproofing. Samples (cocoon and hairs) were prepared in a standard way [7].

## III. RESULTS

The results were obtained by microscopic analyses in light as well as in nonlinear laser microscopy. In *O. cornuta* and *O. bicornis*, the cocoon is consisted of three layers, while in *O. caerulescens* of two layers, Fig. 2 [7].

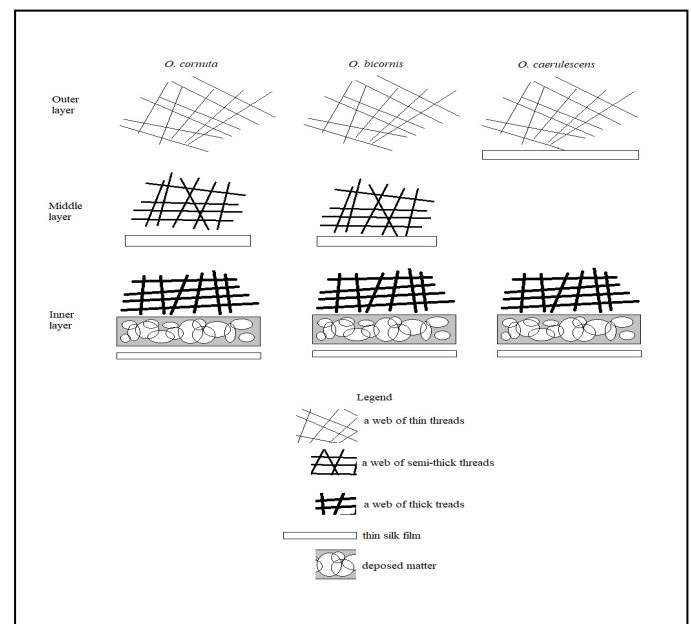


Fig. 2 Structural presentation of the cocoon wall of three *Osmia* bees. A legend shows the types of sub-layers.

Sub-structure of the cocoon layers in *O. cornuta* and *O. bicornis* shows that the outer layer is a net of silk threads, the middle layer is also a net of silk layers supported from the inner side by a thin silk film, and the inner layer is made of three sub-layers: a net of silk threads, a layer of deposited material, like pollen, and a thin silk film as the most inner sub-layer. In *O. caerulescens*, the outer layer has two sub-layers: a net of silk threads supported by a thin silk film, and the inner layer is of same structure as in *O. cornuta* and *O. bicornis*. Increasing the number of layers more serves as protection against predators than against water, since only one layer is sufficient for the defence.

The thin silk film of the outer layer in *O. caerulescens* probably originated from widening of silk threads, as indicated by red arrows in Fig. 3.

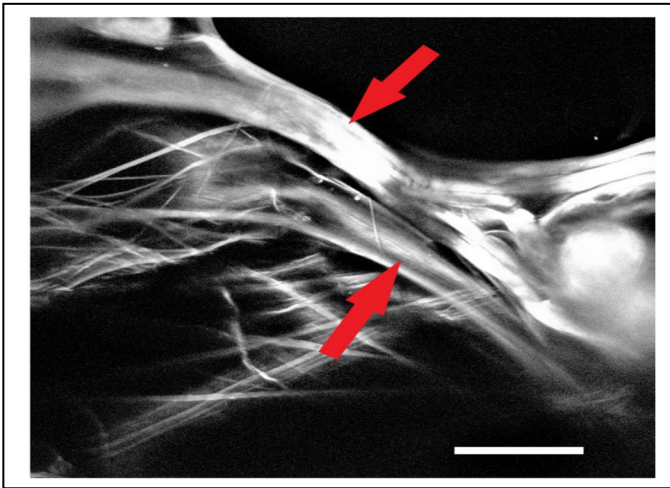


Fig. 3 Nonlinear laser microscope image of the outer layer of *O. caerulescens*. Red arrows indicate widening of the threads. White bar denotes 50  $\mu\text{m}$ .

Fluorescence microscopy shows the middle layer of *O. bicornis* is consisted of silk threads supported by a thin silk film, Fig. 4. Some of the threads are widened and formed a thin film which supports the other threads. The threads of the outer layer are thinner and their color is lighter than the threads of the middle layer.

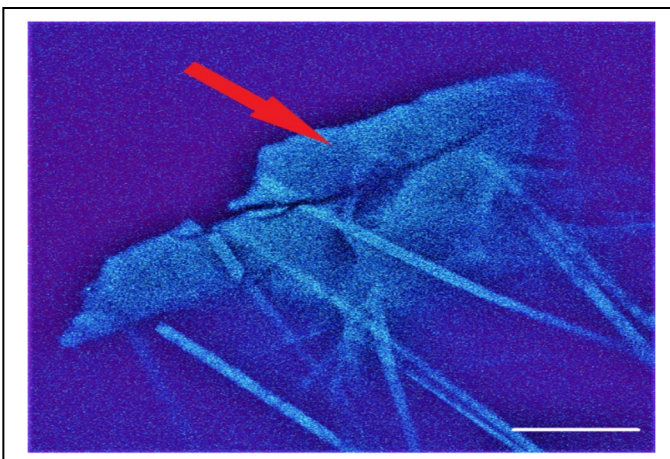


Fig. 4 Nonlinear laser microscopy image of the middle layer of *O. bicornis*. Red arrow indicates a sublayer of a thin silk film. White bar denotes 50  $\mu\text{m}$ .

The advantage of using laser fluorescence microscopy over common light microscopy is the visibility of the thread structure. In Fig. 5, the middle layer noticed in both *O. cornuta* and *O. bicornis* is consisted of two sub-layers, similarly to the outer layer of *O. caerulescens*. The outer sublayer is a net of silk threads, but the net is thicker and more compact than in the outer layer and is supported by a thin silk film.

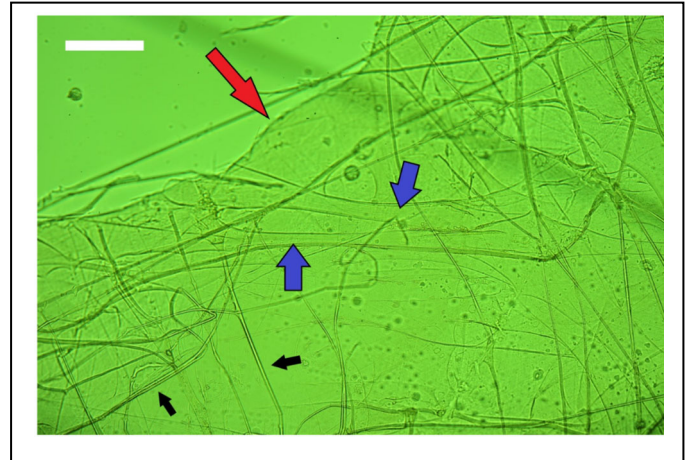


Fig. 5 Light microscopy image of the middle layer of *O. bicornis*. Red arrow indicates a sublayer of a thin silk film, blue arrows indicate widened threads which integrate into the thin silk film, and black arrows indicate free silk threads. White bar denotes 50  $\mu\text{m}$ .

In Fig. 6, red arrows point to surface bumps in threads of *O. cornuta*, where the silk accumulated. White arrows point to elongated parts, probably tensioned. The threads of the inner layer are around 5  $\mu\text{m}$  thicker than the threads of the outer layer.

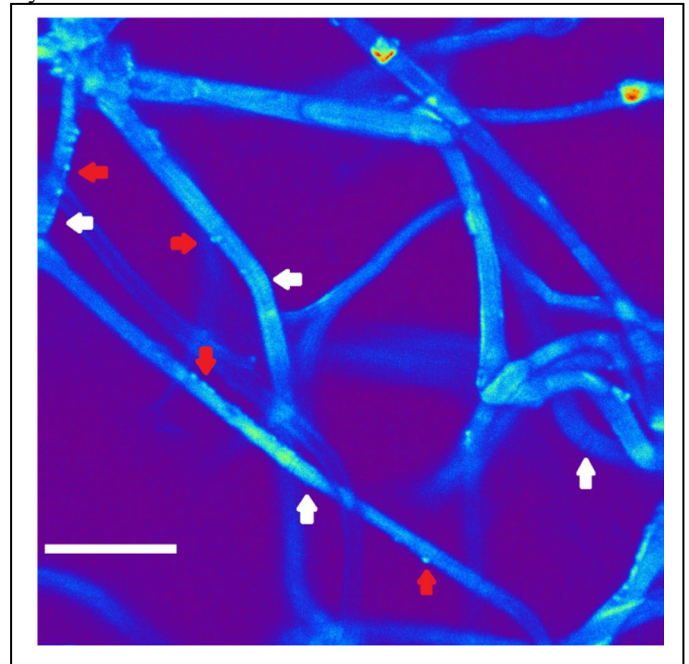


Fig. 6 Nonlinear laser microscope image of the middle layer of *O. cornuta*. White arrows indicate tensions of the threads, while red arrows indicate bumps on the threads. White bar denotes 50  $\mu\text{m}$ .

The hair from the scope of *O. bicornis* is around 20  $\mu\text{m}$  thick and it exhibits spiral rows on the hair surface, Fig. 7. The

presence of the rows probably improves the fetching of pollen grains on the hair surface.

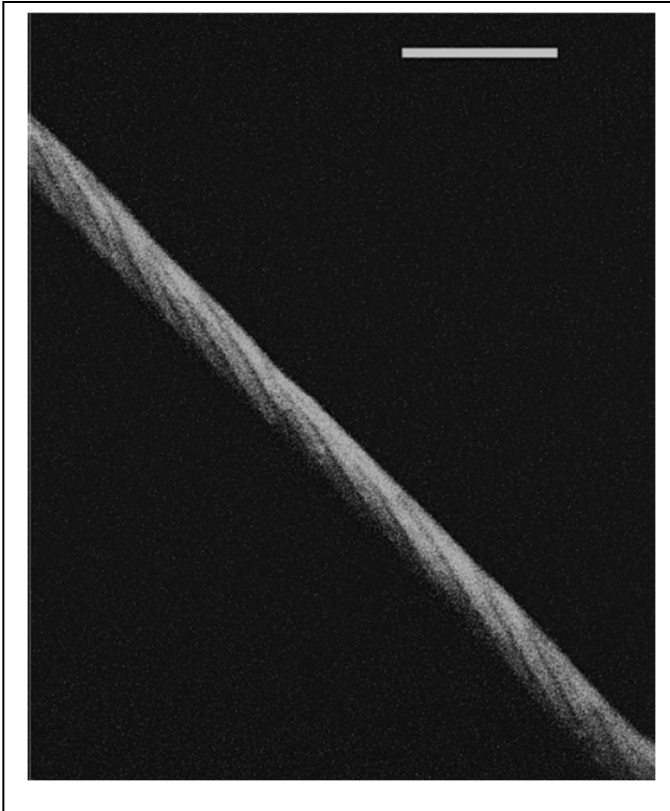


Fig. 7 Nonlinear laser microscope image of a hair from the scopa of *O. bicornis*. White bar denotes 50  $\mu\text{m}$ .

#### IV. CONCLUSION

The implementation of physical concepts of two-photon absorption, confocal laser beam scanning and fluorescence, the investigation in entomology is significantly improved. Through nonlinear laser microscopy a better insight in the morphology of the cocoon and hairs of the taxa within the genus *Osmia* is given. Due to this method, the investigation and the characterization of the structures of *O. cornuta*, *O. bicornis* and *O. caerulescens* is more precise and simpler than it would be by the means of common light microscopy.

In *O. cornuta* and *O. bicornis*, three layers in the cocoon wall have been determined, while in *O. caerulescens*, the cocoon wall is built of two layers. There is a similarity in substructure of the inner layer of all three species. In all three species, the hairs from the scopa have spiral rows on the surface which improves pollen collecting.

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## PREFACE

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The aim and main idea of the Conference was to present science and scientific way of thinking and working closer to the students, as they will be able, in the future, when they are employed, to connect science and industry. For this reason, the right to participate had only undergraduate and master students, who, with help and monitoring by their teachers and/or colleagues, wrote and prepared papers and presentations.

The aim of this Conference was, also, to provide a Forum for students and researchers from various countries to exchange their ideas and achieved results.

The Conference brought together the participants from Universities, Innovation Centres and Institutes from different countries: Croatia, Romania, Bosnia and Herzegovina, Macedonia, France, Russia, Montenegro, Spain, Republic Srpska, Slovenia and others.

The aim of the conference was, also, to connect different/various fields of science, because we can find many common points between different research areas, and by doing that, to open possibilities of developing new technologies or improving the old ones. Therefore, the Conference covers various topics from the following fields: mechanical science, transport and traffic engineering, material science, metallurgy, electrical engineering and other engineering areas, but all other sciences as well, including for example medical science, which uses different techniques of experimental examination and testing.

Although, the Conference had multidisciplinary character, the participants had very active discussion after the presentations and we hope that it will provoke the further cooperation between them with a new point of view.

The paper presentation was by oral and poster, due to limited time of Conference duration and traffic conditions of participants. The all papers are reviewed. Considering that this was the Students Conference and the age and experience of the first authors, the reviewers *had neglected* language and textual mistakes which were not provoked the ambiguity of the papers.

We would like to thank all authors who have contributed to this Proceedings and also to the Scientific Committee, Organizing Committee, reviewers, speakers, chairpersons, and all the conference participants for their support for a successful scientific meeting.



## CONTENT

1. BIOINFORMATICS ANALYSIS OF ASXL1 PROTEIN MUTATIONS: IMPORTANT BIOMARKERS OF MYELOID MALIGNANCIES Emilija Jovanovic, Branislava Gemovic.....	1
2. THE POSSIBILITIES FOR EXPLOITING GEOTHERMAL ENERGY IN PUBLIC INSTITUTIONS IN THE NOVI GRAD MUNICIPALITY Operta Adi, Haris Jahic .....	10
3. A SURVEY OF LANDSLIDES OBSERVATION METHODS Dorotea Markasovic, Marina Jozic, Nina Krljar, Krunoslav Minazek.....	15
4. PRODUCTION AND CHARACTERIZATION OF CERAMICS FROM CLAY AND CONSTRUCTION & DEMOLITION WASTE Stefanija Petrovska, Monika Fidanchevska, Biljana Angjusheva.....	29
5. OPTIMIZATION OF THE MAIN PROCESS PARAMETERS OF CERAMICS PRODUCED FROM CLAY AND DEMOLITION WASTE Nebojsa Levkovski, Luka Iloski, Biljana Angjusheva.....	35
6. PVC/FLY ASH POLYMER COMPOSITES Kosta Petrovski, Tamara, Georgievska, Raman Aliti, Anita Grozdanov.....	42
7. TESTING OF METALWARE BY THE ED-XRF SPECTROMETRY Ana Janicijevic, Milena Radomirovic, Antonije Onjia, Slavka Stankovic.....	54
8. ORGANIZATION AND MANAGEMENT OF PE "NP ĐERDAP": ANALYSIS OF STAKEHOLDERS ATTITUDES IN THE AREA OF MAJDANPEK MUNICIPALITY Mladen Prvulovic, Jelena Nedeljkovic.....	62
9. VARIABILITY OF MORPHOLOGICAL LEAF CHARACTERISTICS OF EUROPEAN WHITE ELM ( <i>Ulmus laevis</i> Pall .) SEEDLINGS Marija Mitrovic Stevanovic, Ivana Radakovic, Marina Nonic, Mirjana Sijacic-Nikolic.....	76
10. INFRARED-THERMOGRAPHY BASED THERMAL ANALYSIS OF THE OLD VS. NEW TYPE OF THE NON-RESIDENTIAL BUILDING CONSTRUCTION AT THE IDENTICAL GEO-LOCATION AND ORIENTATION, Marina Irina, Isidora Rilak, Ivan Zlatanovic.....	87
11. SMARTPHONE APPLICATIONS IN TRAFFIC Slavko Srdic.....	94
12. MODERN APPROACH TO TESTING AND EDUCATION OF CHILDREN Andjela Josic, Ivana Stokic, Svetlana Čicevic, Aleksandar Trifunovic.....	100
13. MULTIDISCIPLINARY APPROACH FOR SOLVING SERIOUS ACCIDENTS IN RAILWAY TRANSPORT Nikola D. Milutinovic.....	107

14. RELATION BETWEEN DIRECT AND INDIRECT TAXES IN REPUBLIC OF SERBIA Jelena Todorovic.....	116
15. MICROECONOMIC APPROACH TO MULTIDISCIPLINARY RESEARCH Luka Jovanovic, Kristina Micoski--.....	123
16. PROGRAM FOR SOLVING SYSTEM OF ORDINARY DIFFENTIAL EQUATIONS WITH NUMERICAL METHOD RUNGE-KUTTA 10 <sup>TH</sup> ORDER Vukasin Brkovic, Stefan Spalevic.....	134
17. MODERN METHODS FOR SUPRESSING TERRORIST ATTACKS Jasmina Bajramovic.....	145
18. DESIGN AND CONTROL OF EDUCATIONAL MOBILE ROBOT FOR MATERIAL TRANSPORT IN INTELLIGENT MANUFACTURING SYSTEM Mihajlo Rusov , Milica Petrovic , Zoran Miljkovic .....	151
19. QUADCOPTER CONTROL BASED ON TELEMETRY DATA USING A 6DOF FLIGHT SIMULATOR Milos Petrasinovic, Mihailo Petrovic, Veljko Petrovic.....	159
20. TORSIONAL RIGIDITY OF A FORMULA STUDENT CAR Branko Milicic, Lazar Stojnic.....	181
21. HYDROXYAPATITE STRUCTURES AND NUMERICAL ANALYSIS OF THE GEOMETRIC CHARACTERISTICS OF THE LOWER JAW FIXATION PLATES Milovan Paunic, Igor Balac, Katarina Čolic, Aleksandar Sedmak.....	190
22. ENERGY MANAGEMENT SYSTEM Milena Radovanovic, Marija Orlovic, Teodora Savanovic, Andjela Mijovic.....	198
23. DURABILITY OF CONCRETE STRUCTURES (MAGNETIC NANO POWDER AS A WAY FOR REDUCING POROSITY FOR LONGER DURABILITY OF CONCRETE STRUCTURES) Bogdan Sakic; Damjan, Stancic.....	211
24. ASTROCYTES INFLUENCE ON DIFFERENTIATION OF STEM CELLS FROM APICAL PAPILLA INTO MATURE NEURONS IN VITRO Jovan Mitic, Andrej Korenic.....	219
25. IMPLEMENTATION OF IMAGE-BASED VISUAL SERVOING FOR NONHOLONOMIC MOBILE ROBOT CONTROL Aleksandar Jokic, Milica Petrovic , Zoran Miljkovic.....	223
26. EXPERIMENTAL ANALYSIS OF SINGLE TRAPEZOIDAL WING, PRESSURE DISTRIBUTION METHOD. COMPARATIVE ANALYSIS OF THE NUMRICAL METHOD (FLUENT) WITH THE RESULTS OBTAINED BY THE EXPERIMENT Srdjan Kostic, Tamara Krzojevic, Aleksandra Veljkovic, Caslav Mitrovic.....	236
27. SELECTION OF THE MOST EFFICIENT TEMPERATURE MEASUREMENT METHOD BASED ON DESIRED RESPONSE TIME AND EXPERIMENTAL RESULTS Lara Laban, Nedzad Rudonja, Milan Gojak.....	247

28. TOWARDS IOT-SUPPORTED BUSINESS PROCESS MANAGEMENT: A CASE STUDY Boro Mijovic, Tatjana Sibalija.....	255
29. PROCESS OF THE "EASYCAR" MOBILE APPLICATION Srdjan Stevanovic, Katarina Kaplarski.....	267
30. THE INTERDISCIPLINARITY OF MOBILE APPLICATION SOFTWARE ENGINEERING, Stefan Radenkovic, Katarina Kaplarski.....	279
31. THE ROLE OF BUSINESS PROCESS MANAGEMENT AND ITS IMPLEMENTATION IN TELECOMMUNICATION SECTOR USING SAP'S METHODOLOGY Stefan Jovanovic, Tatjana Sibalija.....	287
32. ANALYSIS OF THE PROPERTIES OF CONCRETE WITH AGGREGATE MADE OF CRUSHED BRICK Kociz Puric.....	296
33. UNE ANALOGIE ENTRE LE DÉVELOPPEMENT DES DROITS CHEZ LES BARBARES APRÈS LA CHUTE DE L'EMPIRE ROMAIN Ostojic Sofija.....	304
34. NANOSTRUCTURES FOR SOLAR CELLS Dusko Kostic, Svetlana Pelemis.....	309
35. IMPROVED CAVITATION RESISTANCE OF ACRYLOID COPOLYMERS REINFORCED BY ALUMINA SUBMICRON PARTICLES Radmila Damjanovic, Marija Vuksanovic, Mina Jovic, Irena Zivkovic, Tatjana Volkov – Husovic, Radmila Jancic Heinemann.....	313
36. CABLE MAINTENANCE AND SOFTWARE TOOLS Jelena Jovanovic, Marko Krezic, Sanja Jevtic, Dragan Jevtic.....	318
37. GAME AND CREATION – SPATIAL ABILITY TEST AIDED BY 3D MODELS AND AR CELL PHONE APPLICATION Filip Milenovic, Mateja Korica, Valentina Marjanovic, Stefan Milosavljevic, Magdalena Dragovic, Aleksandar Trifunovic.....	327
38. WHERE PHYSICAL AND VIRTUAL MODELS MEET: VISUALISATION OF 3D GEOMETRIC TASKS Tamara Matijevec, Filip Gramic, Nemanja Krtinic, Filip Kojadinovic, Magdalena Dragovic.....	336
39. CONTEMPORARY SOLUTIONS FOR 3D VISUALISATION OF A CULTURAL HERITAGE MONUMENT Marina Markovic, Katarina Jeremic, Danijel Demir, Suzana Polic, Magdalena Dragovic...	345
40. TESTING METHODOLOGIES OF PASSINGER BODYCARS Sasa Vasiljevic .....	352
41. MODIFIED APPROACH FOR VEHICLE TESTING AS A MEASURE TO IMPROVE AERODYNAMICITY OF A VEHICLE Sasa Vasiljevic, Marko Radisavljevic.....	360

42. CONTEMPORARY MEASURING METHODS IN PHARMACEUTICS FOR PREPARATION AND DIAGNOSTIC OF MEDICAMENT QUALITY WITH OPTICAL METHODS APPLICATION Biljana Fidanovski, Bojana Fidanovski , Svetlana Pelemis .....	369
43. APPLICATION FOR THE SERVICE OF THE ICT SERVICE (UK + BUS) Radenko Malesevic.....	375
44. EFFECT OF TEST GROUPS ON THE EXTENT OF NONDESTRUCTIVE TESTINGS OF PRESSURE VESSELS WELDS Dimitrije Maljevic, Aurel Valentin Birdeanu, Sanja Petronic.....	387
45. SOLVABILITY BY RADICALS AND APPLICATION Matija Srečković.....	396
46. SOME EXAMPLES OF LOW FLUENCE LASER APPLICATIONS IN THE PROTECTION OF PLANTS AND CULTURAL HERITAGE Ana Kovačević, Aleksander Kovačević.....	403
47. SOCIO-ECONOMICAL IMPACTS OF BIO-CONSTRUCTION Kasija Bojanic.....	409
48. PURIFICATION OF WATER CONTAMINATED WITH PHENOL BY THE PROCESS OF HYDRODYNAMIC CAVITATION Djordje Ogrizovic, Borivoj Adnadjevic.....	410



# SOME EXAMPLES OF LOW FLUENCE LASER APPLICATIONS IN THE PROTECTION OF PLANTS AND CULTURAL HERITAGE

Ana Kovačević, Aleksander Kovačević

**Abstract:** Many objects of cultural heritage are susceptible to degradation caused by biological factors such as bacteria, fungi, lichen and insects. Stone walls and facades, frescoes, paintings on canvas/paper, books and clothes are specially susceptible if being present in the conditions convenient for the development of biodegradable factors (higher humidity, low ventilation, non-frequent cleaning). Non-invasive means for the detection of these factors are needed, as well as the analysis of the materials for better understanding of their structure with the aim of more efficient preservation. This is achieved by the application of low-fluence lasers, due to the non-destructive nature of the interaction, and with laser induced fluorescence and reflection microscopy with methods. Moreover, it is possible in this way to detect the infections of plants, e.g. caused by fungi. In this work, some cases of the application of low fluence lasers in the protection of plants and cultural heritage objects is described.

## 1 INTRODUCTION

Being present in the environment, the cultural heritage objects are exposed to the influence coming from the environmental parameters or from the biosphere (biological mechanisms), where bio-transformation process is spread worldwide. One of the methods is to detect the presence of agents of weathering process in order to slow down the deterioration.

In this work, after describing and analyzing, a convenient method has been selected for the detection of various layers – linked to living microorganisms or their prehistorical remains. For this reason, it is valuable to create abundant database on various characteristics and descriptions of specific micro-organisms or remnants of macroscopic artefacts for which it is assumed that are of bio-organic origin having in mind the importance of the aspect of bio-receptivity as the aptitude of a material to be colonized by bio-agents.

Among the others descriptions, “more classical” descriptions are, in the areas of biology, tied to the apparatuses and elements of specific disciplines, as well as to the diagnostic methods in biochemical developed theories and practice. The shift to the diagnostic methods utilizing optics needed, and still needs, specific optical characteristics of given ensembles where individual element (bacteria, virus, ...) is observed as an micro-object. Some time ago, obtaining first images of macro-molecules has been accepted as great achievement of descriptive science; nowadays, contemporary techniques linked to theories enter the world of nano-dimensions and, in the coupling of diffraction results with the possibilities of electron microscopy and dispersion, already pose solid base for research.



One of the common analyzing methods is using coherent radiation for the excitation of atoms/molecules of target object. The interaction of low-fluence laser beam with the target does not provoke damage of the material, but provides the excitation leading to the emission of photons as relaxation – laser-induced fluorescence (LIF). Commonly, the materials are excited by ultraviolet (UV) radiation, but it is not unexpected to implement the radiation from visible (VIS) region as well.

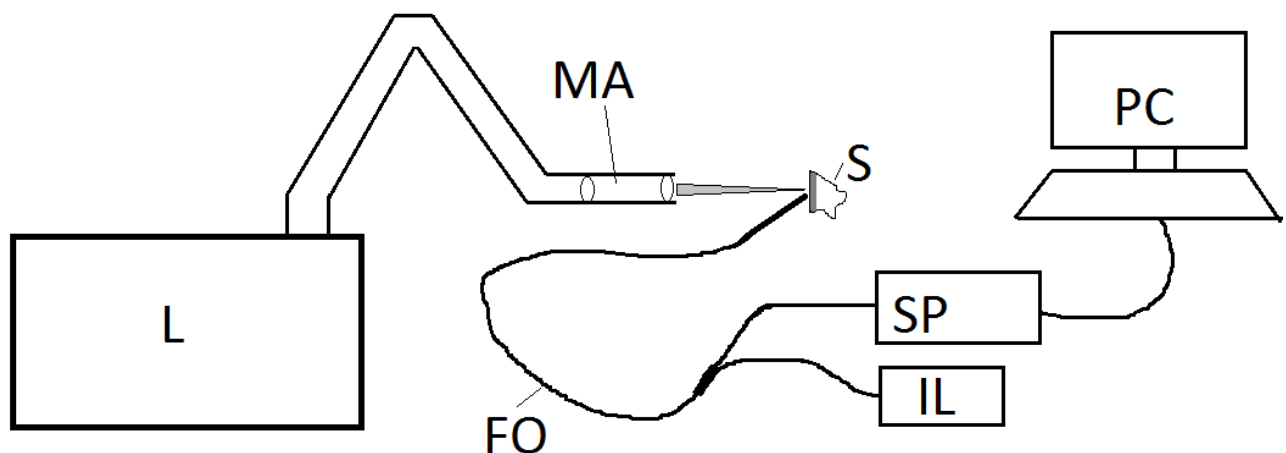
For cultural heritage protection, the bio-agents possessing unique characteristics susceptible to the interaction with coherent radiation and that could lead to deterioration can be effectively detected by passive methods of coherent detection. The presence of chlorophyll in the bio-agents is detected by its fluorescence lines in the spectra obtained after the laser action.

The light reactions of photosynthesis are carried out by four large protein complexes: Photosystem II (PSII), 2 cytochrome b6/f, Photosystem I (PSI), and ATP synthase, where PSI – located in the thylakoid membrane of cyanobacteria, algae, and plants – is considered to be the most efficient light-capturing and energy conversion apparatus found in nature, due to its almost 100% quantum efficiency [Drop2011].

Here is described the method of the detection of the presence of the bio-agents containing chlorophyll, with some discussions on the origins.

## 2 EXPERIMENTAL SETUP

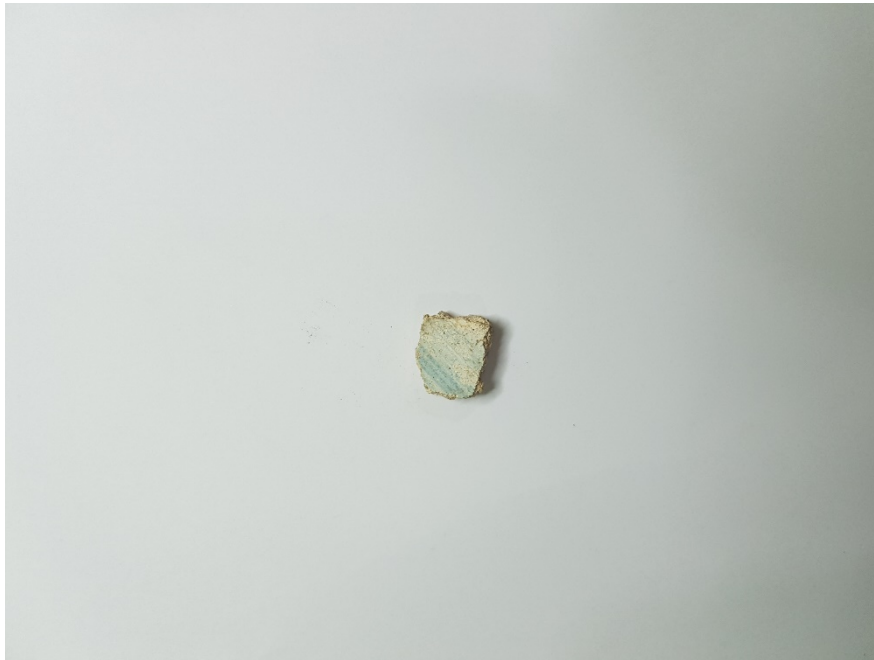
For the illustration of the LIF on objects of cultural heritage, the setup has been performed as presented in Fig. A.



**Fig. A.** Experimental setup for monitoring the LIF on the fragments of frescoes, L – laser, MA – manipulating mechanical arm with directing and focusing parts, S – simple, FO – fiberoptic cable, SP – spectrometer, IL – illuminating unit, PC – personal laptop computer with software.

The laser used in the experiment was “Thunder Laser Q1” DNA from “Quanta System”. The spectrometer was “Black Comet CXR-SR-50” by “StellarNet”. The illuminating unit was a standard “Illum-A” illuminator, “SL-1 Filter” by “StellarNet”. The software for fetching and processing the data from the spectrometer was “SpectraWiz” by “StellarNet”.

The sample used in the experiment was a fresco fragment labeled “Blue 37-1” from late antiquity archeological site Mediana near Niš. The fragment is of oval shape, 15x10 mm, one flat side painted in shades of blue (photo presented in Fig. B).

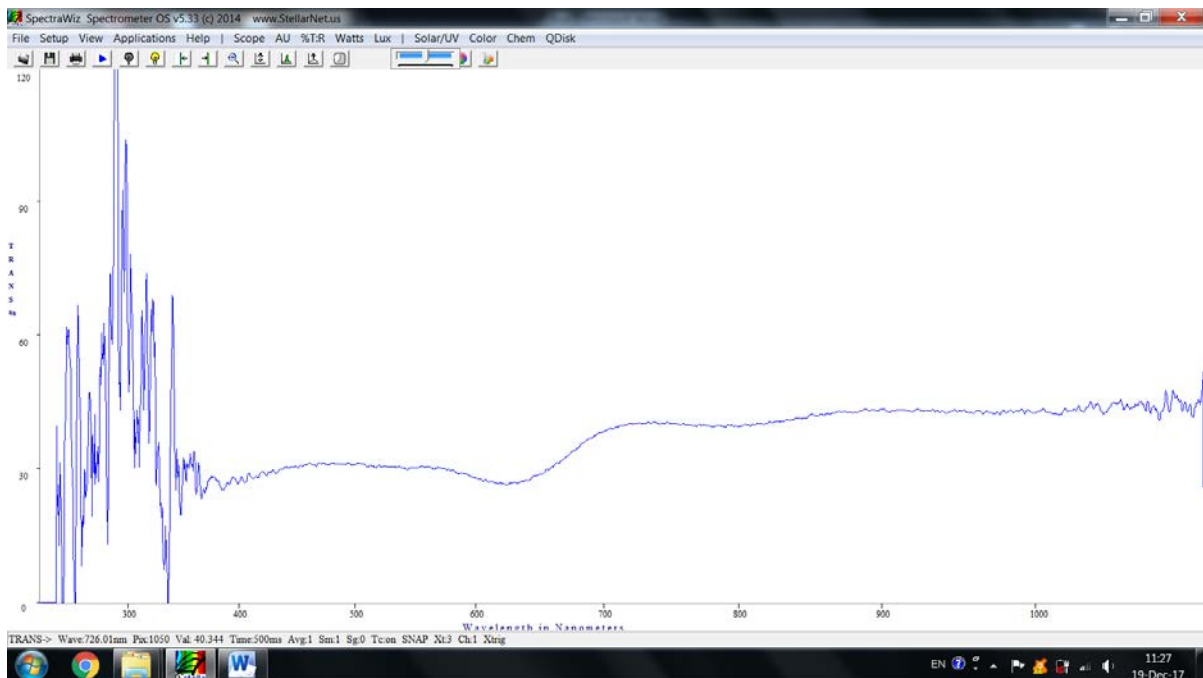


**Fig. B.** A photograph of a sample “Blue 37-1”.

### 3 RESULTS

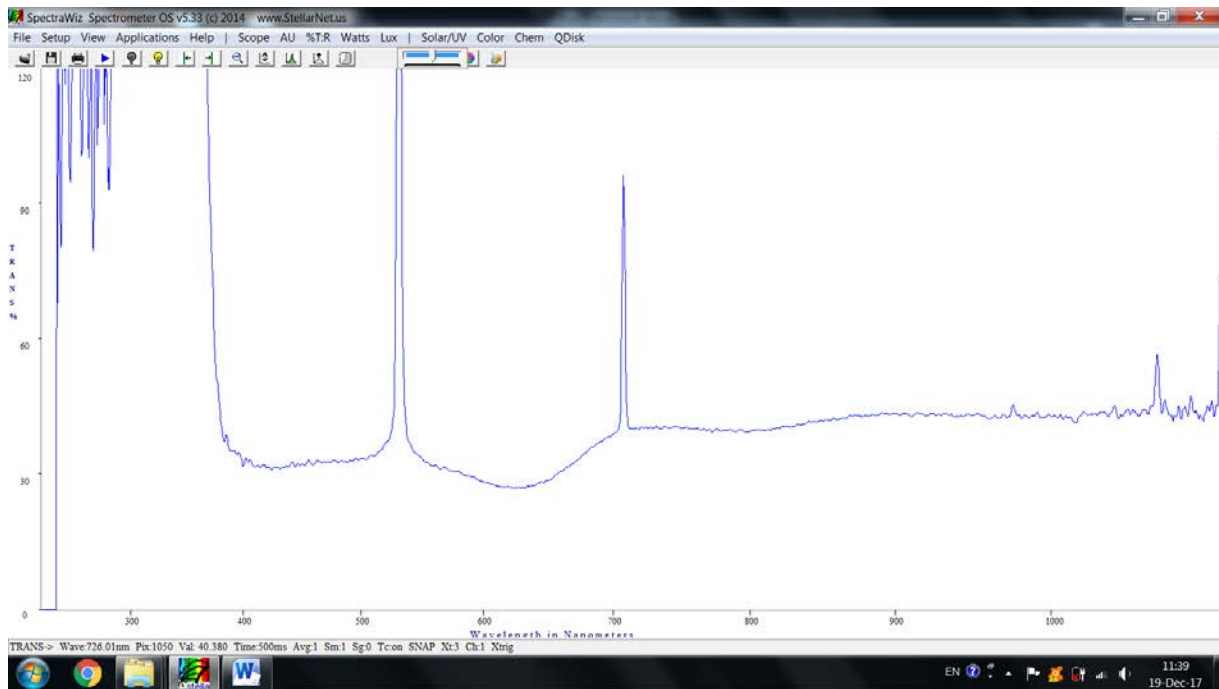
The samples were irradiated with a laser beam of the characteristics: wavelength 355 nm, output power fluence  $\sim 32 \text{ mJ/cm}^2$ , pulsed regime with 20 Hz repetition rate and pulse duration of  $\sim 10 \text{ ns}$ . Simultaneously with the irradiation, a fiber from a fiber-optic spectrometer was applied to the sample surface to fetch the fluorescence.

A spectrum from the sample surface during no laser irradiation is presented in Fig. C, while during the irradiation of UV 355 nm beam is presented in Fig. D.



**Fig. C.** A spectrum from the sample “Blue 37-1” during no laser irradiation.

The spectrum during no laser irradiation shows relatively high level of reflection in blue-green and (particularly) infrared (IR) areas. The depression in the zone 550-650 nm shows the lack of “red” in the reflected light. High reflection for wavelengths higher than 700 nm gives no impression for the human observer.



**Fig. D.** A spectrum, from the sample “Blue 37-1” surface during the irradiation of pulsed 355 nm beam.

During laser irradiation, several features are notable. The rise of the UV part (wavelengths less than 400 nm) is due to the laser irradiation. The presence of the peaks at 532 and 1064 nm are parasitic radiation from the laser (basic and 1<sup>st</sup> harmonics) but of low fluence. The shape of the spectrum in the visible and IR areas has not changed significantly.

Strong peak at ~710 nm, as well as weak peaks at ~980 and ~1020 nm are the result of fluorescence.

#### 4 DISCUSSION

Specifically, the methods of coherent optics helped the broadening of the database for “less common” descriptions of micro-organisms: bacteria, fungi, lichen and insects.

Main processes that find the place in the measuring method are linked to the dispersion (elastic or inelastic) techniques, absorption and fluorescence, and also to the beams of coherent photons in different diapasons of electromagnetic (EM) spectrum, of electrons, or of nuclear radiation radiations and particles and photons originated from various quantum generators. If linear and non-linear phenomena are included, which depend on the conceptual experimental support, the choice of possible methods is great, nevertheless the relatively less number of them is included in standard research. Some of the spectroscopies are in operation for more than several decades [Kovačević2015].

Description of micro-objects, dispersion theory, including the angular distribution and methods of photon mixing, provide data on the dimensions and shape of micro-objects, their layers,





regularity (sphere, stick, Gauss curve). Developed theories, towards the dimensions of micro-objects, determine the spans of photon wavelengths or DeBroglie joined wavelengths, by which specific parts of a measurement system and apparatus parameters are determined.

More exotic view of bacteria would be by equivalent dipoles and Kerr constants linked to electro-optical and electromagnetic and other effects.

Among the most successful (and relatively less intricate) methods, resonant methods are being used for a long time, where – by convenient excitation based on: nuclear radiation and particles with chosen parameters or frequencies of a specific quantum generator – excite spectra characteristic for bio-organic materials. Quantum generators, with possibilities of higher harmonics, parametric effects and frequency tuning, represent abundant base for finding specific convenient frequencies for the excitation of fluorescent spectra. Excitation spectra should be searched for pathogen conditions of attacked objects.

The peak at ~710 nm is of high intensity. The assumption is that olive oil has been used in production of wall paint in Roman times. Recent research has shown that olive oil is distinctive to other edible oils for its fluorescence after excitation at 355 nm [Taotao2013], which might be due to the presence of chlorophyll.

In this work, we propose complex excitation-fluorescence scheme of chlorophyll present in the paint on the surface of the sample. PS I absorb 355 nm and emit at ~685 nm; PSI-LHCII supercomplex [Galka2012] as well as PSI-LHCI A1-A5 complexes absorb 650-690 nm and emit at 708-715 nm [Drop2011, Fujita2004].

Peak at ~980 nm could be also explained by Chl b phosphorescence at 973 nm [Krasnovsky2014].

## 5 CONCLUSION

In this work, the implementation of laser induced fluorescence to the detection of painted artifacts of cultural heritage has been described. Samples of late antiquity fresco fragments have been irradiated with UV laser beam and fluorescence monitored. By the presence of the peaks in the fluorescence spectra, it could be concluded that there might be the presence of the chlorophyll in the paint. Some mechanisms of chlorophyll reaction have been proposed. The results point to interesting direction of research and it would be valuable to continue the investigation with greater number of samples.

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## CONTENTS

1. The Project for Laboratory Scale Tunnel Dryer for Convective-Conductive Drying of Food Materials Ivan Zlatanovic, Vasileios Firinis, Mihailo Milanovic, Biljana Boškovic, Nedžad Rudonja.....	2
2. Diagnostics of the State of Trees Using Infrared Thermography Dragan Milenkovic, Sladana Alagic, Ilija Radovanovic, Misa Stevic, Elena Ponomaryova, Zoran Stevic.....	9
3. Design of the Impulse Laser Power Measurer Ilija Radovanovic, Misa Stevic, Elena Ponomaryova, Dragan Milenkovic, Zoran Stevic.....	15
4. Application of manipulation techniques in brand impression Dorotea Markasovic, Matea Podgornjak, Terezija Berlancic, Ivona Juros, Monika Trbojevic.....	19
5. Application of noise barriers with rubber concrete on certain sections of the Southern bypass of Osijek Stjepan Fotak, Aleksandra Markovic, Dragana Gavric, Karla Popic, Dorotea Markasovic, Miroslav Simun.....	29
6. Overhead contact line influence on local railway telecommunication cables Stefan Mihajlovic, Sanja Jevtic.....	38
7. An Improved Particle Swarm Optimization Algorithm for Scheduling of Single Mobile Robot Aleksandar Jokic, Milica Petrovic, Zoran Miljkovic.....	46
8. Organization of private forestry sector in Finland Danilo Krgovic, Jelena Nedeljkovic.....	56
9. Application of IoT in smart household control Nenad M. Pantelic, Jovan D. Petrovic, Nikola N. Babic.....	71
10. HSR AND MARKETING Nikola D. Milutinovic, Dragana Macura.....	78
11. The Effect Of Honey Bee On Pollination Of <i>Chester Thornless</i> Blackberry Variety Aleksandar Arsic, Nebojsa Nedic, Dragan Radivojevic.....	87
12. Influence of the Object's Emissivity on the Accuracy of Infrared Temperature Measurement Nevena M. Petrov, Nedžad R. Rudonja, Milan D. Gojak.....	92
13. Measuring customer satisfaction with railway service by using a multicriteria approach Selena Antic, Dragana Macura.....	102
14. Laser Cleaning of the Paper From the 19th and Early 20th Century Boris Rajcic, Sanja Petronic, Zoran Stevic, Branislava Savic, Elisaveta Doncheva.....	112
15. Research and Development Cartel Luka Jovanovic, Kristina Micoski.....	120



16. Continuity of architectural heritage through transformation Aleksandra Mladenovic.....	125
17. Continuous variable transmission on vehicles - effects of application Vasiljevic Sasa, Nadica Stojanovic, Ivan Grujic, Jasna Glisovic.....	132
18. The Application of Safety Gate in the Safety Automation with Theoretical Grounds and Practical Examples Ivan Milankovic, Milovan Janicijevic.....	141
19. Metrological Characterisation of Optical Neutral Density Filters Used in Spectrophotometry Danilo Zarubica, Mihailo Zarubica, Veljko Zarubica.....	145
20. Analysis of Guidelines 'A' Directive 2014/68 / EU Related to Overlapping of PED and TPED Directive Marija Jecmenica Ducic, Sanja Petronic, Aurel -Valentin Birdeanu, Katarina Colic.....	153
21. Static Analysis of Airplane Wing Made of Composite Materials Renata Grigorova, Nevena Langura, Mina Radojevic, Nikola Sovilj, Bojana Skaric.....	160
22. Modeling of Honeycomb core composite plates at micromechanics scale Srdjan Kostic, Mirko Dinulovic.....	172
23. Development of The Industry and the Importance of the Transmitting Systems Ljubinko Janjusevic, Suzana Polic.....	180
24. Visualization of the Medieval Church of Monastery Savinac Dušan Petković, Dijana Stojanović, Boris Kosić, Marko Radosavljević, Zoran Stević, Magdalena Dragović.....	187
25. The market effects of youth's alimentary changes in European Union Nastasija Bojanic.....	195
26. Understanding Milutin Bojic's Work as a Compound of Three Scientific Fields: Heritology, Science Of Literature And History Jovana Ruzicic, Suzana Polic.....	196
27. Digitalization and Multidisciplinary Access to History: Slobodan Jovanovic and Leposava Bela Pavlovic Katarina Milkic, Suzana Polic.....	197
28. Evolution of plasma channel in triggered lightning discharges Dragan M. Pavlovic, Jovan M. Cvetic.....	198
29. Sokol Movement in Modern Serbia Dragan Pavlovic, Milan Ignjatovic.....	199
30. Diferential forms Matija Sreckovic.....	200
31. Using of Software Packages For Calculation of Electromagnetic Effect of Energy Transmission Line to Telecommunication Line Tomislav Rajić.....	201



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## САДРЖАЈ

1. Предавање по позиву Сања Петронић, Методологија испитивања чишћења папира из XIX века применом ласерског снопа.....	8
2. Љиљана Никшић, Методолошки прилаз у стварању концепта међународне мултидисциплинарне научне изложбе „Јасеновац – право на незаборав“, УН 2018.....	10
3. Зоран Карастојковић, Павле Карастојковић, Сузана Полић, Материјали за израду иконостаса.....	12
4. Владимир Симић, Методологија изградње звучног архива као ресурса херитолошких вредности.....	14
5. Милеса Срећковић, Сања Јевтић, Драган Јевтић, Слађана Пантелић, Оптички каблови и комуникације у железници 20. и 21. века.....	15
6. Светлана Пелемиш, Владан Мирјанић, Срђан Вуковић, Nanotechnology and biomaterials in dentistry.....	17
7. Анђелка Милосављевић, Димитрије Маљевић, Методолошки приступ испитивањима обрада аустенитних материјала неконвенционалним технологијама.....	18
8. Милош Ђурић, Exercises in Bad Style: The Case of Transgressing Quantum Gravity Hermeneutics.....	19
9. Братислав Иричанин, Методолошки путеви транспоновања у делима Софије Ковалејевске и Данила Киша: једна могућа перспектива.....	21
10. Лука Јовановић, Кристина Мицошки, Херитолошки метод и економска историја: два догађаја из Краљевине Србије и Краљевине СХС.....	22
11. Вељко Зарубица, Катарина Зарубица, Савремени методолошки прилаз оцени мерне несигурности у разним методама мерења са конкретним примерима од значаја за културну баштину.....	23
12. Александра Стевановић, Документарни филм „Три логоса“ као методолошки оглед у разматрању речника технологије.....	25
13. Станко Остојић, Најзаступљеније математичке методе у постављању модела од интереса за културну баштину.....	27
14. Жељка Томић, Методолошко-предметни наслов, Хартлијева класификација и индустријско наслеђе.....	29
15. Анастасија Вигњевић, Екстензија, промишљање и артикулација херитолошких проблема.....	30
16. Матија Срећковић, Методологија у прихватању фундаменталних /егзактних метода и статистички прилаз као основа моделовања од значаја за бионауке и економију.....	31
17. Милена Давидовић, Квазирасподеле од интереса у дигиталној обради података.....	33
18. Сања Јевтић, Драган Јевтић, Методологија у индустријском наслеђу везаном за железницу.....	34
19. Сузана Полић, Универзалност херменеутичког феномена и херитологија.....	36
20. Ведран Ибрахимовић, Методологија примене микроталасних мерења и паралела са оптичким подручјем.....	37
21. Братислав Иричанин, La Place и Saint Simone: методолошке опсервације, о каузалности и метонимији.....	38

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22. Зоран Стевић, Сања Петронић, Драган Миленковић, Наташа Томић, Методологија дијагностике и заштите културног наслеђа применом нових технологија.....	39
23. Магдалена Драговић, Светлана Чичевић, Александар Чучаковић, Александар Трифуновић, Алина Дута, Нови приступи и примена савремених технологија у едукацији инжењера: практични примери из херитологије.....	40
24. Александер Ковачевић, Препознавање облика неуронском мрежом у области заштите културног наслеђа.....	42
25. Светлана Рогановић, Методологија компаративних истраживања естетика Биомбо стила и дадаистичког израза Sophie Henriette Gertrude Taeuber-Arp.....	44
26. Милеса Срећковић, Станко Остојић, Лазар Кричак, Слободан Бојанић, Електрооптички, електромагнетни, акустооптички, пиезоелектрични и други линеарни и нелинеарни процеси у дијагностицирању материјала или у системима мерења.....	45
27. Анђелка Милосављевић, Микроструктурне промене ласером заварене суперлегура никла М-252.....	47
28. Софија Бекрић, Драгољуб Бекрић, Наслеђе Великог рата и аквизиције у Завичајном музеју Жупе – Александровац.....	48
29. Драган Миленковић, Дијагностика стања и заштита културног наслеђа применом инфрацрвене термографије.....	49
30. Магдалена Драговић, Зоран Стевић, Борис Косић, Александар Чучаковић, Марко Радосављевић, Методе обраде дигиталног модела објекта културне баштине за сврхе реконструкције.....	50
31. Сања Петронић, Зоран Стевић, Алекса Маљевић, Важност примене ласера у чишћењу и одржавању херитолошки значајних тканина од памука, тексаса и свиле.....	52
32. Горан Трбојевић, Бард критичке педагогије и методологија академског индустријског комплекса.....	53
33. Никола Славковић, Перцепција асинхроног мотора и приказ Николе Тесле у делу Леса Драјсдејла.....	54
34. Зоран Фидановски, Бојана Фидановски, Биљана Фидановски, Софтвер од интереса за културну баштину и другу савремену проблематику.....	55
35. Драган Павловић, Јован Цветић, Утицај руских архитеката на изградњу Београда.....	57
36. Љубинко Јањушевић, Александар Седмак, Михаило Петровић и методологија изабраних проналазака у индустријској и научној баштини Србије.....	58
37. Томислав Рајић, Савремени software и методологија његове примене у енергетици мерењима.....	59
38. Ђорђе Ђорђевић, Ивана Ћировић, Гордана Ђукановић Основи заснивања методологије дефинисања идентификационе карте уметничког дела - параметризовањем његових карактеристичних геометријских-морфогених својстава, применом поступака микрофотограметрије и математичке статистике/вероватноће.....	61
39. Бранислав Давидовић, Моделовање у простору ласерске технике у циљу модела за специфичне примене.....	65
40. Зоран Лазаревић, Милош Јечменица, Богдан Брковић, Савремени проблеми у пројектовању и даљинском управљању енергетски ефикасне микромреже на подручју Манастира Хиландар.....	67
41. Милеса Срећковић, Станко Остојић, Милена Давидовић, Функције корелације, метода момената и кумуланата, дефиниција и практичне примене у савременим	

---

статистичким опсервацијама .....	69
42. Анђелка Милосављевић, Утицај легирајућих елемената током ласерске обраде високотемпературних материјала .....	71
43. Вишеслава Рајковић, Студије случаја кроз призму науке о материјалима .....	72
44. Катарина Чолић, Филип Вучетић, Примена савремених оптичких метода мерења у биомедицини .....	73
45. Силвана Б. Димитријевић, Стеван П. Димитријевић, Зоран Стевић, Заштита од тамњења металних предмета културне баштине.....	74
46. Сузана Полић, Интегритет и безбедност културног наслеђа у светлу новијих методолошких и технолошких истраживања .....	75
47. Сања Петронић, Борис Рајчић, Димитрије Маљевић, Интеракција ласера са бакром и његовим легурама са аспекта херитологије.....	76
48. Катарина Чолић, Побољшање механичких карактеристика биоматеријала применом савремених ласерских метода.....	77
49. Марија Крстева, Превођење техничких терминолошких конструкција са енглеског на македонски језик.....	78
50. Предраг Јованић, Прелиминарна грађа и прилог за рестаурацију и конзервацију манастира Раковица.....	79



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**ТЕХНИЧКА ОБРАДА**

Саша Костић

**ПРОГРАМ И САДРЖАЈ ЗБОРНИКА**  
 Друге националне конференције  
**Методолошка истраживања у херитологији и новим технологијама**  
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**ПРЕДАВАЊА ПО ПОЗИВУ**

1.	Сања Петровић, Зоран Стевић	Методологија усвајања знања о ласерском чишћењу папира на основу изабраних руских научних извора	1-3
2.	Саша Милић	5G мрежа у светлу културне баштине нове индустријске револуције и интернета ствари	4-7

**СЕКЦИЈА 1**

3.	Зоран Здравковић, Јулијана Мирчевски	Дигитализација и конзервација културне баштине и историјског наслеђа	8-11
4.	Томислав Рајић	Примена софтверског алата XGSLab за процену нејонизујућег зрачења у близини далековода	12-14
5.	Милеса Срећковић, Александер Ковачевић	Ретроспектива методолошких приступа примене ласерских техника у херитологији: изабране студије	15-17
6.	Анђелка Милосављевић	Материја у методолошком приступу Анђија Bergsona – опсервација са становишта технологије материјала	18
7.	Станко Остојић	Методологија у постављању модела за припрему конзервације/рестаурације објеката индустријске и нематеријалне културне баштине	19-20
8.	Светлана Пелемиш	Нанотехнолошки материјали у културној баштини	21

**СЕКЦИЈА 2**

9.	Жељка Томић	Баштина и феномен светлости у делу Bele Hamvaša „Невидљиво збивање“: методологија истраживања	22-23
10.	Милош Ђурић	Констрактивна анализа енглеско-француских термина регистра конзервације и херитологије	24-25
11.	Анастасија Вигњевећ, Антон Ето	Изоловање нејасних значења по методи Rolana Barta и рефлексије	26-28
12.	Драган Павловић, Милеса Срећковић	Историја астрономске опсерваторије у Београду	29-31
13.	Слободан Бојанић	Хеуристика и научни рад на пољу техничких наука	32-33

**СЕКЦИЈА 3**

14.	Никола Поповић, Јулијана Мирчевски	Реконструкција и конзервација старих и избледелих докумената у државној управи	34-37
15.	Милош Ђурић	Метафразе у преводима текстова из области техничко-технолошких наука: методолошки изазови	38-39
16.	Светлана Рогановић	Интеракција етике, естетике и политике, као методолошки концепт у интердисциплинарности Grizelde Polok	40-41
17.	Горан Трбојевић	Методолошки поглед на употребу антитезе у истраживањима Stivena Grinblata о ренесансној култури	42-43

18.	Братислав Ирчанин	Филозофија у технолошком добу, дело Hansa Lenka, педесет година касније	44-46
-----	-------------------	-------------------------------------------------------------------------	-------

#### СЕКЦИЈА 4

19.	Љиљана Никшић	Методолошка искуства истраживања односа Србије и САД у контексту културе сећања	47-50
20.	Жељка Томић	Изоморфизам у контексту примене електротехнике у заштити културног наслеђа: методологија по Фукуоу	51
21.	Катарина Насрадин, Сузана Полић	Мултидисциплинарност у истраживању старих култура у стваралаштву Nagalda Natmana	52-54
22.	Бранка Радојчић, Слађана Пантелић	Значај система противпожарне заштите за очување објеката културне баштине – пример пожара у Нотр Даму	55-56
23.	Анђелка Милосављевић, Сања Петронић	Спецификум истраживања микроструктурних и микроморфолошких промена за израду протокола заштите индустријског наслеђа	57

#### СЕКЦИЈА 5

24.	Никола Славковић	Предикције примена 5G стандарда у креирању модела проширене реалности у заштити културног наслеђа	58-59
25.	Слађана Пантелић, Бранка Радојчић	Примена оптичких сензора у испитивању и обезбеђивању уметничких предмета и музеја	60-61
26.	Вељко Зарубица	Методологија за развој протокола примене мерних техника у заштити наслеђа	62-67
27.	Милош Павловић	Новија методолошка решења у примени нуклеарних техника у заштити културног наслеђа	68
28.	Љубинко Јањушевић, Александар Седмак, Сузана Полић	Методолошки приступи у херитолошкој валоризацији старих патената	69

#### СЕКЦИЈА 6

29.	Зоран Стевић, Сања Петронић, Миша Стевић	Конструкција апаратуре за дијагностику објеката културног наслеђа RTI методом	70-71
30.	Милеса Срејковић, Зоран Недић, Александер Ковачевић, Зоран Карастојковић, Милица Винић, Миливоје Ивковић	Проблеми интерпретације и идентификације примене разних дијагностичких техника у културној баштини	72-73
31.	Зоран Карастојковић, Радиша Периф, Павле Карастојковић, Сузана Полић	Методологија и дизајн у производњи златних православних крстова	74-80
32.	Сања Јевтић, Ана Илић, Драган Јевтић, Милеса Срејковић	Методологија изградње специфичних железничких система и мрежа	81-82
33.	Милена Давидовић, Милош Давидовић	Утицај квалитета ваздуха на очување културног наслеђа	83-85

## СЕКЦИЈА 7

34.	Братислав Ирчанин	Улога математике у концептуално-критичким методолошким приступима <i>Simona Mozega</i>	86-88
35.	Никола Славковић	Методичке оријентације и рефлексije научних истраживања у електротехници, погодне за креирање наставних активности у херитологији	89
36.	Љиљана Никшић	Методологија истраживања за изложбу о доприносу Пупина и Вилсона у мобилизацији и Версајском миру	90
37.	Сузана Полић	Методолошке основе у истраживању визуелних представа феномена <i>sensus communis</i>	91-93
38.	Милован Јанићијевић	Коherentни снопови и критични феномени у материјалима са фракталном микроструктуром	94-95
39.	Лука Јовановић	Менаџмент и економија херитологије	96
40.	Бранимир Давидовић	Одржавање трасе 6 kv каблова у површинским рудницима у Србији	97-99
41.	Зоран Латиновић	Улога вештачке интелигенције у менаџменту	100
42.	Нада Ратковић Ковачевић, Зоран Стевић, Драган Шаиновић	Примена термалних - инфрацрвених сензора у херитологији	101-107

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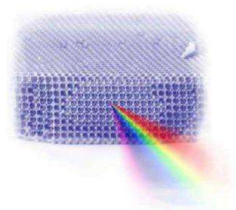
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Kopaonik, March 14-17, 2021



Book of Abstracts  
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## **14<sup>th</sup> Photonics Workshop (2021)**

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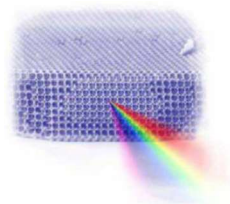
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Kopaonik, March 13-16, 2022



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(Conference)



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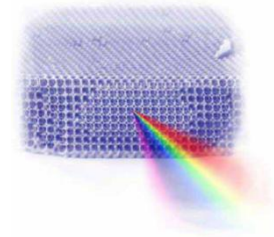
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Kopaonik, March 12-15, 2023



Book of Abstracts  
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(Conference)



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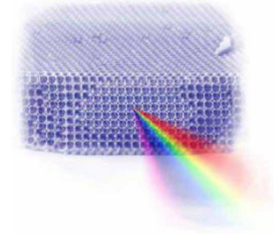
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University of Belgrade  
Institute of Physics Belgrade  
Kopaonik, March 10-14, 2024



Book of Abstracts  
**17<sup>th</sup> Photonics Workshop**  
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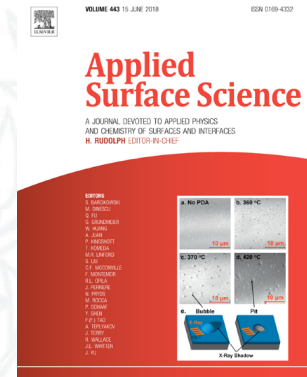
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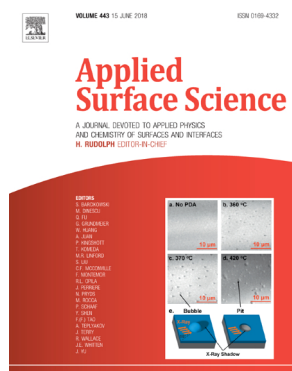
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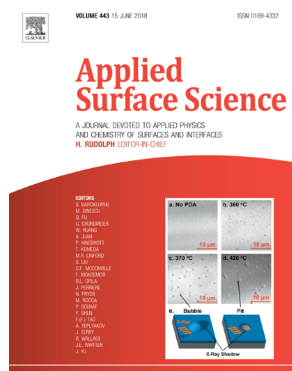
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Composite Structures

1



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To: 31 December 2019

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## Composite Structures

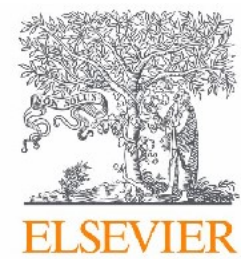
1 reviews completed

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Manuscript title	Revision	Date completed
Analytical analysis on temperature of CFRP laminates subjected to CW laser irradiation	0	15 October 2019

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Applied Surface Science

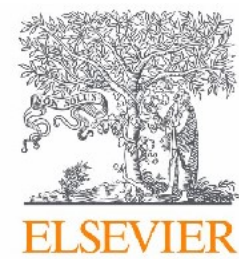
2

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Optik

1



# Review History Report

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## Applied Surface Science

2 reviews completed

Manuscript title	Revision	Date completed
Defects rich nanostructured black zinc oxide formed by nanosecond pulsed laser irradiation in liquid	1	26 July 2021
Defects rich nanostructured black zinc oxide formed by nanosecond pulsed laser irradiation in liquid	0	3 May 2021



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## Optik

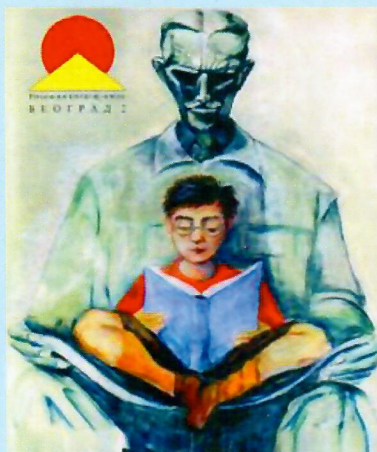
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Manuscript title	Revision	Date completed
Mechanic Properties Modification of SiO <sub>2</sub> Thin Films by Femtosecond Laser	0	11 October 2021

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АКТИВНОСТИ РЕГИОНАЛНОГ ЦЕНТРА ЗА ТАЛЕНТЕ  
БЕОГРАД 2, И РАДУ СА НАДАРЕНОМ И ТАЛЕНТОВАНОМ  
ШКОЛСКОМ ПОПУЛАЦИЈОМ,**

**ЦИКЛУС РАДА 2017/2018**

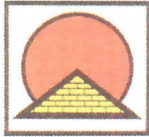
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ТЕКУЋИ-РАЧУН 840-2014660-24

МБ 17242024

## ПОТВРДА

Којом се потврђује да је др АЛЕКСАНДЕР КОВАЧЕВИЋ, као члан комисије из области ФИЗИКА учествовао у реализацији СМОТРЕ ИСТРАЖИВАЧКИХ РАДОВА УЧЕНИКА СРЕДЊИХ ШКОЛА.

Смотра истраживачких радова ученика средњих школа, организује се под покровитељством Министарства, просвете, науке и технолошког развоја и налази се у Календару такмичења.

Организатор Смотре је Регионални центар за таленте Београд 2, а домаћин смотре 2019. године је Образовни систем „Руђер Бошковић“.



У Београду, 30. Март 2019.

РЕГИОНАЛНИ ЦЕНТАР ЗА ТАЛЕНТЕ БЕОГРАД 2

Никола Срзентић

директор



Contact:

tel/fax 011 243-13-13,

mob. 065/243-13-13

064/4233-734

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[www.centarzatalente.edu.rs](http://www.centarzatalente.edu.rs)

Комисија за именовање добитника награде младом истраживачу Центра за фотонику у области фотонике (у даљем тексту: Комисија) у саставу др Александер Ковачевић, др Душан Арсеновић и др Марина Лекић доноси

### ПРЕДЛОГ

да се **Годишња награда младом истраживачу Центра за фотонику за 2019. годину** у области фотоника додели

### др Даница Павловић

Комисија је добила материјал за више кандидата. На основу увида у поднети материјал и на основу квантитативне и квалитативне процене научног доприноса кандидата, а узимајући у обзир број и квалитет објављених радова и њихов утицај на научну област, односно на проблематику којој припадају, стваралачки удео у оствареним резултатима и степен реализације у Институту, Комисија је утврдила да је др Даница Павловић испунила све услове за стицање награде, да је у свом раду исказала широки спектар активности, дала значајан допринос у својој области истраживања и показала велику самосталност уз високи степен реализације истраживања у Институту за физику.

#### Кратка биографија предложеног кандидата

Даница Павловић је рођена 1990, на Биолошком факултету Универзитета у Београду дипломирала је 2013, одбранила мастер 2014, а докторирала 2019. Од 2015. је запослена у Институту за физику Универзитета у Београду. Учествује у реализацији два међународна билатерална пројекта, са Савезном Републиком Немачком и Народном Републиком Кином. До сада је објавила шест радова категорије M20 и коаутор је поглавља у истакнутој монографији националног значаја. На три међународне патентне пријаве је коаутор, објавила је рад у врхунском националном часопису и саопштила је девет радова на међународним, а три рада на националним скуповима.

Награда у износу RSD40000,00 нето се додељује из средстава Центра за фотонику.

Др Александер Ковачевић

Др Душан Арсеновић

за Др Марина Лекић



УНИВЕРЗИТЕТ У БЕОГРАДУ

ИНСТИТУТ ЗА ФИЗИКУ			
ПРИМЉЕНО:		02. 04. 2021	
Рад.јед.	б р о ј	Арх.шифра	Прилог
0801	266/1		

Адреса: Студентски трг 1, 11000 Београд, Република Србија  
Тел.: 011 3207400; Факс: 011 2638818; E-mail: kabinet@rect.bg.ac.rs

Београд, 22. децембар 2020. године  
02: 612-45/344-20  
ЛД

На основу члана 81 Закона о високом образовању („Службени гласник РС“, број 88/17, 73/18 и 67/19) и одредби Правилника о условима и начину учешћа научноистраживачких установа које су у саставу Универзитета у Београду и лица изабраних у научно звање у остваривању дела наставе („Гласник Универзитета у Београду“, бр. 132/06)

Универзитет у Београду, Београд, Студентски трг 1, кога заступа проф. др Гордана Илић-Попов, проректорка (у даљем тексту: Универзитет), са једне стране и

др Александер Ковачевић, виши научни сарадник Универзитета у Београду – Институт за физику, запослен са пуним радним временом, са адресом у Београду, Видиковачки венац 29, општина Раковица, ЈМБГ 3101963714035 (у даљем тексту: виши научни сарадник), са друге стране закључују

## У Г О В О Р О АНГАЖОВАЊУ ЗА ИЗВОЂЕЊЕ НАСТАВЕ -ДОПУНСКИ РАД-

### Члан 1

Предмет овог уговора је ангажовање др Александра Ковачевића, вишег научног сарадника Универзитета у Београду – Институт за физику, на извођењу наставе на студијском програму: „Биофотоника“, у школској 2020/21. години.

### Члан 2

Одлуком Већа за студије при Универзитету у Београду виши научни сарадник из члана 1 овог уговора је ангажован за извођење наставе, испита и других студијских активности у складу са Законом, Статутом и другим општим актима Универзитета у Београду.

### Члан 3

Наставу из члана 2 овог уговора виши научни сарадник је обавезан да изводи по утврђеном распореду у просторијама Универзитета, у складу са општим актима Универзитета.

УНИВЕРЗИТЕТ АД БЕОГРАДА			
ПР.М.В.НО.:		ОН.В.Д.М.Н.П.	
Датум:	Лист:	Број:	Датум:

#### Члан 4

За обављене послове из члана 2 овог уговора виши научни сарадник подноси извештај руководицу студијског програма.

На основу извештаја из става 1 овог члана руководилац студијског програма, уз извештај, подноси захтев за исплату накнаде надлежном проректору, на сагласност.

Исплата новчане накнаде из става 2 овог члана извршиће се из средстава студијског програма „Биофотоника“, преко текућег рачуна вишег научног сарадника.

#### Члан 5

Уговорне стране су сагласне да ће евентуалне спорове из овог уговора решавати договором, а ако договор не успе уговара се надлежност суда у Београду.

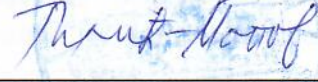
#### Члан 6

Овај уговор је сачињен у 3 (три) истоветна примерка од којих 2 (два) примерка задржава Универзитет, а 1 (један) примерак виши научни сарадник.

ВИШИ НАУЧНИ САРАДНИК

  
др Александер Ковачевић

ПРОРЕКТОРКА УНИВЕРЗИТЕТА

  
проф. др Гордана Илић-Попов

