NANOINDENTATION STUDY OF COARSE GRAIN ALUMINA

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Abstract
The nanoscale characteristics of the coarse grain alumina were studied by various nanoindentation loads with a Berkovich indenter. A significant indentation size effect (ISE) was found. Maximum hardness in the ISE region was 35 ± 3 GPa, in the non-ISE region it was 27 ± 1.5 GPa. The indentation modulus was 410 ± 15 GPa in the ISE region and 257 ± 12 GPa in the non-ISE region. The hardness of basal plane was 31.2 ± 0.8 GPa and prismatic plane was 26.9 ± 1.2 GPa. The indentation modulus was 420 ± 11 GPa for basal planes and 385 ± 13 GPa for prismatic ones.

Keywords: nanoindentation, alumina, hardness, modulus, basal and prismatic orientation

INTRODUCTION
Alumina, the most well known structural ceramic finds applications such as in the wear-resistant inserts, biomedical implants, high strain rate impact-resistant plates, high temperature electronic components, very high end optical components and devices. Hardness is one of the most important surface mechanical properties of alumina ceramics. Grain boundaries have a significant influence on important sintering processes such as grain growth, creep and mechanical properties [1-4].

Alumina can exist in several crystalline phases which all revert to the most stable hexagonal alpha phase at elevated temperatures. A sapphire, a single crystal form of pure aluminium oxide, is a leading candidate material for many extreme applications. Sapphire is most commonly manufactured in basal and prismatic surface orientation [5]. Hardness exhibits anisotropic behaviour due to different crystallographic orientations of such crystals. Apart from that, indentation size effect (ISE) of hardness, i.e. varying measured values independently of the applied load, is also typically found for this material [6-7].

The aim of the present work is a precise characterization of ISE and accurate measurement of values of indentation hardness and elastic modulus for basal and prismatic planes of coarse grained alumina.

EXPERIMENTAL MATERIAL AND METHOD
Alumina was supplied by a private company without the specification of processing parameters. It is a transparent material in a tube shape with length 4 cm, diameter 9 mm and wall thickness 1.5 mm (see Fig.1a). The microstructure of the material is illustrated in Fig.1b. It has a regular shaped large alumina grains with typical size 50 μm.
Instrumented indentation with Berkovich indenter was used for nanoindentation hardness testing carried out on Nanoindenter NHT, by CSM Instruments. Single loading-unloading regime was used in all tests. In order to observe the ISE indentations load ranges from 5 up to 500 mN were performed. In these experiments the anisotropy of values due to the crystallographic orientation was neglected and the results were statistically treated in the usual way. For precise measurements of crystal anisotropy, indentation with maximum load of 25 mN and a 15x15 grid were used. Here, the loading/unloading rate 50 mN/min and holding time of 10 s at the maximum load were implemented. Indented grains were carefully chosen so that their orientation was as close to a specific (basal or prismatic) orientation as possible. This was established by comparing it with the results of authors [1,5].

RESULTS

Strong indentation size effects of hardness and indentation modulus were found, see in Fig.2. The statistical distributions of the results are illustrated in Figs.3 and 4. The indentation hardness and modulus have been automatically calculated using the Oliver&Pharr method. The values of hardness and indentation modulus are tightly clustered. Occasional values with a great deviation correspond to the irregularities of measurement. The averaged load-depth curves of indents made in respective orientations are illustrated in Fig.5.
Fig. 2. Indentation size effect.

Fig. 3. Relationship between depth and hardness.
Fig. 4. Relationship between depth and indentation modulus.

Fig. 5. Averaged load-depth curves for indents made by 25 mN load on differently oriented (basal or prismatic orientation) grains.
DISCUSSION

Determination of ISE on material studied. In the investigated load range 5 – 500 mN the maximum hardness value was 35 ± 3 GPa, the hardness at high loads (> 200 mN), where the ISE was no longer observed, was 27 ± 1.5 GPa, Fig.2. These results agree qualitatively with the literature data by Mao et al [6]. They analyzed the ISE of hardness and indentation modulus for similar material and found that the maximum hardness is 46.7 ± 15 GPa at the ISE region and 27.5 ± 2 GPa at the non-ISE region. The absolute values they reported were, however, different due to the indentation load range they used, which was 0.8 – 8 mN.

The second aim was to determine anisotropy of values of the hardness and indentation modulus for basal and prismatic planes, respectively. According to Haney and Subhash [5] the basal planes are harder than the prismatic ones. The hardness is 17.4 GPa for basal and 15.6 GPa for prismatic planes. The indentation elastic modulus is 435 GPa and 386 GPa, respectively. These measurements were performed at load 10 N [5]. We also found that the values of hardness and indentation modulus for basal planes were greater than prismatic ones. The hardness of basal plane was 31.2 ± 0.8 GPa and prismatic plane was 26.9 ± 1.2 GPa and indentation modulus was 420 ± 11 GPa for basal and 385 ± 13 GPa for prismatic planes. The values between these regions are connected with “mix-mode” indents. In this case, the indent is not in the grain with typically basal and prismatic planes, but is in the interface between grains.

CONCLUSIONS

Significant indentation size effect (ISE) of the alumina material was identified. The maximum hardness at the ISE region was about 29% greater than that outside the ISE region. Similarly, the difference in indentation modulus values was 59%. The results show higher hardness for the basal planes than that for the prismatic ones. The hardness of basal planes was 14% greater than that of prismatic ones. In the case of the indentation modulus the difference between basal and prismatic planes was only 9%.

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