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Scientific Report

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Project Title:
The study of polymer-laser radiation interactions in controlled atmosphere. Laser ablation nanostructured thin films layers. Applications/Studiul interactiunii polimer-radiatie laser in atmosfera controlata. Obtinerea de filme nanostructurate prin ablatie laser. Aplicatii

Context

The developing of nanotechnologies applied to nano-sensors and nano-composites are based on knowledge about the physical and chemical processes at nanometric scale. The modern materials are distinctive characterized by taking into account the quantic effects that mainly differ by initial structure. In this context was evidenced that a quota of organic polymer-based substrates offering a number of advantages over inorganic ones: are more flexible, offer a variable rigidity and can take different forms following the action of external factors. In addition, the polymeric material properties can be accustomed according to specific requirements by chemical modification or varying the polymerization conditions. Micro and nano-structures obtained using a polymeric support can be extremely useful to a variety of applications as well as conductors or circuit substrates obtained for adhesion control. Whatever the nature of their origin, the polymeric nanoparticles are found a stable structure, in contrast to other systems.

The nanostructuring phenomena intensely discussed in the international specialty literature, assess to obtain a capable material to generate a controllable surface structure. The current problems relate to the fact that a direct connection between chemical structure and nanostructuring mechanism of the used polymer does not exist. In this context, the literature talks about developing a number of theoretical models and tools, based on two types of phenomena which try to explain the reordering processes of azo-materials surfaces. The first involves a re-organization of the polymer material and its compression under the UV radiation action and the second, assumes the displacement of the polymeric material (photo induced flow). Our studies are mainly focused on the chemical structure of the polymer and especially to influences over mechanism types and nanostructuring process. To “Alexandru Ioan Cuza” University of Iasi, Faculty of Physics, Atmosphere Optics, Spectroscopy and Lasers Laboratory (LOA-SL) Bd-ul Carol I nr 11, Iasi 700506, Romania Tel: +40232201197; Fax:

+40232201150 Email: sgurlui@uaic.ro; Web: spectroscopy.phys.uaic.ro elucidate these issues, it have been studied in a first step, the expertise literature, regarding certain classes of polymers with different architectures, with based chain flexible and semi-flexible or rigid side and with lateral chain connected to different types of chromophores.

Among the processes that have drawn attention in this multidisciplinary field of nanotechnology, molecular and supramolecular ordering is considered of perspective, because at the molecular level, chromophores such as azobenzene groups are known for photo induced arrangement, after reversible reaction of trans-cis-trans isomerization. In this case it results an almost perpendicular distribution of the transition dipole moments to the direction of action of the electric field intensity vector belonging to optical radiation linearly polarized.

Research activities

The project objectives were achieved by following specific research activities:

A. Study of polysiloxani type polymers, polymers having diazobenzen group perpendicular to the polymer chain:
   - We obtained polymer thin films on quartz glass support and special glass transparent in the UV up to 260 nm, using spin coating deposition method.
   - FTIR-ATR was studied for 80-120nm thick polymer layers
   - We studied the effects of photochromic optical radiation in thin polymer layers and in solutions of dimethyl formamide.
   - We mentioned the dynamic processes of trans- and cis photo isomers.

B. Synthesis and characterization of nanostructures (polymers, metals, ferromagnetic materials, chalcogenides) obtained by PLD:
   - The synthesis of nanostructures by metallic nanoparticle implementation in polymer matrix..
   - We obtained thin films of stoichiometric cobalt ferrite un-doped and doped with rare earth elements (PR) by laser ablation using different values of various experimental parameters.
   - We analyzed a number of highly applicative chalcogenides (with important applications in many branches of science: biology, medicine, chemistry, physics, etc.), noted in particular by their low cost and miniaturizing trend of the industry for ultra-fast memories, amplifiers and laser sources, optical sensors etc.

C. The influence of laser pulse characteristics and other experimental parameters on the laser induced plume dynamics:
   - We used spectral techniques, ultrafast ICCD imaging for the characterization and space-time resolving of the laser ablation plume and also electrical diagnosis methods (mobie cilindrical probes). These techniques were applied to laser induced plasmas obtained in different experimental conditions: fluence, pulse length, repetition rate, gas pressure etc.
   - We investigated the electrical oscillations appearing in the electronic circuits of some electrodes (targets, walls, probes, etc.) placed near the laser ablation plume;
   - The influence of the different materials (target type) on the dynamics of the laser ablation plume was investigated. The results were compared with those obtained in the study of stationary plasmas, related to the inhomogeneous plasma configurations with nonmonotonous potential structure
   - We also performed studies on the simulating operation of Hall effect engines (PPS100-ML, Hall Effect Thruster) propulsion used at geostationary satellite orbit correction (see Pivoine-2G, ICARE laboratory, CNRS Orleans, France).

The completed measurements, of great importance not only in terms of basic research but also applicative point of view, considered finding various experimental conditions to identify critical values to obtain competitive results. The approach and development of nanotechnologies of great impact on the development of leading applications aimed at obtaining nano-components and nano-sensors involves the study of new materials governed by quantum effects and physical properties that differ fundamentally from those of the structures of bulk material.

Main research results

A. Study of polysiloxani type polymers, polymers having diazobenzen group perpendicular to the polymer chain

The electronic spectra, absorption and fluorescence of polyurethane coumarin were obtained in various polar solvents and film state. Electronic absorption bands in polar solvents suffer shifts towards higher wavelengths. Electronic absorption spectrum of polyurethane-coumarin has two bands, and they π-π transitions assigned. Electronic spectra of the fluorescence of polyurethane coumarin were obtained in dimethyl-formamide (DMF), tetrahydrofuran (THF) and in the film state. In order to obtain fluorescence spectra were used the following sources of radiation: 310nm Hg; 365nm Hg; N2 laser radiation (337nm) and Nd-YAG laser radiation (Quantel) 266nm, 532nm respectively. It has been found that shifts of the fluorescence maxima in polar solvents bands are greater than the displacement of the absorption bands peaks in the same solvent. This shows that the electric dipole moment in the excited state (μ_e) is higher than the ground state dipole moment (μ_g) ($\mu_e > \mu_g$). These shifts to higher wavelength peak of electronic bands ($\Delta\nu$ = 3000-5000 cm^{-1}) indicated that the state of the film and solutions of DMF and THF are nano-aggregates of the polyurethane-coumarin. To get more information about these nanostructures were studied photo-physical properties and photo-chemical properties of polyurethane-coumarin excitation under the action of molecular systems using λ_{exc} = 266nm respectively λ_{exc} = 532nm. Studies of absorption and fluorescence spectra showed that, under the action of radiation with λ> 310 nm, polyurethane- coumarin forming photodimers. By comparing the fluorescence intensity of the bands at 375nm and 375 nm in a state film and DMF showed that photodimerization of polyurethane coumarine film state is produced, in particular its surface. To confirm this result we studied the photodimerization of polyurethane coumarin in film state using AFM methods. We studied also the surface of a film obtained by spin-coating in two cases. Surface area non-irradiated and irradiated with the nitrogen laser pulses (λ = 337 nm). Images deflection confirms that nanoagregates formats appear in greater numbers when the film surface is irradiated with nitrogen laser radiation with, λ = 337nm. We highlighted some preliminary results on action pulsed laser beams (2 picoseconds) on the same polyurethane-coumarin-type polymers. Spectral measurements were performed and ICCD imaging of laser ablation plume and PLD thin layers of different thicknesses.

B. Synthesis and characterization of nanostructures (polymers, metals, ferromagnetic materials, chalcogenides) obtained by PLD

metallic nanoparticles (Cu, 99.4% purity and Ni, 99.6 % purity) were implemented by ablation of polyimide substrates. The studies that refer to surface structuring of polymer thin films showed that the irradiation conditions have a significant influence (frequencies range and energy values). Implementation of metallic nanoparticles in polymer films must be made by taking into account several factors which include: pptic and electric properties of the obtained nanostructures, the thickness control of the deposited films, detailed research of the

shape of the implemented nanoparticles, study of the diffusion processes that lead to changes in particle size and shape distribution, optic and electric properties measurements of the obtained matrices (polymer - metal nanoparticles). We obtained polymer thin films on quartz substrate and special glass with transparency in the UV and IR (CaF) region using spin coating deposition method. Synthesized polymers Polyimide (six polymers) and Polyurethane coumarin. For this study, polyimide polymer films were synthesized at Institute of Macromolecular Chemistry " Petru Poni " of Iasi. Structural and chemical analysis techniques confirmed the chemical structure of the specified compounds. The structural analysis of doped cobalt ferrite films revealed that a higher substrate temperature is required to obtain spinel structure. After analyzing the results of Raman spectroscopy we observed the presence of compression stress, with the use of higher temperatures during deposition. The presence of RE in cobalt ferrite thin films did not cause the formation of residual phases of RE orthoferrite as in bulk materials. Moreover, structural and magnetic results confirmed the substitution of Fe from the spinel structure by RE ions. It was noted that the use of higher substrate temperatures resulted in improved magnetic response. To avoid micrometer-sized particle deposition on the substrate, but not significantly diminish the deposition rate, for material removal we used a radiation from a fs Ti-Sapphire laser. The results of structural analysis performed showed the formation of a spinel crystal structure with preferential crystallographic growth direction. Due to much lower fluency and repetition rate we obtained thin films with high uniformity in a much shorter time. Estimated deposition rate was higher by about one order of magnitude than that observed for Nd-YAG laser deposition. In the case of these layers we detected internal mechanical stresses but of expansion.

C. The influence of laser pulse characteristics and other experimental parameters on the laser induced plume dynamics:

For a special class of chalcogenides (Pure and rare-earth doped (Erbium or Praseodymium) gallium lanthanum sulphide (GLS)) we highlighted the importance of laser regime (laser pulse duration) and laser fluency on the dynamics of laser induced plume (densities, excitation temperatures, expansion speed etc.) and on the plume geometry. Different laser regimens were used as follows: In the nanosecond regime we used an Nd-YAG laser with a wavelength of 532 nm, pulse duration of 10 ns, repetition rate 10 Hz and fluence of 4 J / cm^2. In picosecond regime we used a TiSa laser with a wavelength of 800 nm, pulse duration 2 ps, repetition rate of 100 Hz and fluence of 1 J/cm^2. For femtosecond laser irradiation the laser pulse duration was 120 fs with the other characteristics similar to picosecond regime. Preliminary results showed significant changes from one regime to another (reflected by ICCD imaging measurements as well as by spatial and temporal resolved optical emission spectroscopy measurements. For an individual characterization of the chemical species present in the plasma, specific spectral measurements were performed. Thus, depending on the laser regime, optical emission spectra of a 0.2 mm thick plasma slices were recorded at different distances from the target surface. From the spatial and temporal profiles, excitation temperatures of different species and temporal evolution of the electron density were studied. Preliminary results showed a laser ablation plume structure in which the chemical composition as well as laser irradiation regime plays an important role. Preliminary measurements also show the existence of the acceleration region of laser ablation plume. This phenomenon has been the subject of some articles and it is assumed that the origin of this acceleration is based on the "plasma double layer" formed by separating electrical charges during expansion. Unlike in the nanosecond regime, in femtosecond regime the plasma presents a completely different dynamics. Doping elements contribute to a substantial increase of time (by an order of magnitude) for which the plume emits in the UV- VIS region. Also, after ~1000 ns the plume, of both dopants, has a tendency to split and the plume

presents a slower expansion. Also, compared with the studies conducted so far, we observed for the first time a filamentary structure beginning with 3000 ns (GaLaSPr3+) or 4000 ns (GaLaSEr3+). Thus the laser ablation plume profile changed substantially during expansion. Thus, if in general, the profile of laser ablation plume behaves after a ⟦cos⟧^n (α) low with n = 8 in the « classic » regime, in the case mentioned above the index has much higher values. The laser ablation plasma gets a filamentary geometry starting from distances of 11 mm from the target and could influence the quality of PLD layer. LOASL together with the group of researchers from the University of Science and Technology of Lille 1 in France (prof. C. Focsa) observed an oscillatory phenomenon in laser ablation plasma. Such phenomenon drew the attention of the international scientific community and has been the subject of several invited papers and ISI publications. To understand the complexity of selforganization and oscillatory phenomena in laser induced plasma, a systematic study based on the nature of the irradiated material, influence of different experimental conditions (working gas, distance of observation, laser fluence, laser treatment etc.) and also mathematical model is required. The first preliminary results were obtained using nanosecond pulsed laser. Our findings reveal that the oscillatory structure is based on elementary processes found in laser ablation plasma (excitation and ionization collisions, re-combinations) and depends on the electric properties of the target and the structure shows different dynamics during its expansion from the target surface to the substrate on which the thin film is formed. Such self-organization mechanisms in laser induced plasma could induce different properties ranging from stoichiometry and optical properties of the layers deposited by PLD. In our opinion, these phenomena are more important when the substrate is closer to the target surface. Our research was also focused on the study of materials with various atomic masses (aluminum, manganese, copper, tungsten, nickel, tellurium etc.) using femtosecond laser.

ISI indexed papers

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2. P. Nica, M. Agop, S. Gurlui, C. Bejinariu, C. Focsa, Characterization of Aluminum Laser Produced Plasma by Target Current Measurements, Jpn. J. Appl. Phys. 51, 106102 (2012).
3. M. Agop, P. Nica, O. Niculescu, D. G. Dimitriu, Experimental and theoretical investigations of the negative differential resistance in a discharge plasma, J. Phys. Soc. Japan 81, 064502 (2012).
4. S. Gurlui, D. G. Dimitriu, N. Cimpoesu, E. Buruiana and M. Strat, Photo-(physical and chemical) properties of polyurethane coumarin in polar solvents and thin films state, Submitted atEuropean Polymer Journal, 2012.
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8. P. Nica, S. Gurlui, M. Osiac, M. Agop, C. Focsa, Electrical Characterization of Femtosecond Laser-Produced Plasma from Various Metallic Targets, in review.

9. S. Gurlui, E. Buriana, Photo-responsive behavior of novel polyurethane coumarins, Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, in review.
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11. D. G. Dimitriu, S.A. Irimiciuc, S. Popescu, M. Agop, C. Ionita and R.W. Schrittwieser, On the interaction between two fireballs in low-temperature plasma, Physics of Plasmas 22, 113511(2015).
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2. M. Agop, E. Poll, D. G. Dimitriu, M. Aflori, L. M. Ivan - Transition to chaos through sub-harmonic bifurcations in plasma. II. Fractal hydrodynamics, *Bulletin of the Polytechnic Institute of Iasi, section Mathematics. Theoretical Mechanics. Physics*, Vol. 58, No. 4, 2012, pp.: 37-55.
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Invited lectures

1. S. Gurlui, C. Focsa, Development of Laser-Produced Plasma Technology. Fundamentals and Applications, 8th BPU, the 8th General Conference of Balkan Physical Union, 5-7 July 2012, Constanta, Romania.
2. O. G. Pompilian, G. Dascalu, S. Gurlui, C. Focsa, Processing and characterization of advanced materials by laser ablation techniques, 9th International Conference on Physics of Advanced Materials, 20 - 23 September 2012, Iasi, Romania.
3. O. G. Pompilian, G. Dascalu, S. Gurlui, C. Focsa, Laser-induced plasma: fundamentals and applications, Physics Conference TIM-12, 27-30 November 2012, Timisoara, Romania.
4. S. Gurlui, P. Nica, M. Agop, M. Osiac, C. Focsa, Peculiar Behavior of Plasma Plumes Generated by Femtosecond Laser Ablation of Metallic Targets, 12th International Conference on Laser Ablation, 6-11 October 2013, Ischia, Italy (O-09).
5. S. Gurlui, P. Nica, M. Agop, M. Osiac, C. Focsa, Two-Temperature Plasmas Generated by Femtosecond Laser Ablation of Metallic Targets, 16th International Conference on Plasma Physics and Applications, June 20-25, 2013, Magurele, Bucharest, Romania.
6. C. Focsa, S. Gurlui, High Technological, Potential Materials, Explored by Laser, Ablation, The 5-th National Conference of Applied Physics, May 23 - 24, 2013, Iasi, Romania.
7. R. Schrittwieser, C. T. Teodorescu-Soare, D. G. Dimitriu, S. Gurlui, C. Ionita, O. Vasilovici, Complex space charge structures excited by a spherical grid cathode, 16th International Balkan Workshop on Applied Physics, July 7-9, 2016, Constanta, Romania.

National and international conference participations

1. R. Cimpoesu, O. Pompilian, N. Cimpoeşu, D. Ghe. Dimitriu, S. Gurlui, and C. Focsa, UV-pulsed laser deposition of polymer thin films: fundaments and applications, Physics Conference TIM – 11, 24 - 27 November 2011, Timisoara, Romania, (poster presention).
2. S. Gurlui, I. Lihtetchi, V. Hurduc, D. Ghe. Dimitriu, and M. Strat, Nanostructured polymer surfaces and nanoagregates of some diazobenzene polymers in film sate, Physics Conference TIM – 11, 24 - 27 November 2011, Timisoara, Romania, (poster presention).
3. L. Balika, C. Focsa, S. Gurlui, S. Pellerin, N. Pellerin, D. Pagnon and M. Dudeck, Laser Ablation in a Hall Effect Thruster for Space Propulsion, E-MRS Spring 2012 - Symposium V, 2012.
4. G. Dascalu, G. Pompilian, B. Chazallon, V. Nica, O. F. Caltun, S. Gurlui, C. Focsa, Rare earth doped cobalt ferrite thin films deposited by PLD, E-MRS Spring 2012 - Symposium V, 2012.
5. S. Gurlui, G. O. Pompilian, P. Nemec, V. Nazabal, M. Ziskind, C. Focsa, Plasma Diagnostics in Pulsed Laser Deposition of GaLaS Chalcogenides, E-MRS Spring 2012 - Symposium V, 2012.
6. R. Cimpoesu, S. Gurlui, O. Pompilian, M. Lohan, N. Cimpoesu, C. Focsa, Thermo-elastic solicitation of a shape memory alloy enhanced with thin polymer films through pulsed laser deposition technique, 8th BPU, The 8th General Conference of Balkan Physical Union, 5-7 July 2012.
7. S. Gurlui, D. Dimitriu, M. Strat, Optical and Spectral Properties of Polyurethane Coumarine. Self-organization phenomena induces nanoagregate formations, 8th BPU, The 8th General Conference of Balkan Physical Union, 5-7 July 2012.
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12. O. Niculescu, M. M. Cazacu, M. N. Dănilă, D. G. Dimitriu, S. Gurlui, M. Agop – Magnetosphere double layers and aurora borealis. Acceleration mechanisms and instabilities, Environmental Legislation, Safety Engineering and Disaster Management (ELSEDIMA), Cluj-Napoca, Romania, 2012.
13. S. Gurlui, O. Niculescu, D. G. Dimitriu, C. Ionita, R. Schrittwieser – Spectral investigations of two simultaneous fireballs in plasma, Physics Conference TIM-12, Timisoara, Romania, 2012.
14. O. Niculescu, S. Gurlui, D. G. Dimitriu, C. Ionita, R. Schrittwieser – Optical Emission Spectroscopy and Nonlinear Dynamics Analysis of Two Fireballs Simultaneously Obtained in a Cold Diffusion Plasma, 31st ICPIG, July 14-19, Granada, Spain, 2013.

15. O. Niculescu, S. Gurlui, D. G. Dimitriu, C. Ionita, R. Schrittwieser – Study on the Interaction of two Complex Plasma Structures. Optical Emission Spectroscopy and Nonlinear Dynamics Analysis, PLASMA-2013, September 2-6, Warsaw, Poland, 2013.

16. G. Dascalu, S. Gurlui, P. Nemec, O. Pompilian, C. Focsa, Nano-, Pico- and Femto-second Laser Ablation of Pure and Rare-earth-doped Gallium Lanthanum Sulphide: A Comparative Study by Space- and Time-resolved Optical Emission Spectroscopy, International Conference on Laser Induced Breakdown Spectroscopy (LIBS 2014), 2014, Beijing, China.

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24. G. Bulai, V. Nica, B. Chazallon, S. Gurlui, C. Focsa, Influence of rare earth addition on structural and magnetic properties of cobalt ferrite thin films, EMRS Spring Meeting, 2015, Lille, France.

25. G. Bulai, B. Chazallon, I. Dumitru, S Gurlui, C. Focsa, Influence of deposition conditions on rare earth doped cobalt ferrite thin films obtained by PLD, Conference on Laser Ablation, 2015, Cairns, Australia.

26. G. Bulai, S. Gurlui, B. Parvatheeswara Rao, Ovidiu Florin Caltun, Alternating target laser ablation deposition of Cu doped cobalt ferrite thin films, International Conference on Magnetism, 2015, Barcelona, Spain.

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31. S.A. Irimiciuc, S. Gurlui, P. Nica, M. Agop, M. Osiac, C. Focsa, Electrical and optical investigation of plasma plumes generated by femtosecond laser ablation of various metals, The 13th Conference on Laser Ablation (COLA-2015) Cairns, Australia.

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33. B.C. Hodoroaba, S. A. Irimiciuc, G. Bulai, C. Focsa, S. Gurlui, Studiul plasmei de ferita de cobalt produsa prin ablatie laser, FTEM, 2015, Iasi, Romania.

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