Effect of electron irradiation on optical absorption of impact diamonds from the Popigai meteorite crater

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\textbf{A B S T R A C T}

The translucent impact diamonds which consist of nanosized grains of cubic diamond (D) or are a mixture of cubic and hexagonal (D + L) diamond were separated using Raman spectroscopy; high pressure high temperature (HPHT) synthetic diamonds and diamond CVD film were taken for comparison. Transmission spectra before and after electron irradiation with 3.0 MeV energy at $10^{18}$ cm\textsuperscript{-2} dose were recorded. Concentrations of neutral and negatively-charged vacancies ($V_0^-$, $V^- \text{'}$) were estimated from absorption in the GR1 and ND1 zero-phonon lines. In case of impact diamonds vacancy concentrations were estimated from the absorption in a broadband of phonon replicas; the relations binding broadband absorption and vacancy concentration were suggested. We established that only neutral vacancies form in impact diamonds at irradiation and their concentration is comparable with the total $V_0^-$ + $V^- \text{'}$ concentration in HPHT diamonds and diamond CVD film.

\section{1. Introduction}

Diamond possesses extremely high atomic density, thermal conductivity, sound velocity and hardness. It is optically transparent in the UV to far infrared and is capable to work in the extreme conditions: in aggressive medium, at high temperatures and under extreme irradiation. This makes diamond promising for design of electronic devices with extremal parameters and sensors of ionizing irradiation [1,2].

To date the technologies of diamond single crystal growth at high pressure and temperature (HPHT), chemical vapor deposition (CVD) diamond films and detonation diamonds are well developed [1–4]. Today diamond is one of the most studied inorganic materials. Much less is known about impact diamond (ID), which is a nanosized polycrystalline diamond aggregate with 10 to 100 nm grain sizes [5,6]. IDs form in the shock process when meteorite falls on the Earth [7,8]: these diamond aggregates were found in different meteorite craters Canyon Diablo in Arizona [9], Ries in Germany, Kara, Popigai and Puchez-Katunski in Russia [5,10,11] and many others. Impact diamonds (ID) were derived at extremely high P-T parameters (pressure up to 140 GPa and temperature up to 4000 °C) from either parental graphite or coal as a result of solid state phase transition [5]. This conclusion is based on the analysis of grain morphology, preferred orientation of crystallites, occurrence of lonsdaleite (a high-pressure polymorph of carbon with hexagonal close-packed structure) associated with target rock inclusions (material exposed at the site of an impact), and diamond occurrence within large bodies of impact melt rocks. The Popigai astrobleme in the north of Siberia in Russia is one of the largest craters all over the world: its diameter is about 100 km [12]. The remarkable feature of this crater is a high content of graphite in the breccia: as a result a large quantity of impact diamonds formed during the impact event and their reserves exceed many times those of kimberlite diamonds all over the world [11]. Intense study of physical properties of impact diamonds including their structural, mechanical and optical properties began in the USSR soon after the crater discovery: main results of the first decades are summarized in [5,8,10,13]. This study is in progress to date. One of the most impressive results is the fact that wear resistance of impact diamonds surpasses several times the parameters of single crystal diamond [11]. This may be a result of nanosized polycrystalline structure and higher hardness is associated with the fact that grain boundaries interfere with moving dislocations. Some authors explain higher hardness of impact diamonds by lonsdaleite presence: \textit{Ab initio} calculations give the lonsdaleite hardness to be 1.58 times higher than that of the cubic diamond [14]. During last decades the laboratory technique for the production of synthetic analogues of impact diamonds was developed in Japan [15] and these materials are considered promising for different applications in science and industry. Possible examples are the anvils of high-pressure apparatus, elements of machining tools etc.
Irradiation with γ-rays, electrons or heavy particles is widely used to modify the structure and physical properties of material. The γ-ray and electron irradiation are the most delicate: they produce the simplest defects in crystal lattice such as interstitial atoms and vacancies. Vacancies in different charge states (neutral V0 and negatively-charged V−) are well studied in diamond: they show vibronic systems GR1 and ND1 with zero-phonon lines (ZPLs) at 741 and 393.6 nm [16], and these systems are responsible for the strong color of irradiated diamonds. Irradiation with heavy particles such as protons, neutrons and different ions is accompanied by strong damage and disordering of the structure. To date the effect of different radiations is studied in detail for different types of diamond: for natural and HPHT synthetic diamonds, diamond CVD films and detonation nanodiamonds [1,4,17–20]. At the same time no information concerning radiation-induced processes and radiation resistance of impact diamonds is present in literature.

The task of present paper is to study optical absorption in impact diamonds after irradiation with fast 3.0 MeV electrons, to determine the type of generated defects and to estimate their concentration in comparison with HPHT and CVD diamonds as well as to estimate the radiation resistance of impact diamonds. Radiation resistance can be estimated from concentration of vacancies produced at irradiation: the higher vacancy concentration, the lower radiation resistance.

2. Materials and methods

2.1. Spectroscopic measurements

The Raman spectra were recorded on a Horiba Jobin Yvon LabRAM HR800 spectrometer using the 325 nm UV emission of a He–Cd vapor laser with maximal beam power 25 mW. Spectra of IR absorption in the mid-IR were recorded with 1 cm−1 resolution at room temperature on an Infralum FT-801 IR Fourier spectrometer from the Simex Ltd. Co, Novosibirsk. Transmission spectra in the UV–visible-near IR (200 to 1000 nm) were recorded at 0.5 nm resolution at liquid nitrogen temperature using a MDR2 diffraction monochromator (LOMO, St.-Petersburg). Deuterium and incandescent lamps were used as light sources, whereas FEU100 and FEU62 were used as detectors. Samples were irradiated with 3.0 MeV electrons on a linear electron accelerator in the Institute of catalysis SB RAS, Novosibirsk. Using our previous experience on diamond irradiation, we selected a dose of 1018 cm−2, which provides considerable coloration of HPHT diamonds [21].

2.2. Samples

We looked through many tens of impact diamonds from the Popigai meteorite crater and selected three translucent samples: they are shown in Fig.1 (panels a–c). We also took three synthetic diamonds for comparison: two HPHT diamonds (Fig.1, d–e) and a polycrystalline diamond CVD film (Fig.1f). When selecting the ID samples, we used Raman spectroscopy, which is commonly used in everyday practice for characterization and identification of various phases of carbon in diamond films and other structures. The first-order Raman spectrum of cubic diamond is known to consist of a single narrow Lorentzian-shaped T2g peak at 1332.5 cm−1 (4 × 1013 Hz, 165 meV), with full width at half-maximum (FWHM) of 1.5 cm−1 in an undisturbed diamond lattice. For impact diamonds this line is strongly broadened: the FWHM values reach 90 cm−1 [5,11]. This line may be rather symmetric, however, in many cases one observes an asymmetry because of the low-energy components associated with lonsdaleite. According to ab initio calculations [11,22], the Raman spectrum of lonsdaleite consists of three components E2g, A1g and E1g, located near 1221, 1280 and 1338 cm−1 according to [11] (or 1206, 1285 and 1319 cm−1 in [22]), respectively. The ratio between intensities of these components is 1:10:1 [11,22]: A1g component is the most intense. All taken ID samples are flattened and translucent: two of them, A2 and H2, demonstrate a symmetric line about 17 cm−1 thick in Raman spectra, however, for B4 sample this line is much broader (FWHM = 57 cm−1) and considerably asymmetric (Fig. 2).

Thin A2 sample is colorless, whereas H2 and B4 samples are gray with a yellow tint. These ID samples are 100, 450 and 450 μm thick, respectively. It is obvious that samples A2 and H2 consist completely of cubic diamond (D) but B4 sample is a mixture of D and lonsdaleite (D + L). For B4 sample the L input was estimated using a technique suggested by Goryanov et al. [23].

Following these authors, Raman spectrum was decomposed into Gauss components related to cubic diamond (D component near 1332 cm−1) and components L1, L2, L3 of lonsdaleite (E2g, A1g and E1g, respectively). The ratio between integral intensities (L1 + L2)/(L3 + D) was found to be 0.53. Using a calibration Fig. 5 in [23], which shows the correlation between the Raman and XRD data on lonsdaleite fraction, we established that lonsdaleite input is 50 ± 10% in ID sample B4. It is necessary to note another reason for broadening and shift of the Raman line in nanostructured materials: Phonon confinement effect [24]. For example, this effect takes place takes place for detonation nanodiamonds where grain size is about 4.5 nm [25]. In impact diamonds grain size is considerably larger (several tens of nanometers) and Raman line is asymmetric (split into three components) when lonsdaleite is present. This allows us to suppose that lonsdaleite presence is dominant reason of the Raman line broadening.

The HPHT synthetic diamonds SD1 and SD3, taken for comparison, were grown in the Fe-Ni-C system using the temperature gradient technique (Fig.1 d–e). Polycrystalline CVD film is shown in Fig.1f. The
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