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Optics

Laser-induced thermal emission of boron carbide

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Abstract. Properties of laser-induced thermal emission of surface layers of boron carbide under irradiation with Q-switched laser pulses were experimentally investigated. A significant dependence of the amplitude and duration of thermal radiation pulses on the intensity and dose of laser irradiation was revealed. In particular, irradiation with a sequence of laser pulses reduced the amplitude and duration of thermal emission pulses by several times. Moreover, anisotropy of the laser-induced thermal emission, which manifested itself as a dependence of the emission pulse duration on the observation direction, was found out. In particular, when the emission was detected along the surface of a sample, the pulse duration was several times longer than in the case of the observation direction perpendicular to the sample surface. The indicated features of the laser-induced thermal emission were explained by influence of melting and solidification processes of boron carbide under laser irradiation, as well as by inhomogeneity of laser heating of rough surfaces.

Keywords: laser-induced thermal emission, boron carbide, anisotropy.

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1. Introduction

Among the variety of modern laser technologies used in research laboratories and industry, the technologies based on thermal effect of laser radiation are of great importance [1–3]. In most cases, significant changes in the pattern of thermal radiation of an irradiated object occur due to local heating of the area, where the laser radiation is absorbed. In some cases of pulsed laser heating, temperatures of thousands of degrees are reached, and, as a result, laser-induced thermal emission (LITE) is observed in the visible spectral range. In such cases, the LITE is easily distinguished against the background of the equilibrium thermal radiation of the surrounding environment and carries useful information about high-temperature properties of the irradiated material, its structure, and the processes of interaction of laser radiation with the irradiated object.

Among the materials, in which the properties of visible LITE have been studied, carbon materials, in particular, aerodispersed soot [4, 5], carbon black suspensions [6–8], carbon nanotubes [9, 10], bulk carbon materials, including carbon electrodes, activated carbon, charcoal [11–14], and semiconductor materials [15–17] are worthy of mentioning.

This work is devoted to the study of visible LITE of boron carbide. Boron carbide is a unique material that attracts attention of researchers for many years. The interest in boron carbide is largely caused by its outstanding properties, in particular, its chemical stability, high hardness, resistance to high temperatures, as well as large value of neutron capture cross section [18–24]. As a result, boron carbide has found diverse applications, including production of abrasive materials, manufacture of armor, protective coatings for sand-blasting nozzles, chemical dishes, *etc*.

Usually, hot pressing at temperatures above 2000 °C is used for synthesyzing boron carbide. Due to high values of the absorption coefficient of boron carbide [21, 25], the temperatures of the order of 2000 °C can be easily achieved under pulsed laser irradiation, which opens up new possibilities for synthesis, processing and modification of this material and raises interest in studying its behavior under powerful laser irradiation [24, 26–32].

In this work, the properties of LITE of boron carbide in the visible spectral region under irradiation by laser pulses with a pulselength of about 10^{-8} s were studied. In particular, the dependence of the amplitude and shape of the LITE pulses on the power density and dose of the laser irradiation was revealed. Moreover, a significant anisotropy of the LITE, which manifests itself as a dependence of the duration of the LITE pulses on the direction of their observation, was revealed.

2. Methods

In this work, a Q-switched YAG-Nd laser (1064 nm, 20 ns) was used in the experiments. The intensity distribution of the laser radiation in the plane perpendicular to the beam axis was close to a bell-shaped one. The LITE was recorded through glass filters in spectral ranges of approximately 20 nm around 560 and 430 nm. A photomultiplier with a rise time of 1.3 ns and a digital oscilloscope with a frequency bandwidth of 250 MHz were used to record pulsed optical signals, similarly to [13]. Measurements were performed in a single-pulse mode with an interval between the individual measurements of the order of 30 s. The studies were carried out in an air atmosphere at normal room temperature and humidity.

Boron carbide samples with rough surfaces formed by cutting with an abrasive material were used for the studies. A typical sample surface relief is shown in Fig. 1.

3. Results and discussion

The experiments show that pulsed laser radiation with a power density of the order of 30 MW/cm^2 excites secondary emission from the samples, which has a broad spectrum covering the entire visible region. The measurements reveal that the intensity of this emission nonlinearly depends on the intensity of the laser excitation. In particular, when the emission is recorded in a relatively narrow spectral range, the dependence of the emission pulse amplitude I_{max} on the laser excitation intensity F can be approximated by the power function $I_{max} = \text{const} \cdot F^{\gamma}$, where the exponent γ takes values from 2 to 3 (see Fig. 2). The mentioned nonlinear dependence $I_{max}(F)$ is typical for laser-induced thermal emission of microparticles and surface layers of light-absorbing materials under pulsed laser irradiation [6, 7, 14].

One should pay attention to the following important circumstance. When the studied boron carbide samples were irradiated with sequences of laser pulses, the amplitude and shape of the emission pulses changed significantly with the laser irradiation dose. In particular, a noticeable decrease in the intensity of the emission pulses with the increase of the number of the laser pulses absorbed by the sample surface was observed. For example, Fig. 3 shows the dependence of the emission pulse amplitude I_{max} on the number N of laser pulses in the sequence, *i.e.* on the laser irradiation dose. The results presented in Fig. 3 correspond to the laser radiation intensity $F = 33$ MW/cm^2. Accordingly, for laser irradiation with a single pulse, the surface density of the absorbed energy is approximately 0.66 J/cm^2. As can be seen from Fig. 3, when the sample is irradiated by a sequence of laser pulses, a significant (fivefold) decrease of the intensity of the LITE is observed.

The above-mentioned decrease of the LITE intensity induced by laser irradiation significantly complicates measurements of characteristics of the emission pulses. The results shown in Fig. 2 were obtained on the samples, which were preliminary irradiated by $N \geq 20$ laser pulses (according to Fig. 3).

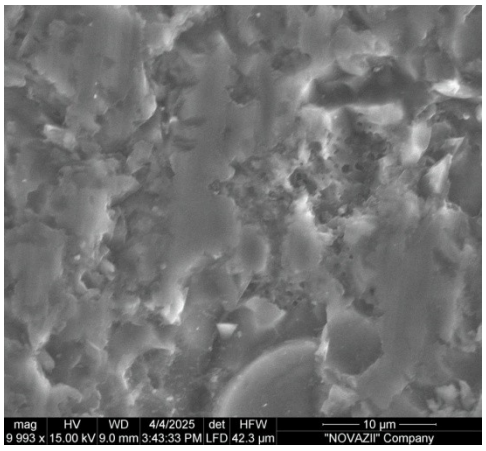

Fig. 1. SEM image of the surface of a studied sample.

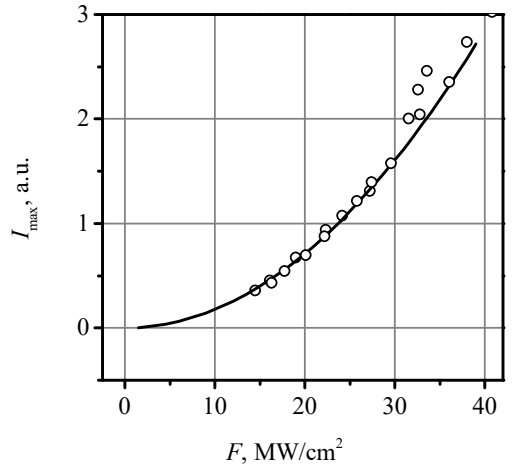

Fig. 2. Dependence of the amplitude of LITE pulses on the intensity of laser excitation (open circles). The solid curve represents parabolic dependence $I_{max} = \text{const} \cdot F^2$.

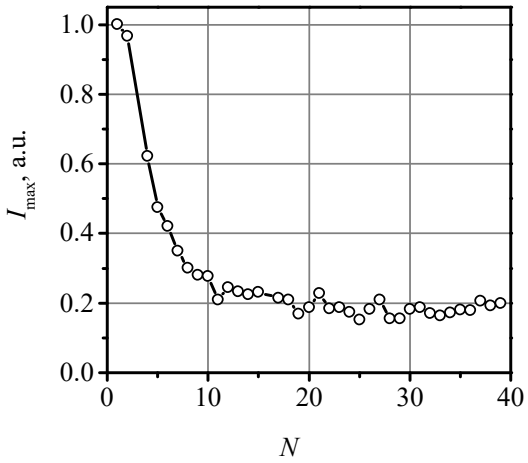

Fig. 3. Dependence of the amplitude of LITE pulses on the number of laser pulses in the sequence.

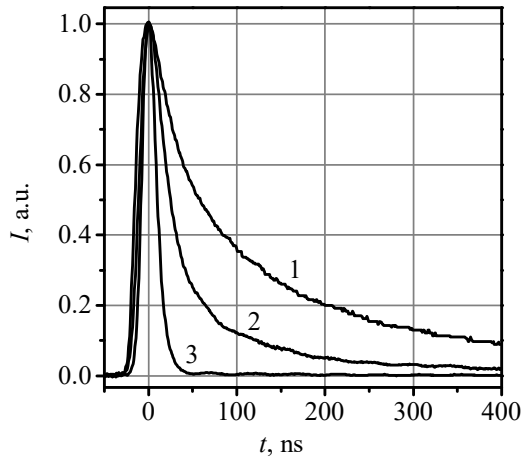

Fig. 4. Normalized oscillograms of LITE pulses (see explanation in the text).

The measurements show that the LITE pulses have short leading edges and relatively long trailing edges. The typical leading edge duration is about 10…15 ns, which is 50…75% of the duration of a laser excitation pulse. The duration of the trailing edges of the LITE pulses significantly depends on the measurement conditions and can exceed the laser pulse duration by an order of magnitude.

Fig. 4 shows oscillograms of LITE pulses at a wavelength of 560 nm, obtained at irradiation of the same surface area with a sequence of laser pulses. All the oscillograms in Fig. 4 are normalized and correspond to the laser excitation intensity of 33 MW/cm^2.

In addition to the effect of the LITE decrease presented in Fig. 3, the experiments also show that the duration of the trailing edge of the emission pulses is significantly reduced after irradiation by a sequence of laser pulses. For the normalized oscillograms shown in Fig. 4, the experimental conditions were the following. All the curves in Fig. 4 were obtained at the same laser excitation intensity of 33 MW/cm^2. The curve *1* is the result of averaging the first two pulses in the sequence shown in Fig. 3. The curve *2* results from averaging the pulses with $N = 35…40$ in Fig. 3, which corresponds to the laser irradiation dose of about 25 J/cm^2. The curve *3* in Fig. 4 is obtained after the curve *2* after additional irradiation of the sample with 20 pulses with the increased intensity $F = 100$ MW/cm^2 thus making the laser irradiation dose equal to 65 J/cm^2. As can be seen from Fig. 4, after the laser irradiation, the duration of the LITE decay decreases by more than an order of magnitude. For example, the trailing edge of the emission pulse before the irradiation (curve *1* in Fig. 4) is almost 20 times longer than after the irradiation (curve *3* in Fig. 4).

The above-mentioned results of the experiments demonstrating the behavior of the boron carbide LITE with the increase of the laser irradiation dose (Figs 3 and 4) require explanation. We consider possible mechanisms of the observed LITE properties. It should be noted first that the laser radiation power used in the experiments is sufficient to reach the melting temperature of boron carbide. To estimate the surface temperature, which corresponds to the maximum of the LITE oscillogram, a dual-frequency pyrometry method was used. The LITE signals were measured at two different wavelengths (560 and 430 nm) under the same excitation conditions. Radiation of a tungsten incandescent lamp with the temperature determined with an optical pyrometer was used for calibration. For the experimental conditions corresponding to $N > 20$ in Fig. 3, the results of the measurements of LITE oscillograms gave an estimate of the maximal surface temperature of about 2500…2600 K, which is 100…200 degrees lower than the melting point of boron carbide (approximately 2700 K). Taking this estimate of temperature of 2500…2600 K for $N > 20$ in Fig. 3 and using the Planck's formula for blackbody thermal emission, we can make an estimate of the temperature corresponding to $N = 1$ in Fig. 3. As far as at $N = 1$ the emission intensity is approximately five times higher than at $N = 20$, it follows from the Planck's formula that the temperature at $N = 1$ exceeds the temperature at $N = 20$ by 250…400 K. According to these estimates, the curve *1* in Fig. 4 corresponds to the following situation: the surface layer melts under the laser pulse, and the resulting liquid is heated up to the temperatures hundreds of degrees above the melting point.

Given the above estimates of the surface temperature during laser irradiation, the kinetics of laser melting for the conditions of the experiments corresponding to $N < 10$ (Fig. 3) may be presented as follows. The irradiated surface layer of the material is gradually heated up and reaches the melting point. After this, heating of the surface slows down, but the inner deeper layers continue to heat up. Over time, the surface layer melts, and the liquid begins to heat up to higher temperatures. When the temperature of the molten layer reaches several hundred degrees above the melting point, the LITE signal from the sample is primarily formed by thermal emission of the molten layer. The process of heating of the surface layer continues at the trailing edge of the laser pulse until the moment when heat transfer from the surface layer exceeds the laser heating power. At this moment, the maximal value of the surface layer temperature is reached, and the peak of the LITE pulse is formed. After that, cooling of the surface layer begins and the trailing edge of the LITE pulse is formed. During cooling of the surface layer, the material's solidification temperature is reached, and the latent heat of phase transition is released, which leads to a noticeable slowdown of the temperature decay. In the experiments, this slowdown causes the observed elongation of the LITE pulses with increasing the intensity of the laser excitation.

The dose dependence of the shape of LITE pulses observed in the experiment should be considered as follows. The decrease of the amplitude of the LITE pulses with the increase of the laser irradiation dose

(Fig. 3) indicates a significant decrease in the efficiency of laser heating of the surface layer. Since the irradiated material is a fine-grained ceramic, it is logical to assume that the surface layer melting may improve thermal contact between the neighboring grains. Then, due to the increase of heat dissipation, laser pulses of the same intensity heat the surface to lower temperatures, hence the amplitude and duration of the emission pulses decrease.

Moreover, it may be also assumed that boron carbide can oxidize in the air [21], which can lead to accumulation of B_2O_3 oxide in the cavities between the grains. This can also improve heat transfer from the heated surface deep into the sample and cause the decrease of the amplitude and duration of the LITE signals.

Finally, another important property of the LITE of boron carbide should be mentioned. The experiments show that duration of the LITE pulses significantly depends on the geometry of the experiment, in particular, on the direction of observation. Fig. 5 shows typical normalized LITE oscillograms obtained under the condition $N > 20$ (Fig. 3) at different positions of the photodetector relative to the laser beam and the irradiated sample. Both oscillograms in Fig. 5 correspond to normal incidence of laser beam on the sample surface. As can be seen from Fig. 5, when the emission is observed in the tangential direction (along the sample surface, perpendicular to the laser beam axis), its pulse length is much longer than in the case of the opposite (relative to the laser beam) direction of observation. For example, if we measure the pulse length at a level of 10% of the peak value, then the length of the oscillogram 2 in Fig. 5 is about 180 ns, while the oscillogram 1 is only 36 ns long.

The above-mentioned anisotropy of the LITE of boron carbide is most pronounced on surfaces with strong roughness formed as a result of sample fracture. Just this case is presented in Fig. 5.

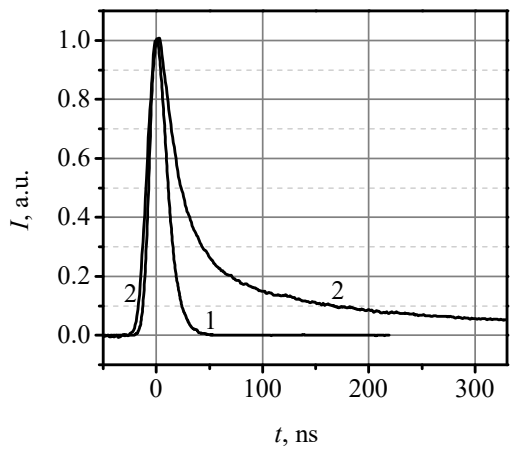

Fig. 5. Normalized oscillograms of LITE recorded at F = 33 MW/cm^2 at a wavelength of 560 nm with the direction of observation perpendicular to the sample surface (*1*) and along the sample surface (perpendicular to the laser beam) (*2*).

The nature of the observed anisotropy of the LITE of boron carbide can be explained similarly to the anisotropy of LITE of carbon materials [35]. As shown in [12] by computer simulations for carbon materials, rough surfaces under laser irradiation are heated inhomogeneously. In particular, protruding elements (peaks) on a rough surface are heated to significantly (by hundreds of degrees) higher temperatures than valleys located between the peaks. The mentioned simulations also show that the laser-heated peaks emit longer LITE signals than the valleys due to the complicated geometry of heat release and transfer in the surface layer. Besides, as far as the overheated peaks melt before the valleys, the LITE signals from the peaks are much longer than the signals from the valleys (according to the results shown in Fig. 4). As a result, due to partial shading of the valleys by the neighboring peaks, the total LITE signal observed along the sample surface is mainly formed by the overheated peaks causing the above-mentioned anisotropy of LITE to be detected.

4. Concluding remarks

Summing up, an additional attention should be paid to the observed strong dependence of the amplitude and shape of LITE pulses on the measurement conditions and previous history of laser irradiation of a selected surface area. On the one hand, these dependences significantly complicate measurements of pulsed LITE characteristics. Furthermore, they cause high sensitivity of LITE to the properties of the irradiated surface as well as to the processes involved in pulsed laser heating.

Acknowledgments

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Authors' contributions

Zelensky S.E.: conceptualization, investigation, formal analysis, writing – review & editing.
Kolesnik O.S.: sample preparation, investigation.

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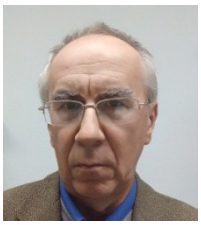

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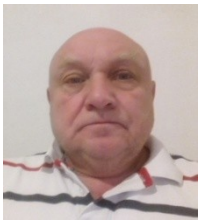

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Індуковане лазером теплове випромінювання карбіду бору

С.Є. Зеленський, О.С. Колесник

Анотація. Експериментально досліджено властивості індукованого лазером теплового випромінювання поверхневих шарів карбіду бору при опроміненні імпульсами лазера з модуляцією добротності. Виявлено значну залежність амплітуди та тривалості імпульсів теплового випромінювання від інтенсивності та дози лазерного опромінення. Зокрема, при опроміненні послідовністю лазерних імпульсів амплітуда і тривалість імпульсів теплового випромінювання зменшуються у декілька разів. Крім того, виявлено анізотропію індукованого лазером теплового випромінювання, яка проявляється як залежність тривалості імпульсу свічення від напрямку спостереження. Зокрема, при реєстрації випромінювання уздовж поверхні зразка тривалість імпульсу у декілька разів довша, ніж у випадку, коли напрямок спостереження перпендикулярний до поверхні зразка. Зазначені особливості індукованого лазером теплового випромінювання пояснюються впливом процесів плавлення та твердіння карбіду бору при лазерному опроміненні, а також неоднорідністю лазерного нагрівання шорстких поверхонь.

Ключові слова: індуковане лазером теплове випромінювання, карбід бору, анізотропія.