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Damage resistance of corundum treated with abrasive and contact-free processing

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The planishing of the surface of solids is aimed to minimize its roughness, downgrade a content of larger cracks in the modified layer, and, in general, reduce a thickness of the latter one. However, the abrasive processing induces residual stresses in the surface layer. The stresses relax through the motion of dislocations but in superhard materials like corundum or silicon dioxide, is extremely limited. In the present work, the role of the modified surface layer in corundum was studied within the context of the method and regime choice which affect the surface processing the mechanical characteristics of corundum. An efficiency of the abrasive treatment was assessed by comparison with the properties of surfaces processed with the ion polishing, sheared surface, and natural face of crystal from the viewpoint of reaching the high mechanical damage resistance of the surface.

Keywords: corundum, surface treatment, microhardness, impact loading

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Introduction

Corundum (leucosaphireα-Al_2O_3) single-crystals have rhombohedral structure and are the only stable phase state of aluminum oxide [1]. Corundum is second only to diamond in strength and has the highest resistance to scratching, particularly abrasion, of any oxide. Attractive mechanical properties are combined with the material's high transparency in the vacuum UV to mid-IR range. The favorable combination of strength and optical properties makesα-Al_2O_3 crystals suitable for many applications where transparent protection of various devices — from outdoor aircraft instruments to hand watches and telephone displays — against environmental contact/abrasion is required.

The downside of the aluminum oxide strength is its high brittleness and ability to fracture virtually without plastic deformation, because the sufficiently high bond energy and activation energy of dislocation motion prevent cold deformation [2]. When a load is applied to the product, the fracture center develops in the modified surface layer, the parameters of which (thickness, cracking, relief) depend on the method/mode of surface preparation. A second factor affecting the fracture pattern may be the rate or duration of mechanical action.

In the present paper, corundum crystal surfaces both prepared by polishing with diamond powder and obtained without the use of abrasives were tested for Vickers microhardness and subjected to point impact fracture. In the latter case, the kinetics of microcrack accumulation was monitored by acoustic emission (AE) and electromagnetic emission (EME) methods.

1. Samples and equipment

α-Al_2O_3crystals were grown by a modified Kiropoulos method (GOI [3,4] method). The initial surfaces (before processing) served as growth facets, which in this method are located normal to the main axis of the crystal. The surfaces of the natural faces were polished with diamond powders with a stepwise reduction of the abrasive grain size. Each transition to the next grain size took place after the relief created by the preceding grain was completely removed. In addition, surfaces obtained by non-abrasive techniques of relief formation, including ion polishing (irradiation withAr^+), ions), chipping surface, and intact growth facet, were tested.

Impact damage to the sample surface was produced by a pointed hardened striker mounted on it, on which a 100 g weight was dropped from a height of 70 cm. The acoustic signal was recorded by a paraffin-embedded sensor made of highly sensitive PZT piezoceramic; the electromagnetic signal was recorded by a Hertz dipole. AE and EME pulses were recorded in the range of hundreds of kilohertz through an analogue-to-digital converter and fed into a computer. To cut off the low-frequency vibrations of the rig during striker impact, the AE signal was digitally discriminated at 80 kHz.

2. Results and discussion

2.1. Microhardness

The microhardness of the samples (except for the chipping with a non-planar surface) was determined by Vickers pyramid embedding with an exposure time of 10 s under a load of 1 N. The time from the beginning of

Vickers hardness depending on surface treatment method

Designation sample	Method processing	Grain size abrasives, d, μm	Note	Hardness. Vickers *, H_V, GPa
A	Polishing by abrasive	40/28	Diamond powder	11.3 ± 0.7
B	To same	1/0	To same	9.4 ± 0.5
C	To same	0.5/0	To same	7.4 ± 0.3
D	Ion ion		Bombardmen polishingAr^+	18.2 ± 0.7
E	Growth facets			19.1 ± 0.7

Note. * — tabulated value of the H_V value for corundum — 23.5 GPa.

the load application to reaching the nominal load value was 1.8 ± 0.2 s. The table shows the calculated values of hardness (H_V). The measurements showed that in the group of samples with abrasive surface machining, the hardness decreased with the reduction in diamond grain size (d). The effect is explained as follows.

Abrasive surface polishing carried out to make products transparent results in a disturbed surface layer consisting of disordered microcrystals. In leuco-sapphire and other super-hard materials with restricted dislocation movement, abrasive particles pressurized by the polishing machine create compressive stresses on the surface [5,6] that do not relax due to the lack of plasticity. Below the compressed layer, there is a fractured layer with tensile stresses at the tips of microcracks [7]. This layer determines the low surface hardness of the abrasive-polished corundum, which does not reach even half of its tabulated value (samples A, B, C in table).

The compressed layer is evident in microhardness measurements because it prevents the indenter from embedding to some extent. The thickness (D) of the modified layer depends on the grain size d according to the approximate ratio $D \cong 1.5 \cdot d$ (for sapphire [8]). As the abrasive grain size decreases, the compacted layer becomes thinner, and the Vickers hardness decreases as shown by measurements (samples A, B, C).

The surfaces of the samples prepared without abrasive showed microhardness close to the tabulated value for corundum. Sample D was polished with an Ar^+ ion beam (ion energy 20 keV;$3 \cdot 10^{15}$ ion/cm^2), which did not create microcracks in the surface layer. In addition, upon ion bombardment of the dielectric sample, the implanted atoms modify the surface topography [9], creating a spatial wave-like structure of submicroscopic scale [10–13]. This regular structure effectively dissipates mechanical energy. This technique has been used to increase the surface strength of α-Al_2O_3 [6,9] crystals.

The natural crystal growth facet (sample E), which does not have a fractured layer, showed the highest microhardness. In addition, the facet surface is formed by flat steps with elevations of microscopic scale, which can hinder the indenter insertion, like the aforementioned microrelief of the Ar^+ion-irradiated surface.

2.2. Impact damage

The modified disordered layer was evident in the morphology of surface damage created by the impact of a pointed striker. Fig. 1 shows optical photographs of the made craters in images with different prehistory. Damage to samples polished with 40/28 and 1/0 μm abrasives had a character typical of plastic materials (Fig. 1, *a, b*) due to the presence of a disordered, abrasive-damaged surface layer. The reduction of abrasive grains to 0.5/0 μm resulted in a partial change in the damage morphology: the cavern has the appearance of brittle fracture features (Fig. 1, *c*) due to a too thin transition layer.

In photographs of damage without an abrasive-modified layer, i.e. applied with a striker to an ion-polished surface (Fig. 1, *d*), to a natural growth face (Fig. 1, *e*), to a chip (Fig. 1, *f*), all damage has a typical appearance of brittle fracture.

The presence of the polishing-formed modified surface layer of corundum was also evident in the emission activity excited by point impact in the samples. Fig. 2 shows the time sweeps of acoustic and electromagnetic signals, which clearly show the delay of the EME generation peak relative to the AE peak. The interval between the peaks in the abrasive polished samples and samples obtained by non-contact surface forming methods were ~ 1 and ~ 2 ms, respectively

2.3. Discussion

The source of acoustic signals during mechanical action on a solid body is the deformation of stressed material or its destruction with the formation of cracks. The AE signal frequency is inversely proportional to the crack size. In our case of point impact damage to a surface, a frequency in the range of hundreds of kHz corresponds to the generation of cracks in the microscopic range. The extent of sound

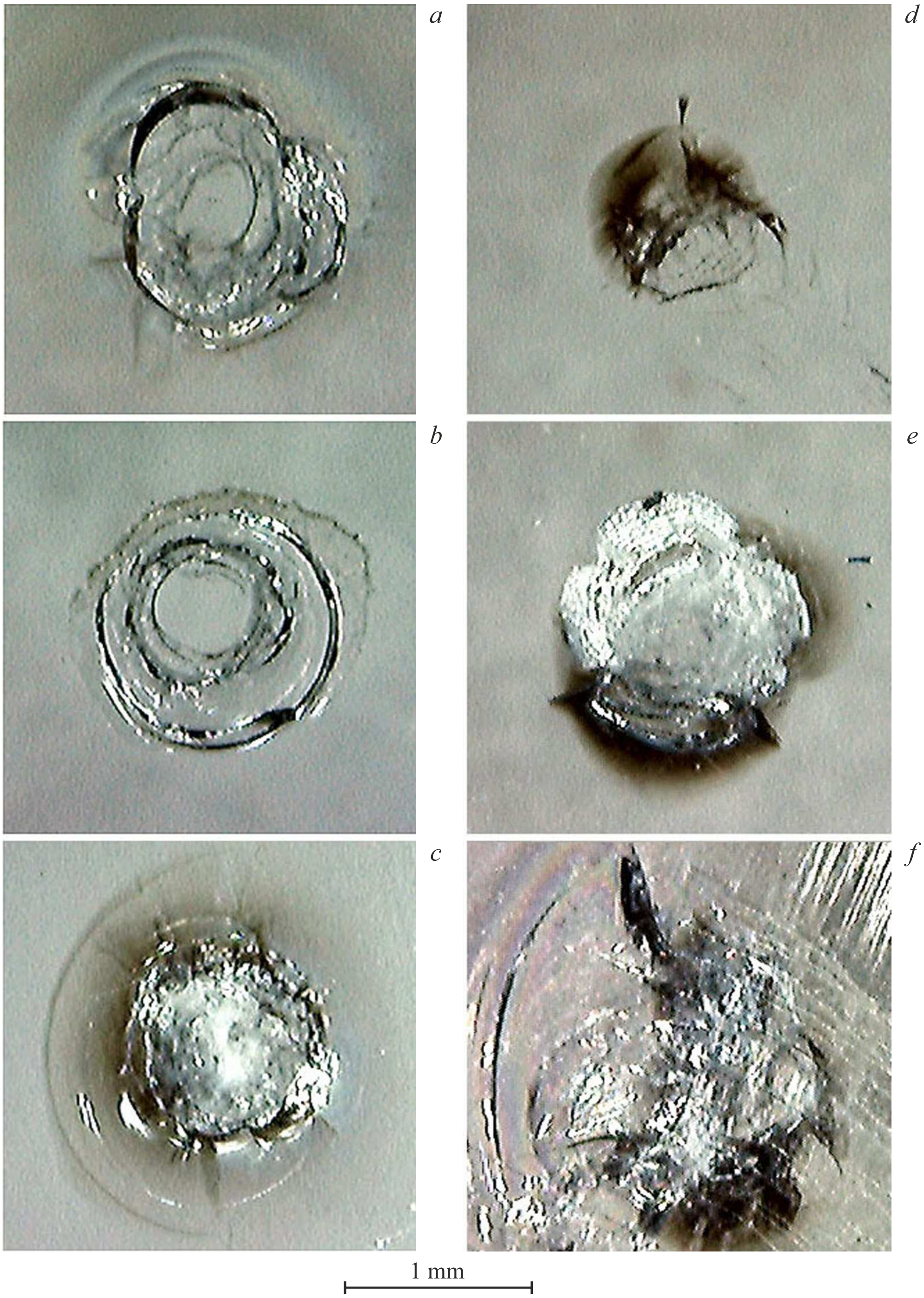

Figure 1. Optical photographs of damage caused by a sharpened striker. Left column: samples polished with diamond powder with grain size 40/28 (*a*); 1/0 (*b*); 0.5/0 (*c*) μm. Right column: ion polishing (*d*); growth facet (*e*); chipping surface (*f*). Crater formation time — 1−2 ms.

emission in all samples was about 1 ms with a peak in the area 400−600 μs.

Electromagnetic radiation during impact on a solid body results from a two-stage process. When microcracks nucleate and develop, electric charges of opposite signs are formed on their banks. After the shock wave passes through, the cracks close and charge annihilation occurs with the emission of electromagnetic waves [14]. Thus, the EME signal is always delayed after the acoustic signal by a time equal to the open crack lifetime. In the present paper, this time was estimated from the interval between maxima in the AE and EME sweeps. As it turned out, the interval was about half as short in samples polished with abrasives compared to samples prepared by techniques that do not leave stresses in the surface layers. Furthermore, in the latter, the duration of both types of emission activity after

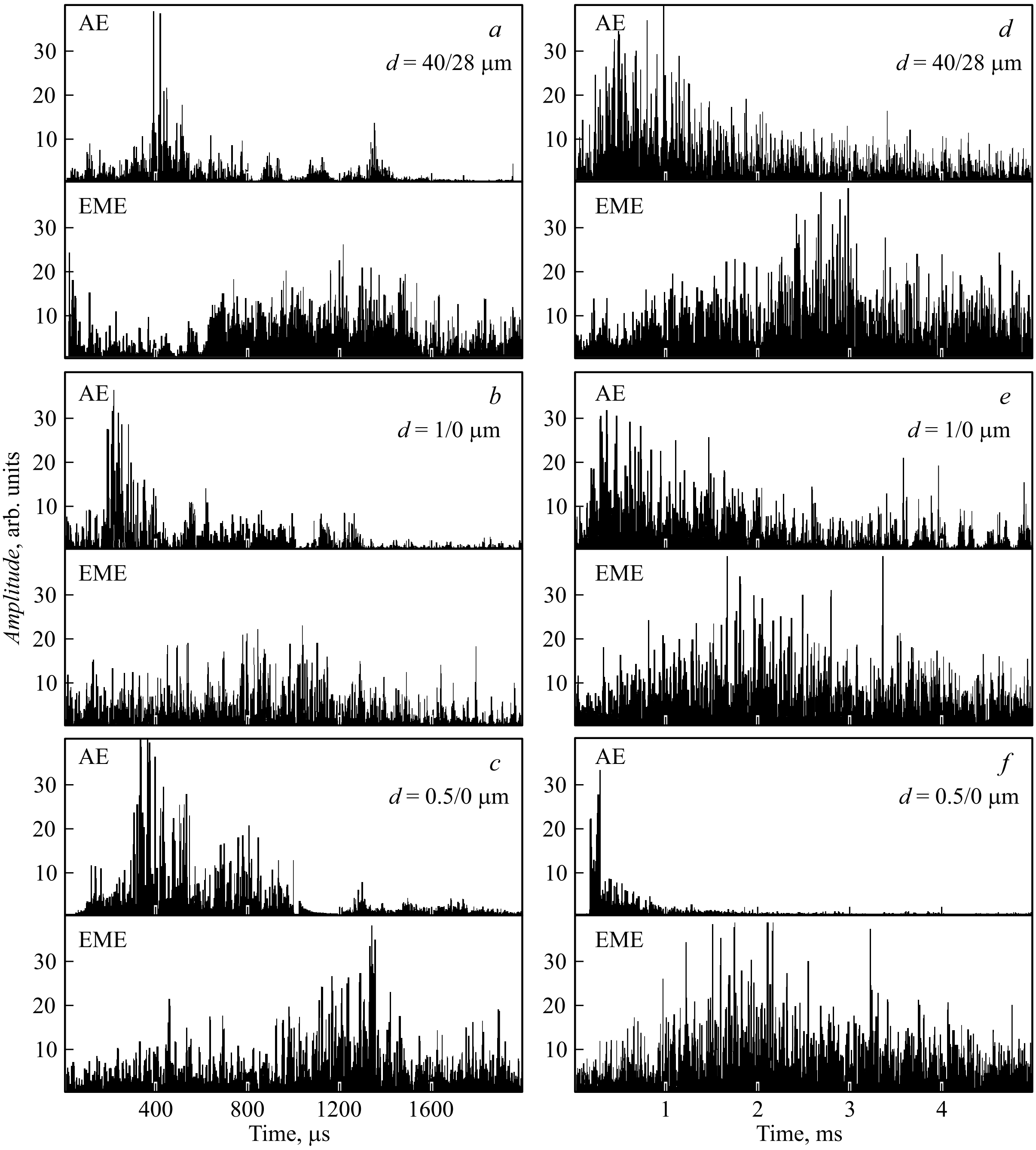

Figure 2. Time sweeps of AE and EME pulses during impact on the surface after abrasive machining ($a-c$), ion polishing (d), growth facet (e) and chipping (f).

impact was much longer than in the samples obtained by abrasive polishing (at least $5\,\mu$s vs.$1-2\,\mu$s).

Finally, the surface microhardness of the mechanically polished samples was far from the tabulated values, while at the growth facet and ion-irradiated surface, the value $H_V$approached these values.

All these differences can be attributed to the emergence under the mechanical action of abrasive grains binary system [5]: a compacted layer and underlying disordered layer [1], which is sometimes conventionally called „amorphized film" [15], although it consists of small crystalline fragments connected by weak bridges [8]. This polishing-modified layer has a high mobility of fragments that facilitate multiple microcrack accumulation when the surface is impacted.

There is no disordered layer on the surface of growth, chipped and ion beam-treated corundum, so it takes longer time for the impact energy to scatter, and microcracks created under the action of the striker have a longer lifetime. Accordingly, the EME activity during monolithic crystal fracture lasts longer and the interval between emission activity peaks increases.

3. Conclusion

The surface of corundum single-crystals was polished with diamond powder and prepared by non-abrasive methods — ion polishing, chipping, and forming a natural growth facet. The surface was subjected to point impact damage in which the kinetics of microcrack accumulation and relaxation were monitored by AE and EME methods, respectively. When microcracks appear, electric charges of opposite signs are formed on their banks, which annihilate after relaxation of open cracks with emission of electromagnetic waves. The interval between the AE and EME peaks characterizes the lifetime of open microcracks after impact, which was half as short in samples polished with abrasives compared to samples prepared using techniques that do not leave stresses in the surface layers.

The Vickers hardness measurements showed a reduced microhardness of the samples obtained by abrasive (diamond) polishing (less than half of the tabulated hardness of the crystal), while for the growth surface and ion beam-treated corundum, values close to the same were obtained. The difference between the above mechanical characteristics of the surfaces of samples polished by diamond polishing and prepared by non-abrasive methods is explained by the presence in the former case of an abrasive-induced disordered layer consisting of bound small crystallites, in which microcracks are easily formed upon impact on the surface. Non-contact methods shape the surface topography but hardly reduce the resistance to impact loading.

Conflict of interest

The authors declare that they have no conflict of interest.

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