

Fracture of the Surface Layers of Alumina Ceramics under the Action of a High-Power Ion Beam of Nanosecond Duration

V. S. Kovivchak^{a,*} and T. V. Panova^a

^aDostoevsky Omsk State University, Omsk, 644077 Russia

*e-mail: kvs_docent@mail.ru

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Abstract—The fracture of surface layers of alumina ceramic (polycor) under the action of a high-power ion beam of nanosecond duration is investigated. The characteristic features of such fracture are determined. An estimation of the thickness of breakaway fragments of the surface layer of the ceramics gives values in the range from 4 to 12 μm at single high-power ion beam irradiation with a current density of 150 A/cm^2 . Oxygen depletion in the surface layer of the ceramics is established. The effect of irradiation on the structural-phase state of the ceramics is considered. The possible mechanisms of the observed fracture are discussed.

Keywords: high-power ion beam, alumina ceramic, surface morphology, fracture, composition

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INTRODUCTION

High-power ion beams (HPIB) of nanosecond duration are one of the promising tools for the high-tech processing of metals and alloys, which provides significant enhancement of the mechanical and chemical properties of the surface layer [1–3]. Compressive mechanical stresses, which can appear at such impact, provide hardening of the surface layer of the material. At the same time, the formation of tensile stresses can lead to the appearance of cracks in the irradiated layer; it is common for different brittle materials [4–6]. The fracture of surface layers under the effect of a HPIB is well studied on sodium-silicate glass which is a brittle model material [7]. It was established that its fracture could be observed a long time (to ~ 170 h) after irradiation. Two kinds of cracks are formed upon irradiation, namely, perpendicular and parallel relative to the irradiated surface. It was shown that such a long fracture time is connected with the existence of residual stresses which appear upon HPIB impact at the surface layer of glass with increased imperfection. Studying the behavior of the surface layers of more complex (than amorphous glass) brittle polycrystalline materials under such irradiation is of both scientific and practical interest. Earlier study of the modification of the properties of the surface layers of aluminum-oxide ceramics under HPIB impact carried out by means of optical microscopy only did not allow the authors to correctly interpret the observed changes in the surface morphology and associate them with the fracture of the surface layer of the ceramics [10].

The aim of this work is to establish the features of fracture of the surface layers of polycrystalline alu-

mina ceramics (polycor) under the influence of a high-power ion beam of nanosecond duration.

EXPERIMENTAL

The samples of alumina ceramics with a size of 15×12 mm and a thickness of 0.5 mm were cut from a standard polycor substrate (60×48 mm). Before irradiation, the surface of the samples was chemically purified, and then the samples were heat treated for 1 h at a temperature of 250°C to remove mechanical stresses after cutting.

Irradiation was carried out using a “Temp” accelerator (Dostoevsky Omsk State University) by a proton-carbon beam (30% H^+ and 70% C^+) with a particle energy of (E) ≈ 200 keV; the duration of the radiation pulse τ was 60 ns in a range of beam-current densities of 50–150 A/cm^2 . The current density of the beam j and the number of radiation pulses n were varied in the experiment. After HPIB impact, the samples were studied using optical (Neophot-2) and scanning electron microscopes (JSM-6610LV, “JEOL”) with an Inca-350 energy-dispersive analyzer. The data of energy-dispersive analysis were averaged over the irradiated surface, and they were interpreted taking into consideration the features of such analysis for polycrystalline materials [11]. A thin layer (~ 10 nm) of platinum was deposited onto the surface of the ceramics with a low conductivity before electron microscopy study. X-ray diffraction analysis of the samples was performed using a DRON-3M diffractometer with CuK_α radiation.

RESULTS AND DISCUSSION

The study of the action a high-power ion beam on polycor in a wide range of beam current densities showed that two types of cracks appear in the surface layer as for sodium-silicate glass: perpendicular (“hairline”) and parallel (“breakaway”) to the irradiated surface. The threshold of the formation of “breakaway” cracks is $\sim 100 \text{ A/cm}^2$ for single irradiation. Upon repeated irradiation, this threshold decreases which is likely associated with the effects of the accumulation of defects and microdamage during each subsequent pulse of radiation. The characteristic size of the fragments of fracture is $\sim 10 \mu\text{m}$ while this value reaches $\sim 50 \mu\text{m}$ for sodium-silicate glass under the same irradiation conditions. Upon studying the cracks by optical microscopy, an interference pattern is formed in reflected light in the surface layer of polycor as it was observed for the glass (Fig. 1a). In most cases, the interference pattern of polycor has the shape of annular bands (Newton’s rings). It indicates a complex bend of the fragment of fracture at which an air gap with variable size forms. Fig. 1b presents a typical image of the irradiated surface obtained by scanning electron microscopy. The characteristic radius of the bend of polycor fragments determined from the radius of interference rings was $\sim 15 \mu\text{m}$; the bending radius of the glass irradiated under similar conditions was $\sim 600 \mu\text{m}$. The multiple effects of a high-power ion beam with the high current density on the polycor can lead to the melting of a thin surface layer of fragments of fracture. The formation of pores localized mainly in the area of “hairline” cracks having increased imperfection is typical for such irradiation regimes (Fig. 2a). At high current densities of the beam ($\geq 150 \text{ A/cm}^2$), repeated irradiation ($n \geq 2$) can lead to the total removal of some fragments of destruction (Fig. 2b). In this case, the relief of the fracture surface is mainly determined by the shape of alumina particles used in the case of ceramics sintering. Since alumina ceramics is a high-temperature material ($T_m = 2015^\circ\text{C}$), reaching such a temperature on the surface layer of fragments can be explained in the following way. At the first pulse, a thin fracture fragment exfoliates from the main part of the sample and an air gap between the part of the fragment and the sample forms which prevents heat dissipation from the fragment to the substrate. Finally, it leads to a significant increase in the temperature on the irradiated surface of this fragment (up to melting) upon subsequent repeated irradiation. According to energy-dispersive analysis, the impact of the high-power ion beam leads to depletion of the surface layer with oxygen. The O/Al ratio (at. %) of the initial (unirradiated) sample is 1.50. For the case of single HPIB impact with a current density of 150 A/cm^2 this ratio decreases to 1.45; when the number of irradiation events reaches three, the O/Al ratio decreases to 1.36. The study of the depth of localization of “breakaway” cracks showed that it varies more significantly than in

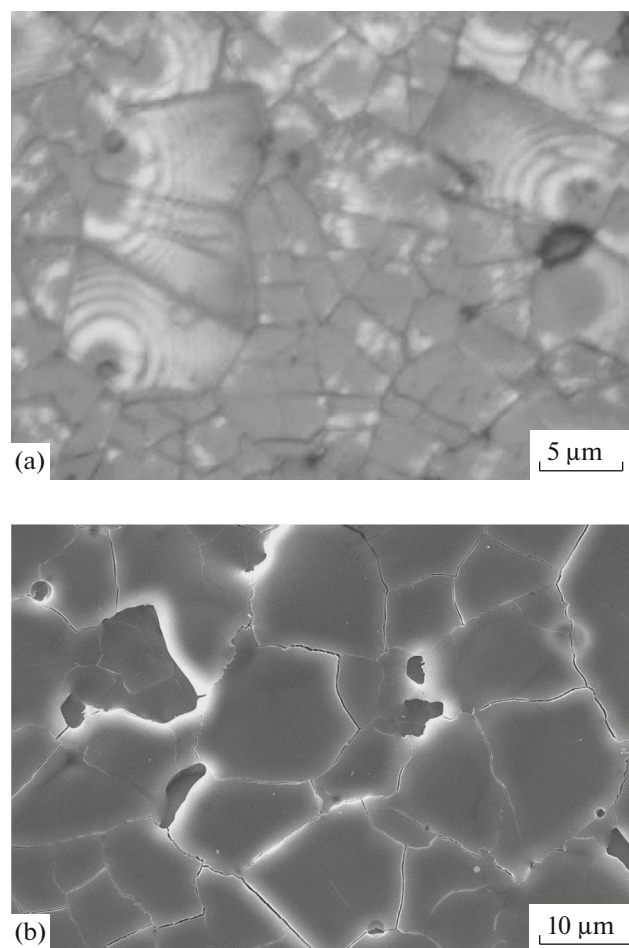


Fig. 1. Surface of polycor after irradiation with a high-power ion beam with a current density of 150 A/cm^2 : optical (a) and scanning electron (b) microscopy.

glass, and its value is $8 \pm 4 \mu\text{m}$. This large scatter is likely caused by the significant inhomogeneity of the mechanical properties of polycor within grains (especially, in the area of boundaries) which is associated with the features of the production of ceramics from alumina powder.

Unlike the sodium-silicate glass, the appearance of new “breakaway” cracks was not observed using optical methods in the surface of polycor after irradiation by a high-power ion beam with current densities from 50 to 150 A/cm^2 . It is possible in this case that the cracks form either during the impact of the ion beam or during the time necessary for the removal of samples from the vacuum chamber of the accelerator. Such behavior is likely connected with the minor demonstration of the elastoplastic properties of polycor in comparison with sodium-silicate glass.

X-ray diffraction (XRD) analysis of the studied materials was performed using a DRON-3M diffractometer with $\text{Cu}_{K\alpha}$ radiation using a β filter. Analysis of the XRD patterns of alumina ceramics (polycor)

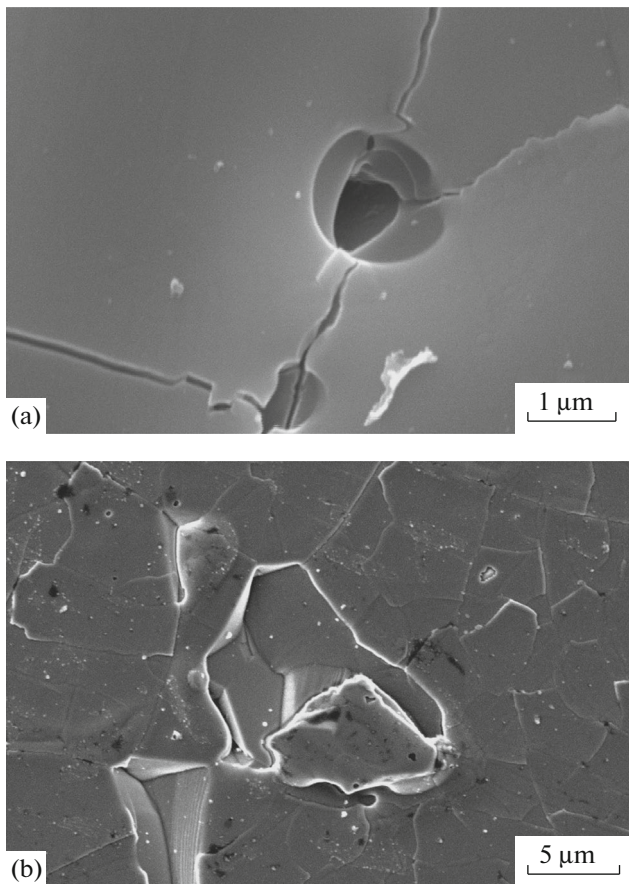


Fig. 2. SEM images of the polycor surface after repeated irradiation with a high-power ion beam with a current density of 150 A/cm²: formed pores (a) and area with removed fracture fragment (b).

showed that in the initial state polycor exists in the α -Al₂O₃ phase with a hexagonal lattice and parameters of $a = 4.758 \text{ \AA}$ and $c = 12.991 \text{ \AA}$ (Fig. 3). After irradiation by a high-power ion beam with a current density of 50 A/cm² by one and three pulses, no changes in the phase state were found but a changing intensity of the reflections at (104), (110), (113), (116), (018), (1010), (134), and (0114) was observed. At a higher number of pulses, the maximal intensity shifted towards lower interplanar distances. The maximum of the intensity in the initial state was observed upon reflection from the (104) planar with $d_{hkl} = 2.55 \text{ \AA}$. After HPIB irradiation with one and three pulses with a current density of 50 A/cm², a higher intensity is observed for the reflections (113) with $d = 2.08 \text{ \AA}$ and (1010) with $d = 1.24 \text{ \AA}$ respectively. The reflections (3012) and (229) disappear when the number of pulses increases.

Upon the action of a high-power ion beam with a current density of 100 A/cm² on polycor, another pattern of a change in the reflection intensity is observed. Thus, in the case of irradiation with one and three pulses the maximal intensity is observed from the

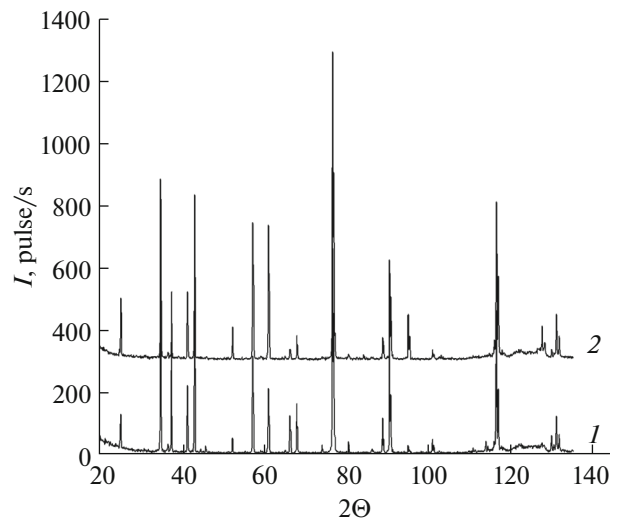


Fig. 3. XRD patterns of polycor in the unirradiated state (1) and when irradiated by three HPIB pulses with a current density of 50 A/cm² (2).

(1010) reflections with $d = 1.24 \text{ \AA}$. However, the intensity of this reflection decreased upon irradiation with three pulses. The reflection from the (1310) plane is enhanced upon irradiation with one pulse and almost disappears in the case of irradiation with three pulses. A slight peak with $d = 3.638 \text{ \AA}$ appears in a low-angle area after irradiation with one pulse which, likely, corresponds to the δ -Al₂O₃ phase. In the case of irradiation with three pulses, this peak disappears.

Upon the irradiation of polycor by a high-power ion beam with a current density of 150 A/cm², no phase restructuring was observed. The pattern of changing intensity upon irradiation with one pulse is similar to that observed upon irradiation with a current density of 100 A/cm² with the exception of the peak of the δ phase in the low-angle range. In the case of irradiation with three pulses, the highest intensity is again observed for the (104) reflection with $d_{hkl} = 2.55 \text{ \AA}$.

Since a phase transition was not found, the intensity changes are likely connected with the rotation of polycrystalline grains upon plastic deformation. Under the influence of stresses caused by HPIB, grain rotation occurs and leads to enhancement of the integrated intensity of reflections from planes which are maximally rotated (with the given irradiation parameters) with respect to the X-ray beam. At high HPIB current densities, fracture of a thin surface layer can also cause a change in the intensity.

The method of approximation by function was used to calculate the residual stresses; high-purity aluminum was used as the standard. Analysis showed that the diffraction maxima are well approximated by a Gaussian function. It was found from the calculations that the sum of the main stresses $\sigma_1 + \sigma_2$ in polycor is maximal in the case of HPIB irradiation by one pulse

with a current density of the ion beam of 150 A/cm², and its value is ~3 GPa. The minimal value is observed after irradiation with three pulses with a current density of 150 A/cm² and its value reaches 0.19 GPa. Such a decrease in the value of the residual stresses is possibly connected with their relaxation upon the formation of cracks. In the case of all irradiation modes tensile stresses are formed. Compared with the initial size, the crystallite size decreases upon irradiation with a small current density and then increases 1.6 times and reaches a maximum upon irradiation by three pulses with a current density of 100 A/cm². In the case of irradiation by three pulses with a current density of 150 A/cm², the size of crystallites decreases 1.5 times compared with the initial one. Apparently, it is connected with growth of the temperature gradient which leads to an increase in internal stresses. The density of dislocations increases significantly upon HPIB irradiation. In the case of irradiation with three pulses with a current density of 50 A/cm², the dislocation density is maximal (14 times higher than the initial one). The minimum of the dislocation density is observed in the case of irradiation with three pulses with a current density of 100 A/cm² which is associated with the annealing of defects.

CONCLUSIONS

Thus, it was established that the single impact of a high-power ion beam with a current density of no less than 100 A/cm² on alumina ceramics (polycor) could lead to the formation of cracks in the surface layer perpendicular and parallel to the irradiation surface. Chipped fracture fragments have a thickness from 4 to 12 μm. In general, the fracture pattern is similar to that for sodium-silicate glass under the influence of a high-power ion beam. However, no emerging fracture was detected on this ceramic after the irradiation pulse as was observed on sodium-silicate glass. X-ray diffraction analysis of the irradiated ceramics showed that no phase changes occur upon irradiation of this material with an ion beam. The obtained data indicate the general regularities of the process of fracture of solids

under the influence of a high-power ion beam, regardless of the structural state of the material (polycrystalline or amorphous).

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