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Influence of a high-power pulsed ion beam on the mechanical properties of corundum ceramics

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Abstract. The mechanical properties of near-surface layers of corundum ceramics treated by high-power pulsed ion beam of carbon are investigated. The samples for investigation were prepared from corundum substrate, which is usually used in microelectronic. The ion treatment was carried out at the TEMP-4M facility under the following conditions: an accelerating voltage of 160-200 keV, the current density in the pulse varied within 15-85 A/cm². It was found that ion irradiation changes the structure and properties of near-surface layers of corundum ceramics. At the same time, melting and erosion of the surface layer takes place. These processes are accompanied by the formation of a network of microcracks. Microcracks are propagated only by the depth of melting layer. The mechanical properties were measured using a NanoTest600 nanohardness testing instrument. It was found that the nanohardness depends of the treatment modes. At a current density of 15A/cm², with an increase treatment dose, the nanohardness of the irradiated surface layer increases in comparison with the initial value before irradiation. At higher current densities, the nanohardness of irradiated ceramics decreases relatively to the initial value before irradiation. The dependences of nanohardness off the irradiation dose in this case have the view of a curves with a minimum at irradiation doses of $2.5 \cdot 10^{14}$ and $1.3 \cdot 10^{14}$ cm⁻², for current densities of 50 and 85 A/cm², respectively.

1. Introduction

At present, ceramic materials are widely used. There are widespread use of oxide ceramics, for example, lithium-ferrite ceramics [1–3], zirconium ceramics [4–7] and corundum ceramics [8–10]. When manufacturing of ceramics for functional and instrumental purposes, both traditional and non-traditional methods of processing finished ceramics are used. It is known that ceramic materials, such as corundum and zirconium, are characterized by high strength. Their mechanical processing, in particular, grinding by traditional methods is a very costly process. In many cases, the properties of the ceramic product as a whole depend on the state of the near-surface layer. The use of ceramics with gradient properties has great prospects, when the surface layer differs in its characteristics from the corresponding parameters of the bulk layers. As shown in works [11–24], surface treatment can be effectively carried out using low energy high current pulsed electron beam (LEHCPBE) [11–13], laser treatment [14,15] and high-power pulsed ion beam (HPPIB) [16–24]. To study thin modified layers of dielectrics, the method of secondary ion mass spectrometry [25–27] and the Rutherford backscattering method [28, 29] have proved to be very useful. The above methods of radiation treatment are used to modify and create structures that can not be obtained by traditional technologies.



By varying the modes of surface treatment, it is possible to impart different properties not only to the surface layers of metals, alloys and semiconductors, but also to dielectric materials, thereby expanding the field of their application. Radiation methods of surface treatment of materials have a whole range of advantages. They allow to carry out high-precision treatment of complex shaped ceramic parts with a short exposure time to the surface, are equipped with simple process automation, etc. Among radiation methods of influence on materials, the method of surface modification by high-power pulsed ion beams (HPPIB) [16–24] widely is used.

At present, studies are underway to modify the surface of metals and alloys by high-power pulsed ion beams [19–22, 30]. At the same time, the physicochemical processes taking place in the near-surface layers of dielectric materials under treatment HPPIB have been little studied. The results of the influence of LEHCPEB and HPPIB on zirconium ceramics are reflected in [11, 24]. It is shown that under the influence of such treatments, the surface layer of the material is rapidly heated to the melting point, and then also rapidly cooled [12, 30]. As a result of such influences, surface modification occurs, which is manifested in a change in the phase and chemical composition of the near-surface layer, structuring the surface in the form of grain recrystallization [9, 15, 24].

Consequently, it is possible to change the optical, mechanical and electrical properties of the near-surface layers of the ceramic, and also significantly change the catalytic and adsorption capacity of the surface.

To study the nature of the radiation effect on ceramic materials, it is of interest to investigate the effect, for example, of HPPIB on a ceramic structure that is resistant to changes in the phase composition.

This study aims of this work is to study the influence of HPPIB on the mechanical properties of near-surface layers of corundum ceramics.

2. Experimental technique

Alumina ceramic (corundum) was researched in the work. The samples measuring 1×1 cm in thickness 0.52 mm was cut from a ceramics plate used in the manufacture of microelectronic devices as substrate material.

Ion irradiation of the samples with accelerated C^+ , N^+ and H^+ carbon ions (85% and 15%, respectively) was carried out with a TEMP-4M pulse accelerator [31, 32] at a residual chamber pressure of $2 \cdot 10^{-2}$ Pa. Irradiation modes are the following: an accelerating voltage of 160 – 200 keV, the pulse duration (at half maximum of the accelerating voltage) of the current was 100 ns, the current density in the pulse varied in the range 15 – 85 A/cm². Pulse repetition rate 8 s⁻¹. The energy density in the pulse varied within $W_i = 0.3 - 1.5$ J/cm².

In the experiment were realized three variants of irradiation. The effect of ion treatment were researched depending on the beam current density and irradiation dose, which was set by the number of pulses. Depending on the density of the ion current, the number of pulses varied from 3 to 300. At current densities of 15, 50 and 85 A/cm² the amount of incorporated carbon ions per pulse was $f \sim 7.5 \cdot 10^{12}$ cm⁻², $2.5 \cdot 10^{13}$ cm⁻² and $4.25 \cdot 10^{13}$ cm⁻², respectively.

The surface of irradiated ceramics was analyzed by SEM using a Hitachi TM-3000 Tabletop Microscope. The static electrical conductivity of the corundum surface was measured in the temperature range $T = 25-300^\circ\text{C}$ by the two-probe spreading resistance method [33]. To assess the degree of change in the mechanical properties of the near-surface layers of corundum was obtained of results of nanoindentation with the NanoTest 600 instrument allowing indentation in a wide range of loads. The study was carried out using the method of an unrestored print using the trihedral pyramid of Berkovich [34, 35]. Indentation was carried out in the range of loads from 1 mN to 100 mN. For the statistical analysis, about 12 fingerprints of the indenter was applied to the surface of the samples.

3. Experimental results

In Figure 1a is an SEM analysis of the surface of a sample of corundum ceramics prior to exposure to HPPIB. It can be seen that on the surface of samples is observed the block structure of ceramics. The

dimensions of the blocks vary between 10 and 60 μm . The image shows pores randomly distributed over the surface of corundum.

It is visually observed that after exposure to HPPIB, the surface of the samples darkens, with increasing current density and treatment dose, the samples become darker. It was shown in paper [24] that such a darkening of the ceramic is caused by the formation of oxygen vacancies due to the violation, under the action of HPPIB, of oxygen stoichiometry. Also, the darkening can be influenced by the pyrolysis process on the surface of samples of carbon-containing oil vapors from the residual atmosphere and contamination of the sample surface by the graphite electrode erosion products generated during HPPIB generation.

The impact of HPPIB on corundum ceramics has a thermal character, which leads to melting, evaporation and subsequent recrystallization of the near-surface layer. SEM analysis showed that after the irradiation of HPPIB, a noticeable change in the microstructure of the material surface (Figure 1b). The surface of the ceramics after recrystallization acquires a mosaic structure in the form of separate blocks separated by microcracks. The average block size is 9...11 μm . The process of crack formation is due to the low thermal conductivity of the material and the heat treatment modes. Traces of melting are observed on the surface. Compared to the initial surface of the ceramics, the pores are not observed after HPPIB treatment.

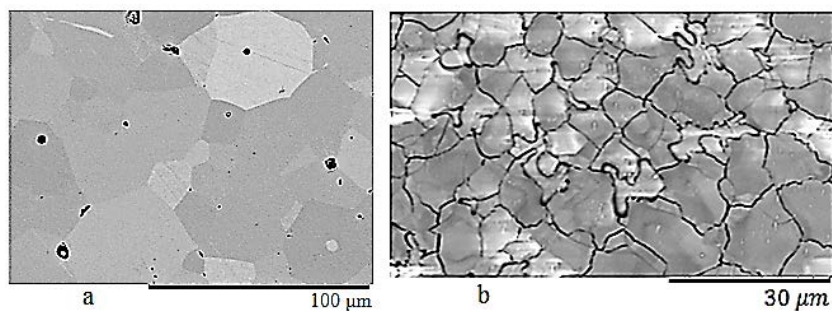


Figure 1. SEM image of the alumina ceramics sample surface: *a* – before HPPIB treatment; *b* – after HPPIB treatment ($j = 85 \text{ A/cm}^2$, number of pulses $N = 3$).

To reduce the intensity of the cracking process, it is possible to choose the heat treatment modes. Such a study is presented in the paper [36], where the optimization of thermal pulse treatment modes is described using the example of the treatment of enamels with a heating light flux. Another possible way to reduce cracking is thermal heating of the sample to a temperature of the order of 0.7 from the sintering temperature and further processing of its HPPIB in the heated state

In Figure 2a–c shows the SEM image of the fracture of ceramic samples exposed to HPPIB. Treatment of HPPIB at an ion beam current density of 15 A/cm^2 does not lead to noticeable changes in the surface layers of the ceramic (Figure 2a). In Figure 2b shows that the modification zone is 6–8 μm . In some areas of this zone a microstructure of the "columnar type" is observed, the grain shapes in the near-surface layers are oriented toward the surface being treated. The process of formation of the "columnar type" structure as a result of the radiation effect was observed in the works [9, 15, 24]. The formed cracks, during the processing of HPPIB, are distributed over the entire depth of the fused layer (Figure 2c). With an increase in the ion current density, the microstructure of the "columnar type" is not observed. This may be due to the high heating rates of the material when impact by HPPIB, as a result of which the material does not have time to recrystallize between pulses. The thickness of the melted layer is practically not increased, since simultaneously with the melting the erosion of the surface. The thickness of the melted layer is 9...11 μm .

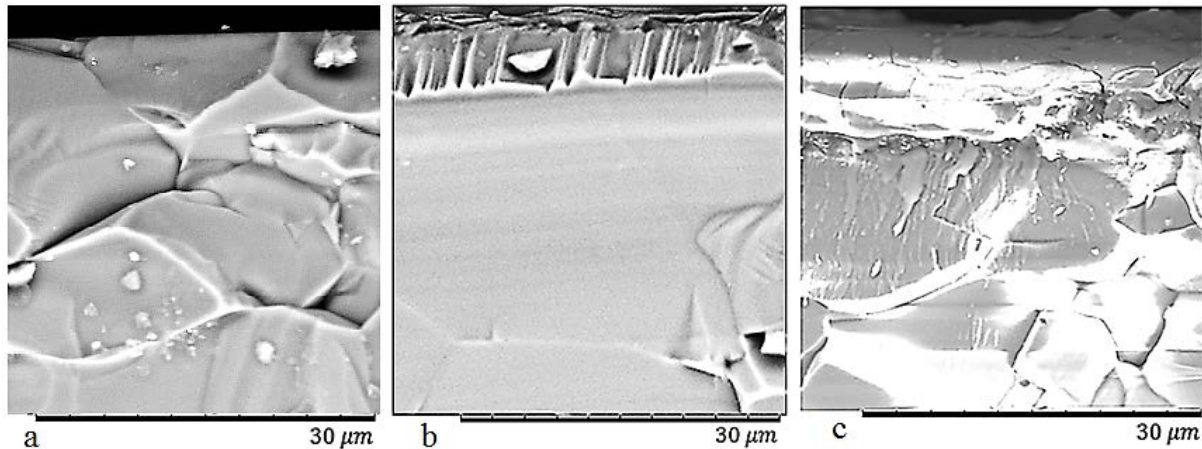


Figure 2. SEM image of the transverse fracture of the ceramics:

- a* – surface treatment of HPPIB ceramics at current density $j = 15 \text{ A/cm}^2$, number of pulses $N = 20$;
- b* – surface treatment of HPPIB ceramics at current density $j = 50 \text{ A/cm}^2$, number of pulses $N = 100$;
- c* – surface treatment of HPPIB ceramics at current density $j = 85 \text{ A/cm}^2$, number of pulses $N = 30$.

The static electrical conductivity of the modified ceramics layers was measured by applying a constant voltage $U_{max} = 5 \text{ V}$ to the electrodes and the sensitive limit of the universal voltmeter V7-21A 100 nA. When measuring the electrical conductivity of corundum, it was found that when the HPPIB ceramics are treatment, the electrical conductivity of the sample remains practically unchanged compared to its initial state. It follows that after treatment by HPPIB, the oxygen content of the material changes insignificantly.

For different ion current densities, curves for the variation of the nanohardness of the samples are plotted against the HPPIB irradiation dose at the maximum applied load P_{max} to the indenter 10 mN (Figure 3a) and 100 mN (Figure 3b). As can be seen from the graphs, the choice of the applied load to the indenter does not affect the character of the change in the curves. From Figure 3 that when the current density of the ion beam is 15 A/cm^2 , the nanohardness increases continuously with the increase in the dose (fluence) (curve 2). The growth of the nanohardness of the near-surface layers is explained by the decrease in the grain size without cracking during the recrystallization. An increase in nanohardness is observed in comparison with the initial value by 22%. With an increase in the current density of the ion beam to 50 A/cm^2 (curve 3) and 85 A/cm^2 (curve 4), the value of nanohardness at the minimum point on the curve H (Q) decreases. Perhaps this is due to the process of cracks formation. As the dose is increased, partial healing of cracks occurs due to melting, which leads to an increase in nanohardness of surface.

Thus, it has been established that the impact by HPPIB carbon leads to a change in the nanohardness of the surface layers of corundum ceramics. The degree of change depends on the chosen modes of irradiation. In the selection of irradiation modes it is possible to achieve hardening of ceramics. This result is confirmed by the data of paper [23].

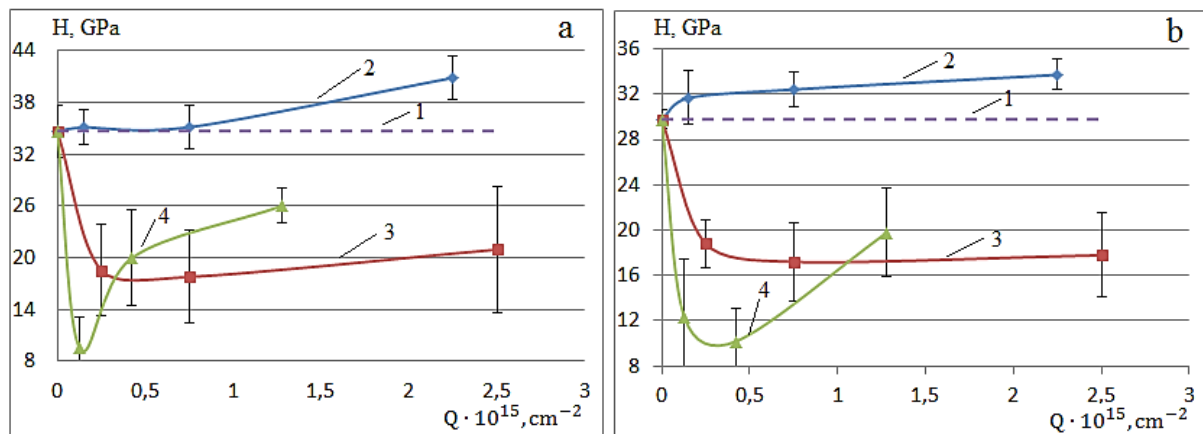


Figure 3. Nanohardness of alumina ceramics as a function of HPPIB treatment dose Q at current densities 15, 50, 85 A/cm² (curves 2-4, respectively. Curve 1 – the value of nanohardness before treatment by HPPIB):

a – is the maximum applied load to the indenter $P_{max} = 10$ mN;

b – is the maximum applied load to the indenter $P_{max} = 100$ mN.

4. Conclusion

The impact of the high-power pulsed ion beam of carbon has been studied on the structure and mechanical properties of corundum ceramics. The following results are obtained.

Under the influence of HPPIB, at a current density of an ion beam of 85 A/cm², a thin near-surface layer melt to a depth of the order of 9-11 μ m. With increasing dose, the thickness of the melting layer remains practically unchanged due to erosion of the surface during ion treatment.

When treatment of HPPIB ceramics, the electrical conductivity of the samples remained practically unchanged from its original value.

The surface treatment of HPPIB corundum with a current density of 50 A/cm² leads to the formation of a "columnar type" microstructure. When treatment ceramics HPPIB, the near-surface layer acquires a block structure with boundaries in the form of cracks, which propagate to the depth of not more than the thickness of the fused layer.

The nanohardness of the corundum ceramics treated with HPPIB depends on the current density and the dose. At current densities on the order of 15 A/cm², the nanohardness of near-surface layers of corundum ceramics increases with increasing radiation dose. The greatest increase in nanohardness is about 20%. At higher ion current densities, nanohardness as a whole decreases and has a minimum at irradiation doses of $2.5 \cdot 10^{14}$ and $1.3 \cdot 10^{14}$ cm⁻², for current densities of 50 and 85 A/cm², respectively.

Acknowledgments

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