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EVALUATION OF LASER BEAM INTERACTION WITH CARBON BASED MATERIAL – GLASSY CARBON

Article Highlights

- Interaction of glassy carbon with Nd^{3+}:YAG laser beam of various energy densities were investigated
- Damage morphology analysis has shown that damage morphology depends on the energy density
- The Image analysis have applied for obtaining quantitative description of the damage topology
- Numerical simulation of glassy carbon interaction with laser was performed by COMSOL Multiphysics
- Numerical simulation was based on thermal model and FEM method

Abstract

Laser beam interaction with carbon based material (glassy carbon) is analyzed in this paper. A Nd^{3+}:YAG laser beam (1.06 μm, i.e., near infrared range (NIR)) in ms regime with various energy densities is used. In all experiments, provided in applied working regimes, surface damages have occurred. The results of laser damages are analyzed by light and scanning electron microscopy (SEM). Image J software is used for quantitative analysis of generated damages based on micrographs obtained by light and SEM microscopes. Temperature distribution in the exposed samples is evaluated by numerical simulations based on COMSOL Multiphysics 3.5 software in a limited energy range.

Keywords: laser, interaction, glassy carbon based material, quantitative analysis, numerical simulation.

Carbon based modern materials in practice can be found in a number of types [1]. Various arrangements of microstructure domains, ordered and disordered, are produced in commercial or laboratory samples, leading to design of carbon materials of different characteristics. The new technological processes make possibility for designing and producing materials with desired mechanical, thermal, electrical, and other properties [2,3]. Laser interaction with well-known graphite - and diamond-like materials remains an unresolved issue. Recently, carbon active materials, carbon nanotubes, ^{60}C and carbon composites, have been attracting attention. Fabrics and generally fiber-based carbon materials have not theoretically studied as much as to the previously mentioned group, despite of laser application in textile industry [4,5]. Activated carbon cloth modifications initiated by pulsed transversely excited atmospheric CO_2 (TEA CO_2) laser radiation (pulse intensities from 0.5 to 28 MW/cm^2) depend on the cloth adsorption characteristics [6]. Simulation of the heating effects for the exposure of carbon textile materials and C/C composite to alexandrite and Nd^{3+}:YAG (yttrium aluminum garnet) laser radiation but under different laser working regimes (mean power 0.2--6.5 kW), have also been examined [7].

Glassy carbon has excellent mechanical, thermal and electric properties [8,9]. On the other hand, it is difficult to treat and process by conventional techniques [8]. Laser pretreatments have been used on carbon electrodes in electrochemical experiments to produce a surface as free as

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possible from contamination and to activate the electrode toward electron transfer as well as for the fabrication of the micro fuel cell flow fields, a tripled Nd^{3+}:YAG were proposed. The possible use of laser for processing of such materials is one of the motivations for experimental research. Depending on the laser working conditions as well as the complexity of the carbon based material, many different processes can be provoked [10,11]. Often, a distribution of thermal processing is a reliable starting point for descriptions of laser beam-material interaction. For select materials it can be expected that most of the laser beam energy will be absorbed and converted into heat.

Image analyzing and processing is a methodology developed and implemented in advanced research methods for various theoretical and practical tasks in industry [12]. Data obtained by light and electron microscopes are applied for material characterization and induced material transformations by the laser beams. Historically, their beginnings can be found in geology [13], where the first methods of image analysis had been stemmed. They were based on the fact that an area that occupied some algorithm in the image was correlated to its contributions in the rock. Various authors worked to the images of two-dimensional cross section based on general models applied to mathematical problems [14,15]. Quantitative analysis and modeling has stemmed from Saltykov [16]. The development of modern informatics tools with new generation of computers opens wide prospects for image analysis. It can be considered that the computer image analyses developed over six decades. In cyber space, the image is represented in its digital form. Modern techniques of scene-object-presentation include beside ordinary digital photo devices, thermal imaging cameras, TV image, holographic image, tomographic presentation, etc. Digital images as vector or raster types are presented by pixels intensities in 2D space. The schematic diagram of an image analysis methodology is presented in Figure 1 [17].

The aim of this work is to analyze in more detail the effect of the Nd^{3+}:YAG laser beam interaction with glassy carbon based material. The experimental damages were analyzed by appropriate microscopes and the obtained micrographs were the object for image analyses processes. For chosen experimental data the adequate analyses by Image J is performed in the form of histograms and appropriate statistics. It can serve to give quantitative information of the distribution or the level in the grey scale. Besides that, mean grey level as well as standard deviation for the characterization are performed, too. All this could be useful for quantitative interpretation of interaction of glassy carbon and Nd^{3+}:YAG laser.

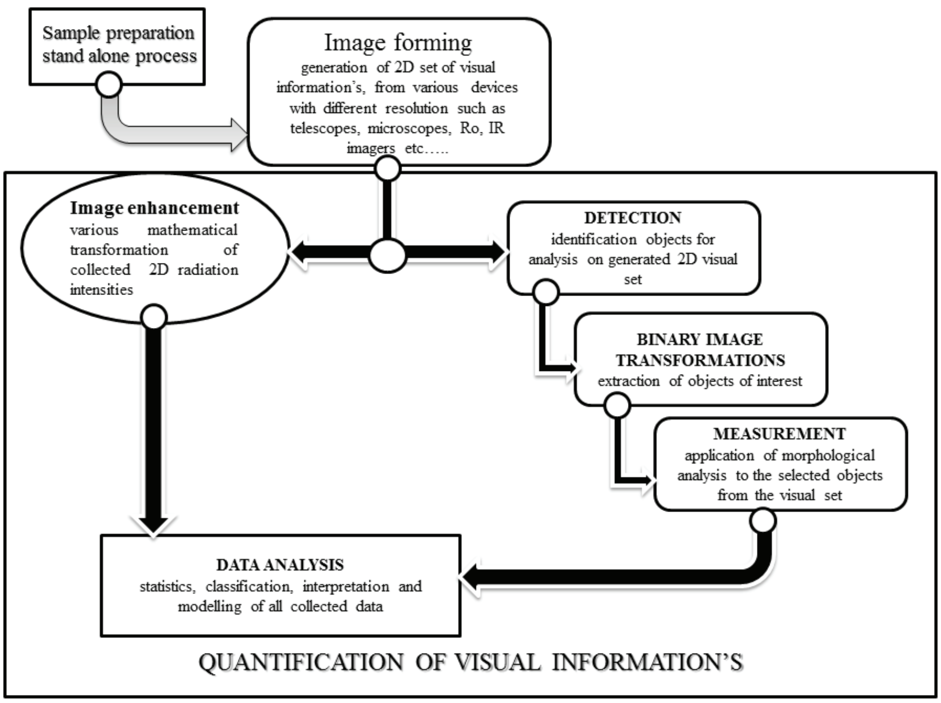

Figure 1. Schematic diagram of an image analysis methodology.

EXPERIMENTAL

The samples of the glassy carbon were exposed to Nd^{3+}:YAG laser beams (Table 1). The direction of the beam was perpendicular to the sample surface. The laser beam was focused by the appropriate lens. The working conditions are presented as mean power, total exposition, one pulse exposition and mean energy density.

Table 1. Parameters of laser system used in experiments

Wavelenght	1.064 μm
Mean power	5 kW
Energy density	1468 J/cm^2
Pulse duration	0.7 ms

Obtained damages are analyzed by light and scanning electron (SEM) microscopy. The appropriate micrographs were the basis for further quantitative analyses of damage shape, damaged surface, damage color, etc.

Samples were in the shape of thin plates (1 mm thickness), *i.e.*, the surface was much larger than the beam spot. Depending on the methods of material production, data for thermal performances can vary.

Images obtained by optical and SEM microscopies were analyzed by the Image pro Plus Premier software package. Dimensions of hole and influence zone of laser beam material interaction were measured by 100 individual measurements.

The properties of glassy carbon are: specific heat (C_p) is 1500 J/(kg K), heat conductivity is 2 W/mK and density (ρ) is 1450 kg/m^3.

RESULTS AND DISCUSSION

Analyses of damages obtained by light and scanning electron microscopes are presented in Figures 2 and 3, respectively.

Analyses of images obtained by the light microscope

By analyzing micrographs obtained by the light microscope, three ring-like parts can be seen. The largest ring is formed by complex processes during the laser exposition. This area is characterized by a number of small grains. A crater shape is formed and the boundary of the ejected material and solidified melts make a wall around the crater. The ejected material forms a kind of semi torus. A white ring-like area is formed between the irregularly ejected material and formatted crater walls (higher than the basic material surface). The crater edges show consequences of melting and solidification processes (Figure 2). Figure 2 presents the optical micrograph analysis of Nd^{3+}:YAG laser, 1.06 μm, 1468 J/cm^2, 0.7 ms and glassy carbon interaction.

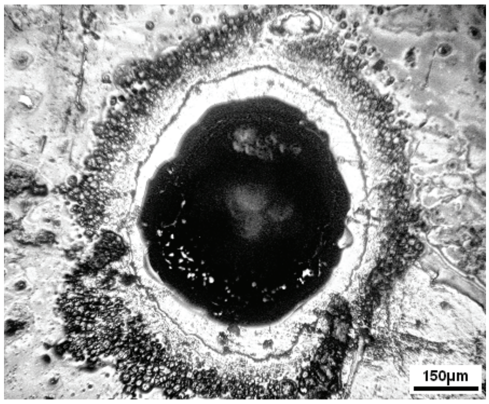

Figure 2. Light micrograph analysis of laser damage.

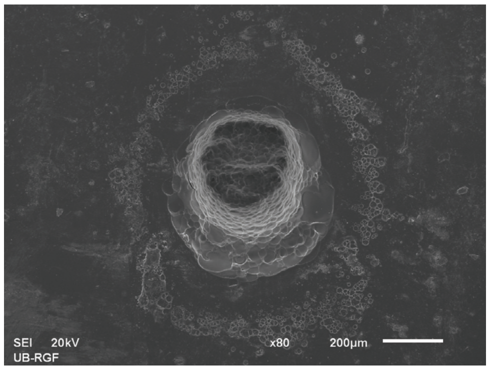

Figure 3. SEM micrograph of laser damage.

Damage diameter and the interaction zones were defined by measurements in 100 different directions (angle 3.6°). Statistical analyzes of obtained measurements are presented in Table 2. Therefore, the mean diameter of affected zone is 247±17.68 μm in reality. Mean crater diameter is 137.77±6.48 μm. All provoked shapes are approx-

Table 2. Statistical analysis of measurements obtained from light microscope images

Length	Mean diameter, μm	Standard deviation	SE of mean	Variance	Coefficient of variation
1st	137.77	6.84	2.16	46.79	0.049
2nd	173.05	18.81	5.43	353.96	0.108
3rd	247.19	17.68	4.29	312.66	0.071

imately circular. The obtained values are graphically presented in Figure 4.

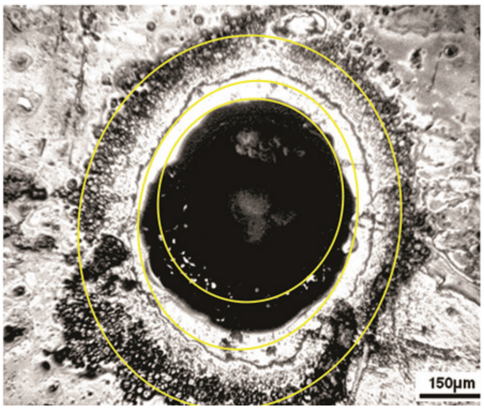

Figure 4. Obtained measured values of laser material interaction.

The crater-like damage with ejected material is very different from the whole image (Figure 4).

The mentioned and marked ring (Figure 4) has a mean grey level 221.51 (closer to the white color) and standard deviation 46.2. This area does not show significant influence of the laser beam. It can be considered that the energy density of beam is under the damage threshold.

The crater area with the wall of ejected material is 209735.7 µm^2, and the surface of internal ring 92381.3 µm^2. If we approximate that the crater with the vicinity (with wall formation) is of circular shape with the diameter of 523.3 µm, its surface should be 215055.98 µm^2. The relationship of the calculated and real surfaces is 1.025, therefore the quantitative measure of deviations of circular crater shape is 2.5%. The crater (without wall formation) with assumptions regular circular shape dia. 383.62 µm should occupy 1155832.0 µm^2. The relationship of the calculated and real surfaces is 1.097, therefore the quantitative measure of the deviation (from the circular shape), factor of merit-FOM is 9.7% from the circular shape.

Since manual counting of grain formation of ejected material would be impractical, its number can be defined using the Analyze Particles function in Image J for the presented analyses. The Figure 2 is taken with some higher resolution, and with larger pixel number (image size is 550×468 pixels). The formation of ejected material grains with circular shape (regular or irregular circles) can be seen in the micrographs (Figures 2 and 3). Accordingly, the criteria for counting can be circularity in the range 0.2-1.0 and maximal area of 100 pixels as no grain is larger than this value. The obtained number of recognized objects with the chosen criteria is 496. The mean area of recognized objects is 7.52 pixels. In the analyzed micrograph (Figure 2), the detail of shot - size is 2.11 µm×2.11 µm, *i.e.*, 4.44 µm^2. Therefore, the mean surface of single characteristic formation of materials is 33.36 µm^2.

Analyses of SEM micrograph

Scanning electron microscope analyses offer more precise description of material damages. The boundary of specified characteristic areas between the indented places, *i.e.*, crater and solidified melt in the vicinity of crater, is more expressive. The wall of the ejected materials (solidified melt) contains two characteristic parts (Figure 3). The part closer to the crater is somewhat higher and wrinkled (ripple-like). The further part is smoother and lower. The details of structure of the ring-like parts can be better seen. (The formatted structures were the consequences of ejected material.)

Figure 3, *i.e.*, the SEM micrograph of the damage sample was obtained at 80× magnification. Therefore, the characteristic value for characterization is 1.58 µm^2. By using the same methodology, as for the light microscope analyses, for SEM image mean diameter 584.66 µm is obtained.

By using selection tools, the highlighted area of the micrograph, which presents the crater with the solidified melted materials, we obtained 263660.8 µm^2. For approximation of the damage of the regular circular shape (dia. 584.66 µm), its surface should be 268473.61 µm^2. The relationship of the calculated and the real surfaces is 1.018, which means that the quantitative measure of the shape deviations from the circle is 1.8%.

By selecting only crater area (without wall formation), the analyses give 66360.15 µm^2. For the approximation of circular shape (diameter 295.66 µm), the calculated crater area should be 68654.06 µm^2. The relationship between calculated and real surfaces is 1.034, which means that the quantitative measure of shape deviation from the circular one is 3.4%.

The relationship of whole damage surface and crater surface (at cross section) is 3.97. Therefore, 25.17% of the total damage was affected material surface.

Modeling of interaction of glassy carbon with Nd^{3+}:YAG laser

One of the principal evaluations for provoked processes is the presentation of the temperature distribution. It is provoked by laser energy absorption and other transformation processes in the material. The treated sample was in solid state and the application mode for conductive processes is the most applicable. The heat equation is used in the form of:

$$\delta_{ts}\rho C_p \frac{\partial T}{\partial t} - \nabla\left(k\nabla T\right) = Q \quad (1)$$

In Eq. (1), δ_{ts} is the coefficient of time scaling, ρ density of the material, C_p specific heat by constant pressure, k component of the tensor of heat conductivity in the most generalized case, Q is heat source or sink [18]. Note that the modeling was by using program package COMSOL Multyphysics 3.5. (The heat conductivity of carbon based materials thanks to various material types can be subject of discussion [19].)

In some cases where it is of interest, transversal convection or radiation can be implemented. In planar 2D cases, two new terms are included in equation (2):

$$\delta_{ts}\rho C_p \frac{\partial T}{\partial t} - \nabla\left(k\nabla T\right) = Q + \\ + \frac{h_{trans}}{dA}\left(T_{ext} - T\right) + \frac{c_{trans}}{dA}\left(T^4_{ambtrans} - T^4\right) \quad (2)$$

The new terms include thermal energy which material reemits to the environment.

For laser-material interaction modeling purposes (here glassy carbon sample and laser pulsed beam), Eq. (2) is applied. This approach considers that the total energy of the laser pulse is absorbed in the exposed material without reflection and transmission, *i.e.*, that it is converted in the material heating. The assumption is that the sample has the characteristics of black body and (emissivity of ε = 1) and that environmental temperature is 300 K. For the laser beam parameters, the data of Table 3 are applied.

Table 3. Parameters of laser source used in simulation

Wavelenght, μm	1.064
Mean power, kW	0.35, 0.4, 5
Energy density, J/cm^2	112, 128, 1468
Pulse duration, ms	0.7

The Eqs. (1) and (2) hold for the case without phase transformations. For glassy carbon that is valid in the temperature range up to 3652 °C (3925 K), when the melting processes start. Based on described assumption, the temperature distributions in the sample after the laser pulse of various energies can be presented as in Figure 5. It means that the simulations are performed in the moment t = 0.7 ms from the start of exposition.

Figure 6a and b represent results of simulation for energy density 112 and 128 J/ cm^2, respectively. The maximum temperatures are the below melting point and this temperature distribution can be considered as correct. In the results

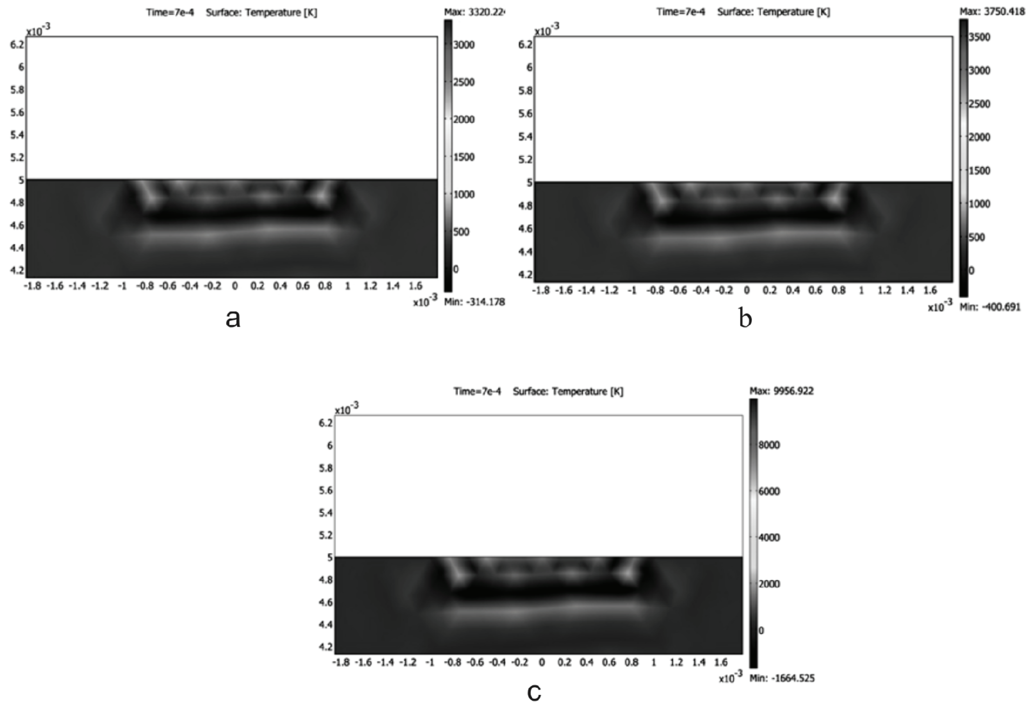

Figure 6. Temperature distribution in glassy carbon after the exposition it to Nd^{3+}:YAG laser power density: a)112, b) 128 and c)1468 J/cm^2; exposition time 0.7 ms.

of simulations presented in Figure 6c, the maximal temperature is on the sample surface in the laser spot area and it is 9956.9 K. That value is the result of the simulation based on Eq. (2), which is without existing phase transformation of material, as mentioned. But, the obtained temperature is significantly above of the melting point of the chosen material, *i.e.*, above 3925 K, as well as above the boiling point (4473 K). Therefore, the chosen model is not applicable to the simulated data, and without the performed experiments, we could not suppose that the crater and other formations will be present. The simulation shows clearly that at chosen densities and time of exposition, phase transitions have to be taken into account. This shows ambiguously the experimental data and microscopic sample analysis after laser beam action in working conditions from Table 1. The micrographs of samples analyzed by light and scanning electron microscopies have shown clear ablation and melting processes, provoked by Nd^{3+}:YAG laser in working conditions of Table 1. So, the value obtained for those simulations (9956.9 K, Figure 6c) cannot be considered as correct because the used model is only valid for cases without melting, boiling and ablation processes.

In the performed experiments with glassy carbon and chosen working conditions of Nd^{3+}:YAG lasers, pronounced effects of interactions were found. The damages of material sample are surficial. Crater formations as well as ejection and melting processes of materials were found, ejected in the crater space and ring-like areas. The SEM analyses are more precise than the light microscope. The light microscope images were taken with higher contrast between the characteristics areas of the damaged surface, which was applicable for the image analysis. This conclusion is possible for further discussion and depends on the experimental parameters of recording. It seems that those micrographs were better for histogram presentation, mean grey level and standard deviation of grey scale level.

The computer simulations based on thermal model and assumed approximations have to be taken into account for further analyses and implementations of new (standard) terms, but for the first understanding of possible provoked temperatures, they can be helpful.

If without experiments we include some parameters for simulations, the obtained temperatures range gives the first answers, if the material is close to melting or not. After that, the model has to be changed and the new terms have to be implemented with phase transformations of some order. More exact modeling has to pay attention to the melt flow, change of surface form due to melting and ablation processes, Stephens models, etc., and to take into account mechanical processes, too. On the other hand, depending on the pulse length, heat model have to be changed or used with different materials constants. For very fast phenomena only the parts of electronic contributions have to be included in conductivity processes, or other modeling have to be applied (hydrodynamic equations, phenomenological models, etc.).

CONCLUSION

Experiments in the area of laser-material interaction give the most exact answers for the phenomena. In this case, the effects of melting, ablations, specific material formations, craters, segregations, ring-like formations were provoked for the case of glassy carbon and Nd^{3+}:YAG laser in applied working regimes.

The image analysis can be a task for further interpretations of the correlations between the applied laser beam distributions (cross section of the beam) and influence of the material porosity and anisotropy.

In the experiments, material ejection is found. Usually, the experiments are divided into considerations of the material state before and after laser interactions or descriptions of the material environment where we could see the processes of ejections and the nature of the ejected particles, ions, etc.

Based on the obtained image histograms, color analysis and recognition of certain types of elementary geometric forms (their numbers and areas) it is possible to get quantified parameters of interaction. These analyses should be linked with experimental processes (surface oxidation, crater formation, ablations, etc.) and to quantify the intensity of interaction. Absorption and heat conduction in the material were calculated in two dimensions based on finite element methods. The results can be considered as initial answers.

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NAUČNI RAD

PROCENA INTERAKCIJE LASERSKOG SNOPA SA MATERIJALOM NA BAZI UGLJENIKA - STAKLASTIM UGLJENIKOM

U ovom radu se analizira interakcija laserskih snopova sa ugljeničnim materijalom (staklasti ugljenik). Korišćen je Nd^{3+}:YAG lasera (1,06 μm, odnosno bliska IC oblast) u ms režimu rada sa snopovima različitih gustina energije. U svim eksperimentima, u primenjenom režimu rada, uočene su površinske povrede na uzorku. Povrede nastale dejstvom lasera su analizirane optičkim i SEM mikroskopima. Program Image J je korišćen za kvantitativnu analizu nastalih povreda na osnovu mikrografa dobijenih optičkom i SEM mikroskopijom. Temperaturna raspodela u izloženom uzorku je dobijena numeričkom simulacijom zasnovanom na programskom paketu COMSOL Multiphysics 3.5 u ograničenom opsegu energija.

Ključne reči: laser, interakcija, staklasti ugljenik, kvantitativna analiza, numerička simulacija.