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Deformation and failure of alumina under high strain rate compressive loading

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Abstract

For development of structural ceramics e.g., alumina for high strain rate resistant applications, it is extremely important to understand the issues involved in compressive microfracture and the role of compressive microfracture in the global failure process at high strain rates. Thus the present work reports compressive strength of a dense (e.g., $\rho \sim 97.2\% \rho_{th}$) 5 µm grain size alumina exposed to high strain rate (e.g., $0.9 \times 10^3 \text{ s}^{-1}$) loading in SHPB experiments. Concomitant utilization of high-speed videography has been exploited to study in-situ the details of the dynamic fragmentation process. The maximum compressive strength is measured to be ~3 GPa. Post-mortem examination of the recovered alumina fragments has been performed by FESEM and TEM. Apart from conventional global brittle fracture, the results show the grain localized microcleavages, intragranular microcracking, plasticity, dislocations as well as formation of subgrain structure to occur in alumina. Based on these experimental results the reasons of compressive microfracture and its probable role in the global failure process of alumina ceramic are discussed.

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1. Introduction

Particularly during the recent period there has been a tremendous resurgence in research on structural ceramics e.g., polycrystalline alumina for high strain rate resistant applications involving both strategic and non-strategic sectors [1–6]. In spite of the wealth of literature [1–6], however, many issues in compressive failure of alumina especially at high strain rates [7–11] remain yet to be resolved. Firstly, it is to be known with certainty, how microcracks grow under compression [12–15]. Secondly, it is not yet clear, how the omnipresent processing flaws play a role in the initiation, incubation and growth of the microcracks in the states of uniaxial and/or multi-axial

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compressive loading [12,13]. Thirdly, the extent of interaction between these omnipresent processing flaws and the compressive loading induced cracks is far from well understood. Also, it is yet to be clearly deciphered the amount of interaction that depends on processing history and schedule and the resultant grain size, grain boundary phase composition, etc. of a given ceramic e.g., alumina [12–14].

It is now well known that the compressive strength (σ_c) of both sintered and hot-pressed [12,13] as well as hot pressed and successively hot-isostatically pressed (HPed plus HIPed) [14] alumina ceramics, beyond the critical strain rate ($d\epsilon/dt$), is sensitive to variations in strain rate. At ($d\epsilon/dt$) $\leq 10^2 \text{ s}^{-1}$ the subcritical growth of axial microcrack [12,13] causes the strain rate sensitivity of compressive strength to occur. However, for ($d\epsilon/dt$) $\geq 10^3 \text{ s}^{-1}$ the crack inertia controls the strength [12–15]. A closer look into the typical reported high strain rate

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Fig. 1. Compressive strength as a function of strain rate, obtained from SHPB tests, for alumina ceramics of different grain sizes (S: sintered, HP: hot pressed, HIP: hot isostatically pressed).

data [12–15] on alumina ceramics (Fig. 1) depict a few more of interesting observations.

For instance at lower strain rate, e.g., 10° s⁻¹ pressureless sintered Cervar alumina, having a larger grain size attains a compressive strength of 3 GPa. In contrast, the hot-pressed JS II alumina of much smaller grain size e.g., 3.9 µm exhibits a nearly similar compressive strength of \sim 3–4 GPa [13]. However, HPed plus HIPed JS I alumina of grain size 1.45 µm achieves a compressive strength up to 6-8 GPa at about the same strain rate [13]. Similarly, HPed plus HIPed MTUalumina of 1.5 µm grain size displays a compressive strength of as high as 6–7 GPa [14]. Therefore, at strain rate of 10° s⁻¹ the magnitudes of dynamic compressive strength of alumina ceramics are strongly sensitive function of both grain size and processing methodology. At a relatively higher strain rate e.g., 10^3 s^{-1} , pressureless sintered alumina of grain size 25 μ m exhibits a compressive strength of about 3–4 GPa [12]. Even a reduction of the grain size to 17 µm fails to achieve a compressive strength better than about 4.8 GPa [13]. In contrast, under appropriate confinement the same alumina [13] attains a compressive strength of 8 GPa at a higher strain rate of 10^4 s^{-1} . On the other hand in flyer plate experiments conducted at a similar strain rate e.g., 10^4 s^{-1} pressureless sintered alumina of 25 µm grain size exhibits a compressive strength of about 5.8 GPa [12]. However, at a strain rate slightly higher than 10^3 s^{-1} HPed plus HIPed fine grain alumina of 1.45-1.5 µm grain size displays an even higher compressive strength of 8-8.5 GPa [13,14]. A slight enhancement of grain size to 3.9 µm [13] drastically reduces the compressive strength to about 5.8 GPa at a similar strain rate of 10^3 s^{-1} . Therefore, the data from literature [12–14] as plotted in Fig. 1 apparently indicates that at strain rates higher than 10^3 s^{-1} the magnitudes of compressive strength of alumina ceramics are sensitive to grain size, processing methodology and experimental conditions. It can be also noted that experimental data are either of grain size smaller than $4 \,\mu\text{m}$ (fine grain) or larger than 17 μm (coarse grain), and strain rates less than 10° s⁻¹ (low strain rate) or greater than 10^{3} s⁻¹ (high strain rate).

Therefore, objective of the present work is to study the compressive failure mechanisms of a dense (e.g., $\rho \sim 97.2\% \rho_{th}$) 5 µm grain size alumina at a high strain rate of $0.9 \times 10^3 \text{ s}^{-1}$ in split Hopkinson pressure bar (SHPB) experiments. In addition, in-situ high speed videography of the damage initiation, incubation and growth processes relevant to compressive failure is recorded. In order to develop a comprehensive picture of the corresponding failure mechanisms, these in-situ observations are coupled with complimentary post-mortem analysis of alumina fragments after failure by field emission scanning electron microscopy (FESEM) and transmission electron microscopy (TEM).

2. Materials and methods

Pressureless sintered alumina disks of 6.00 + 0.05 mmdiameter (d) and 3.00 + 0.03 mm thicknesses (l_s) have been used for the SHPB experiments. A high purity (99.7%) commercial variety (CT3000SDP, ALMATIS, Germany) alumina powder is utilized to prepare the disks for this purpose. The powder possesses a sub-micron average particle size e.g., $d_{50} \sim 0.6 \,\mu\text{m}$. The pressureless sintering of the alumina disks has been done for 2 h at 1600° C in air. Evaluated by the water immersion technique through the Archimedes' principle the sintered samples exhibit a density (ρ) of ~97.2% of the theoretical (ρ_{th}) value (3.98 g cm⁻³). The phase purity of the pressureless sintered alumina samples has been evaluated by the conventional X-ray diffraction (XRD, monochromatic CuKa₁ radiation, 35 mA, 40 kV, PANalytical X' pert Pro MPD diffractometer, The Netherlands) technique. The grain sizes of the alumina ceramic are evaluated by image analysis of the FESEM (Supra VP35, Carl Zeiss, Germany) photomicrographs taken from the polished and thermally etched microstructure. The same FESEM technique is also utilized for the microstructural observations of the samples after failure from the SHPB experiments. This is done with a view to elucidate the details of the high strain rate induced deformation and fracture features in the alumina ceramic. A 50-70 nm carbon coating is deposited on the samples/fragments by the arc deposition technique to avoid charging. This step is done prior to insertion of the alumina ceramic in the sample chamber for electron microscopy.

The SHPB technique has been commonly used for the dynamic characterization of brittle materials including alumina [6,8,9,11–17]. A schematic diagram of the SHPB setup used for the present experiments is shown in Fig. 2. The setup comprises of maraging steel bars of diameter 12.7 mm. The incident, transmitter and striker bars are respectively 1530, 1300 and 100 mm long. A soft copper disc is used to change the rise time of the pulse. Impedance matched tungsten carbide platens are used between the specimen and the bar ends to prevent damage at the bar ends from the hard alumina fragments. The platens are themselves jacketed with shrunk fit maraging steel collars to prevent their failure during the test. Molybdenum di-sulfide grease has been used between the bars

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