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New observations in micro-pop-in issues in nanoindentation of coarse grain alumina

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Abstract

The present experiments were focused on nanoindentation behaviour and the attendant "micro-pop-in" in a dense (~95% of theoretical) coarse-grain (~20 μm) alumina ceramic as a function of loading rate variations at three constant peak loads in the range of 10^5–10^6 μN. Based on the experimental results here we report for the first time, to the best of our knowledge, an increase in intrinsic nano scale contact resistance as well as the nanohardness with the loading rate. These observations were explained in terms of the correlation between the nanoscale plasticity and shear stress active just underneath the nanoindenter.

Keywords: Alumina; Contact deformation rate; Nanohardness; Nanoindentation

1. Introduction

Alumina, the most well known structural ceramic finds applications such as 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 in this connection as it defines the intrinsic contact resistance of alumina ceramics. The defects that ultimately define the mechanical integrity of a structural ceramic originate at the nanoscale of the microstructure during its service life time. Thus, the mechanical integrity of a structural ceramic like alumina in service gets determined at the nanoscale of microstructure. Therefore, the nanoscale hardness or nanohardness of alumina measured at the nanoscale of the microstructure assumes explicit importance in this regard. In spite of the wealth of literature, however, the studies on nanohardness of dense, coarse grain alumina ceramics which characteristically exhibits an R-curve behaviour are far from significant. In recent times the nanoindentation technique has emerged as a very powerful technique to characterize the nanomechanical properties of a wide variety of materials [1–5]. That is why there exists a wealth of literature on hardness of alumina measured at macro- or micro-scale [6–8] as well as at the nanoscale [9,10]. However, these measurements [7–10] are reported mostly for fine grain and/ or submicrometer grain sized alumina ceramics and reports on coarse grain alumina ceramics are really rare [6]. Interestingly alumina is capable of localized plastic deformation even under ultra-low loads used in the nanoindentation experiments [11]. The pop-in mechanisms in nanoindentation experiments are yet to be well understood [12–16]. A lot of factors can affect the initiation of pop-in e.g. nanoindentation load, nanoindenter tip radius, temperature etc., while strain and loading rate variation may [12–15] or may not [16] influence the measured value of hardness. Recently, our work demonstrated significant effect of loading rate on nanohardness of glass [17–22] and alumina [21,22]. The occurrence of the pop-in behaviour were also reported for glass [17–20], polycrystalline alumina [21,22], bulk metallic glass [23–25], sapphire [26], GaN [27] and ZnO [28]. The occurrence of pop-in has often been associated with shear

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localization [17–33]. However, there are many contradictory view points about their genesis in a wide variety of materials [33–38] and an unequivocal picture is yet to emerge. Therefore, the major objective of the present work was to study in detail the role of *"micro-pop-in"* issues in influencing the loading rate dependence of the nanoindentation response and in particular, the nanoscale contact deformation resistance of a high density (~95% of theoretical) coarse grain (~20 μm) alumina ceramic.

2. Materials and methods

Alumina discs of ~10 mm diameter and ~3 mm thickness were prepared by pressureless sintering in air at a temperature of 1600 °C. The density of the sintered alumina discs was measured by Archimedes's principle. The polished alumina discs had surface roughness (R_a) of 0.01 μm. The polished alumina discs were thermally etched for 1 h. in air at a temperature of 1500 °C. The photomicrographs of the thermally etched alumina discs were taken using a Field Emission Scanning Electron Microscope (FE-SEM, Supra VP35, Carl Zeiss, Germany). These photomicrographs were used to measure the average grain size of the pressureless sintered alumina by an image analyzer (Q500MC, Leica, UK).

The load controlled nanoindentation technique was used in a commercial nanoindentation machine (Fischerscope H100-XYp; Fischer, Switzerland) to evaluate the nanohardness and Young's modulus of the alumina samples. A 5×5 array matrix was utilized for this purpose. The machine had depth sensing resolution of 1 nm. The load sensing resolution of the machine was 0.2 μN. The nanoindentation experiments were conducted with a Berkovich indenter attached to the machine. The indenter had a tip radius of ~150 nm and a semi-apex angle of 65.3°. The area function of the indenter tip was evaluated prior to each experiment. The dedicated software available in the control system of the machine corrected the experimentally obtained load (P) versus depth of penetration (h) data for tip blunting effect. Following the DIN 50359-1 standard the machine was finally calibrated with nanoindentation based independent evaluation of nanohardness, H (4.14 GPa) and Young's modulus, E (84.6 GPa) values of a reference BK7 glass block (Schott, Germany). The standard reference glass block was provided by the supplier of the machine. The calibration was repeated before each and every experiment to make sure that the data generated remains reproducible. Next, the Oliver-Pharr model [33] was used to measure the nanohardness and Young's modulus data of the alumina samples from the experimentally measured load-depth (P-h) data plots. No particular bias was associated with the location selection for the positions of the nanoindentation arrays. Thus, at least 25 individual measurements of nanohardness values were used for each reported average data. In the present experiments, three different constant peak loads of 10^5, 5×10^5 and 10^6 μN were used. Further, both the loading and the unloading times were varied from 10^0 to 10^3 s to obtain the variation of loading rates in the range of 10^3–10^6 μN s^{-1}. Thus, the loading rate was calculated by dividing the peak load by the time to reach the peak load. For instance a loading time of 1 s to reach a peak load of 10^3 μN gives the loading rate of 10^3 μN s^{-1}. The error bars represent ±1 standard deviation of the data for all experimental data reported in this work. The Field Emission Scanning Electron Microscope (FE-SEM, Supra VP35, Carl Zeiss, Germany) as mentioned earlier was also used to observe the microstructure and the nanoindents in the present alumina samples.

3. Results

The two most relevant experimental data of major concern in the present work are the instantaneous depth (h) and the reduced depth (h') of penetrations recorded during the nanoindentation experiments. Here $h'\left(=h-h_f\right)$ is called the reduced depth of penetration following [33]. The quantity h_f represents the final depth of penetration. The experimental data on load (P) versus depth of penetration (h) are shown in Fig. 1a. The exploded view of the same data is shown in Fig. 1b. These data confirmed that a large number of serrations occurred during the loading and unloading cycles. The very presence of these

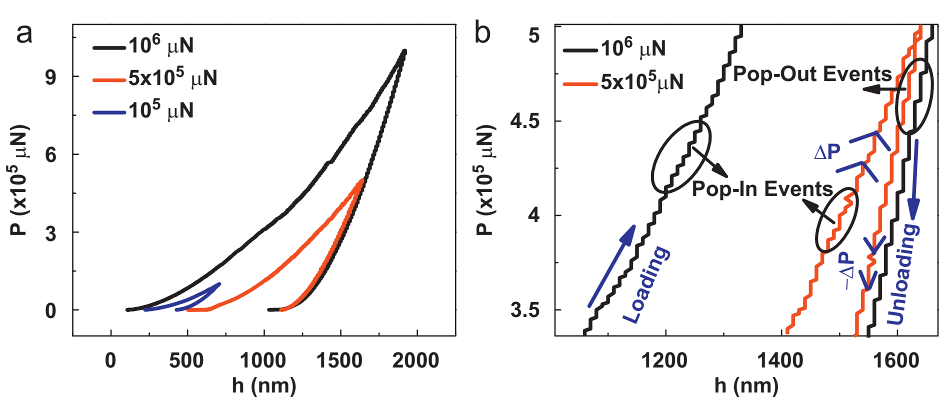

Fig. 1. (a) Load-depth plots at 3 different loads of 10^5 μN, 5×10^5 μN and 10^6 μN (b) Exploded views of Fig. 1(a).