Micromechanics of Deformation in Porous Liquid Phase Sintered Alumina under Hertzian Contact

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Abstract

A series of fine-grained porous alumina samples, with and without a liquid phase, were fabricated in compositions matched closely to commercially available alumina used as microelectronic substrates. Hertzian indentation on monolithic specimens of the glass-containing samples produced a greater quasi-ductile stress-strain response compared to that observed in the pure alumina. Maximum residual indentation depths, determined from surface profilometry, correlated with the stress-strain results. Moreover, microstructural observations from bonded interface specimens revealed significantly more damage in the form of microcracking and under extreme loading, pore collapse, in the glass-containing specimens. The absence of the typical twin faulting mechanism observed for larger-grained alumina suggests that the damage mechanism for quasi-ductility in these fine-grained porous alumina derived from the pores acting as a stress concentrator and the grain boundary glass phase providing a weak path for short crack propagation.

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Introduction

Localized damage in engineering ceramics due to single or repeated blunt contacts can significantly impact a product's lifetime or useful service. In the fabrication of microelectronic components such as substrate assemblies, repeated contact through automated handling and manufacture can cause small-scale deformation that may lead ultimately to catastrophic failure. Hertzian (or spherical) indentation has been shown effective for simulating contact damage under laboratory conditions.¹ As a result, Hertzian indentation was used to obtain a fundamental understanding of the underlying microstructural causes of deformation in a widely used liquid-phase-sintered (LPS) polycrystalline alumina.* Specifically, this investigation sought to understand the interplay amongst microstructural parameters such as porosity, second phase and grain size for several compositions of alumina similar to that of widely-used commercial material.

Recently, there has been increased interest in Hertzian contact damage of advanced ceramics (for a comprehensive review, see Lawn, 1998.¹) Under single-cycle quasi-static loading, dense fine-grained monolithic polycrystals or isotropic materials such as a soda-lime glass exhibit a wholly brittle response to Hertzian indentation: elastic deformation up to a critical load, followed by ring-crack formation near the perimeter of contact extending conically subsurface into the sample.² In contrast, other materials will behave in a macroscopically ductile manner with a deviation from a linear-elastic stress-strain response.^{3,4} The nature of the irreversible response to increased loading under the Hertzian indenter depends strongly on the microstructure of the material.

* Coors AD96R; 4 wt% glass, 3vol% porosity, 4 μm average grain size

For purely elastic Hertzian contact behavior[†], the functional relationship between the contact radius and the indentation load is:

$$a^3 = \frac{3}{4} \frac{PR}{E^*} \quad (1)$$

where

$$\frac{1}{E^*} = \frac{(1-\nu_1^2)}{E_1} + \frac{(1-\nu_2^2)}{E_2} \quad (2)$$

The radius of contact is a , the load is given by P and R is the radius of the indenter. Material constants are E , Young's modulus, and ν , Poisson's ratio, where the subscripts differentiate between the two contacting bodies. This relation derives directly from Hertz's generalized solution⁵ and can be rearranged to produce an elastic indentation stress-strain relation:

$$P_m = \left[\frac{4E^*}{3\pi} \right] \frac{a}{R} \quad (3)$$

where $P_m = P / \pi a^2$ is the mean indentation stress and a / R is a relative indentation strain. It can be seen that a plot of P_m versus a / R should result in a straight line whose slope is determined by the elastic properties of the material and indenter.

As mentioned previously, after attaining a critical indentation load some dense ceramics respond to Hertzian indentation in a way that appears macroscopically ductile (continuous deviation from elastic stress-strain behavior); this behavior results from homogeneously distributed brittle grain-scale fracture. The distributed damage initiates from beneath the indented surface in a triaxially compressive stress field at a depth of approximately one half the contact radius, where shear stresses are a maximum.⁶ This is in contrast with the brittle cone cracking

[†] Frictionless surfaces, loading force normal to point of contact.

that initiates from the surface near the indenter-sample contact boundary.²

A microstructural mechanism to accommodate shear-induced damage in brittle materials under compressive loading was first proposed by Brace *et al.*⁷ and consists of a preexisting crack that, under the actions of shear stress, nucleates wing cracks at its ends (Fig. 1(a)). Subsequent investigations have implemented successfully this basic model to describe brittle damage in both geological and ceramic materials.⁸⁻¹⁴ In advanced ceramic systems, shear-induced fault mechanisms can derive from weak interfaces, often between second phases or elongated grains and the surrounding microstructure.^{4,11-17} This mechanism is the most common under Hertzian contact and is usually associated with the macroscopic observation of long-crack toughness due to bridging elements in the crack wake.^{13,15}

A variation on the shear fault is the twin fault; an undeformed grain develops a mechanical twin, often parallel to the plane of maximum shear stress, which then acts as the fault plane. As an initiation site for distributed damage under Hertzian contact in advanced ceramics, twin-faulting has most commonly been found in alumina,^{3,4,18} but can also occur in Mg-partially stabilized zirconia.¹⁹ Guiberteau *et al.*¹⁸ have shown that the critical grain size for twin formation and subsequent shear faulting in alumina is around 20 μm as determined by acoustic emission measurements. However, subsequent work by Wei and Lawn²⁰ with thermal wave imaging suggested that the critical size may be closer to 10 μm . For microstructures with grains larger than this critical size, the distributed damage scales with increasing load. Below the critical load for fault formation, only a minimal amount of damage is observed in the sub-surface region.^{3,4}

Though porosity has only recently been addressed in Hertzian contact investigations,^{21,22} it has been a source of concern in the study of brittle-ductile processes in geological systems for much longer²³⁻²⁸ with recent emphasis on effects at the microstructural level.²⁹⁻³⁶ Experimentally,

cracks have been found to initiate from pore spaces in sedimentary rocks^{29,30} and can affect the development of distributed microfracture leading to macroscopic ductile behavior. Theoretically, Sammis and Ashby³⁷ have addressed the case where cracks initiate from pores and interact under compressive stress states to produce macroscopically ductile behavior. The fundamental pore mechanism is shown in Fig. 1(b), where the pore radius is defined by r and diametrically opposed cracks of length l are positioned at the points of peak tensile stress. The localized peak tensile regions (positive stress) are aligned with the most-compressive principal stress σ_1 ($\sigma_1 < \sigma_2$) and propagate the cracks into a compressive stress field. Note, for simplicity the mechanism is shown in two dimensions, whereas in reality the maximum stresses are triaxially compressive. However, when $\sigma_1 < \sigma_2$ and $\sigma_2 = \sigma_3$, the stress intensity factor solutions are similar in character.³⁷ The 2D mode I stress intensity factor under biaxial compressive stress was estimated in closed form by Sammis and Ashby³⁷ and is reproduced below. The mode I stress intensity factor takes the standard form:

$$K_I = -F(\lambda, L) \cdot \sigma_1 \sqrt{\pi l} \quad (4)$$

where $F(\lambda, L)$ was approximated as:

$$F(\lambda, L) \cong \frac{1.1(1 - 2.1\lambda)}{(1 + L)^{3.3}} - \lambda \quad (5)$$

such that K_I may be expressed as:

$$K_I = -L^{1/2} \left\{ \frac{1.1(1 - 2.1\lambda)}{(1 + L)^{3.3}} - \lambda \right\} \cdot \sigma_1 \sqrt{\pi r} \quad (6)$$

In Eq. (6) l is the crack length, r is the hole radius, $L = l/r$ is the normalized crack length, λ is the ratio of the remotely applied stresses σ_1/σ_2 and symmetry with the loading axes is assumed in the hole/crack geometry (*see* Fig. 1(b)).

Very little work has addressed the fundamental effect of porosity in the micromechanics of deformation beneath Hertzian indenters in advanced ceramics. Clearly the work by Latella *et al.*²¹ suggested that porosity played a role, but no microstructural evidence was presented. It was the objective of this investigation to understand the underlying relationship between the porosity and the glass phase in LPS alumina.

Procedure

Powder Processing:

To vary systematically key microstructural features of LPS alumina compositions, two series of alumina materials were prepared with and without a liquid phase over a porosity range between <1 vol% to 8 vol%. Samples were prepared from high purity alumina powders (Sumitomo USA, Edison, NJ) of three different average particle sizes (0.3, 2, 5 micron). These were processed both with and without the addition of approximately 10 vol% anorthite glass ($\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$). A summary of the samples tested, together with the processing conditions and sample designation is given in Table 1. Briefly, pure alumina samples were made by clean-room processing and sintered subsequently in a hot press dedicated to clean-room-processed materials. Additional pure alumina samples were pressureless sintered with MgO impurities to control abnormal grain growth.³⁸ A single alumina sample containing an added liquid phase was hot pressed in a system separate from the clean room materials. The remaining LPS alumina samples were processed and sintered under ambient laboratory conditions. Commercial material was sectioned into squares (76.2 x 152.4 x 3.0 mm) from the as-received substrates.

To make the air-sintered pure alumina, reagent-grade magnesium nitrate (Johnson Matthey, Part no. 10799) was dissolved in methanol (99.9%) to produce a MgO dopant level of 1000 ppm. After mixing with the high purity alumina, the methanol was evaporated and the dried powder was calcined in a box furnace (Lindberg Model 54434, Watertown, WI) at 800 °C for 2 hours. The calcined powder was crushed, ball milled, sieved (180 μm ; VWR Scientific, 16126-130), and pressed into 25.4 x 4 mm disks at 35 MPa. They were then cold isostatically pressed at 350 MPa and calcined in air at 800 °C for 5 hours. The heating rate up to the calcining

temperature was 5 °C/min, and 10 °C/min up to the sintering temperature and back to room temperature for all pressureless sintered samples.

The hot-pressed samples were produced using a similar procedure outlined by Wang *et al.*³⁹ and the details of LPS alumina processing have been reported elsewhere.²² Submicron high purity alumina was used to vacuum hot press a single LPS sample to full density in an "unclean" hot press (Astro Model HP20-4560-FP20). All sintered samples received a final polish down to a one micron finish. Some of the LPS samples used for sub-surface investigations received additional hand polishing with 0.03 µm colloidal silica (Syton HT50, Remet Corporation, Chadwick, NY) to preferentially polish down the glassy phase.

Microstructural Characterization:

In order to facilitate grain size measurement, selected samples were etched thermally to reveal the grain structure. Etching was performed in air and ranged in temperatures from 1300°C to 1500°C for 18 to 60 minutes depending on the material: lower temperatures and longer times were used for the higher porosity samples to prevent further densification. Images for grain size analysis were obtained from scanning electron microscopy (SEM) (Au-Pd coating, 20 keV; ETEC Autoscan). The grain size was determined with the random line intercept method described by Underwood.⁴⁰ The average grain sizes of all the alumina samples are shown in the final column of Table 1. Density measurements of all samples were made on the sintered and polished disks using the Archimedes method.

Mechanical Characterization:

Single-cycle (0.1 Hz) indentation was performed with a servo-hydraulic test frame (Model 1350, Instron, Canton, MA) using loads from 45 to 4000 N on polished specimens

sputter coated with gold-palladium. Tungsten carbide indenters of 1.59, 3.18, and 4.76 mm radius were used.

Typically, observations of the subsurface damage patterns have been obtained from a bonded interface specimen configuration.^{4,41} The technique relies on sectioning the sample in two, polishing and reassembling the material with an adhesive. Indentations are placed along the interface and the subsurface damage may be examined by separating the two halves. The microscopic damage was first examined under Nomarski (interference contrast) light optical microscopy (LOM) (Olympus Vanox, Japan) and then characterized further with SEM (uncoated, 3.1 keV; Model 6300, JEOL USA, Peabody, MA).

The Nomarski microscope was also used to photograph indentations in plan view on monolithic samples. A sputter coating of Au-Pd on the polished surface revealed the contact area between the indenter and the sample. The contact area, measured from calibrated photographs, was used to determine the value a in Eq. (1) and Eq. (3). Images were recorded at magnifications from 40x to 200x.

Selected indented samples were analyzed in a profilometer (Model P2, Tencor, Santa Clara, CA) to measure the geometry of the residual impression. A three-dimensional scan was obtained by making a series of line scans over a fixed distance using the profilometer's automated software. Unless otherwise noted, three indents per sample were measured.

Results and Discussion

Indentation Stress-Strain Behavior:

Fig. 2 shows the indentation stress-strain behavior of the commercial polycrystalline alumina (AD96R), a laboratory-produced sample with similar composition (A5_G10) and a sample of similar porosity and grain size without a glass phase (A5). The linear elastic behavior determined from Eq. (3) for the commercial material is plotted as the dashed inclined line in Fig. 2. All three curves display a deviation from linear elastic behavior around 5 GPa, which is consistent with earlier observations of alumina.^{3,4,18} The deviation from linearity is most pronounced in the AD96R sample. The A5_G10 sample with a microstructure closely resembling that of AD96R experienced slightly less fall-off from linear elastic behavior. Removing the glass phase and keeping a similar grain size and porosity (A5) further shifted the stress-strain behavior towards a linear elastic response. This was the first indication of the influence of the glass phase in the presence of porosity on quasi-ductility in this system: addition of a glass phase increased the quasi-ductility under Hertzian indentation for the given porosity in the fine-grained alumina.

Estimation of Uncertainty:

Because the materials in this investigation have very similar microstructures, differentiating between their mechanical response to indentation must be done with care. To compare the accuracy of the curves obtained in Fig. 2, a closer look at the potential uncertainty of the measurements involved is warranted. Expressions for the relative uncertainty in the mean indentation stress (P_m) and relative indentation strain (a/R) can be derived in terms of the independent measurements of a and P from a straightforward statistical analysis. The indentation

strain is defined only by a and the constant R , therefore its relative uncertainty is simply da/a . It follows that the relative uncertainty in the indentation strain is then:

$$\frac{dP_m}{P_m} = \frac{dP}{P} + 2 \cdot \frac{da}{a} \quad (7)$$

The relative uncertainty in the load measurements varied from 1% for the largest loads to 5% for the smallest and the uncertainty in the contact area measurements varied between 1% and 2%, respectively. The error bars in Fig. 2 were calculated from Eq. (7) using the preceding error estimates. It is apparent that the relative uncertainty in the mean indentation stress is greater than in the indentation strain and is also more strongly dependent on the uncertainty in the contact area than on the load. Furthermore, although many points of adjacent curves in Fig. 2 have overlapping error bars, there is definite separation between the A5 and AD96R curves in the highest part of the indentation stress-strain curve with A5_G10 behaving more like AD96R. It is in this region where the majority of subsurface microstructural damage takes place; thus the data are sufficient to substantiate the increase of subsurface damage resulting from the interaction between glass and porosity.

Characterization of Residual Impressions:

Additional surface characterization consisted of observing residual impressions optically with the Nomarski microscope. The results are shown for the commercial alumina in Fig. 3(a) and A5_G10 in Fig. 3(b). During testing of both the AD96R and A5_G10 alumina it was noted that a residual surface impression developed prior to the indentation stress required to cause the appearance of ring cracks on the surface of the sample. The stress levels corresponding to the first observations of a residual impression and ring cracking are indicated in Fig. 2 as solid horizontal lines. Conversely, in the pure alumina, the residual impression formed *after* or at the same time as the observed ring cracking. As indicated previously, the observation of a residual

surface impression is correlated with the initiation of irreversible damage in the high shear zone beneath the indenter.⁶ This result suggested further an augmenting of quasi-ductility deformation under Hertzian contact from the interaction of the glass and porosity.

Determination of the surface profile was carried out for indents at a fixed load and indenter radius. The purpose of this test was to illustrate clearly how the change in microstructure affected the macroscopically observed deformation. A load indenter combination of 800 N and 1.59 mm radius was chosen from the stress-strain results because it represented conditions under which substantial subsurface damage was anticipated. The three-dimensional profilometer surface traces corresponding to the indents in Fig. 3 are shown in Figure 4(a) and 4(b). The maximum residual depths for both the AD96R and A5_G10 were about 1 micron as shown by the single traces in the lower part of the images.

The average maximum residual indentation depth is plotted in Fig. 5 for all the laboratory produced samples given in Table 1. The upper curve is for samples containing 5 wt% glass phase (A0_G10, A5_G10, A6.5_G10), and the lower curve represents specimens without glass (A0, A1, A5, A8). The residual indentation depth increased for both sample series as porosity increased. However, the residual depth was greater at a given porosity level for the samples containing glass compared to the pure alumina samples. The data from Fig. 5 is also consistent with the observations in the indentation stress-strain curves (Fig. 2); the porous LPS alumina experienced greater fall-off from the linear elastic behavior compared to the pure alumina. Moreover, the fully dense pure alumina and the LPS alumina revealed no residual indentation at this load. The lack of a residual indentation for the dense LPS alumina also correlates with previous observations by the authors: in the subsurface region of maximum shear stress, large glassy pockets lacking porosity remained undamaged after Hertzian indentation.²²

Thus, while increased levels of porosity increases the macroscopically observed quasi-ductility and hence, the residual impression for all samples, the presence of the glass phase enhances the effect at a given porosity. The presence of the glass phase alone, however, does not result in quasi-ductile behavior, as both fully dense samples of LPS and pure alumina respond in a similar manner, with no residual impression. Therefore, it can be stated that the increase in the residual impression, and hence the quasi-ductility, under Hertzian contact for the porous LPS alumina is a function of the porosity and the glass phase together.

Microstructural Analysis:

The next step of the investigation was to examine the subsurface damage to elucidate microstructural influences on the contact behavior. A5 and A5_G10 were chosen for comparison. Nominally these samples had similar average grain sizes (7 μm versus 5 μm) and serve to demonstrate the fundamental microscale processes controlling the development of damage. Bonded interface specimens were indented in two distinct loading regions. The first load (~ 6 GPa), was just above the macroscopically observed indentation yield stress. This was done to observe damage as it initiated within the microstructure. The second loading was more severe, corresponding to the very well developed part of the indentation stress-strain curve (~ 10 GPa). Extreme loading would ensure the maximum amount of subsurface damage, providing a distinct transition in the microstructural response from the lower loading condition.

Fig. 6 reveals the subsurface indentation character for these two samples. The A5 sample (Fig. 6(a) *left*) indicated virtually no observable damage at an indentation stress of 6 GPa. The post-indentation microstructure retained the original grain shape and morphology and little evidence of cracking was observed. By comparison, the A5_G10 sample (Fig. 6(a) *right*) revealed grain boundary microfracture through the glass phase with an occasional intragranular

fracture under a 6 GPa indentation stress. Increasing the indentation stress to approximately 10 GPa produced only a marginal change in the A5 sample. Most pores remained intact, as did the majority of the grains. Some grains were traversed by a crack (Fig. 6(b) *left*; arrow) but retained their shape. The behavior of the A5_G10 sample was in sharp contrast to the behavior of the pure alumina. Fig. 6(b) *right*, shows that the microstructure of the A5_G10 sample was substantially damaged by the 10 GPa indentation stress, undergoing pore collapse, grain rearrangement, and grain fracture.

Quasi-Ductile Mechanism:

The microstructural damage observed in Fig. 6 revealed two important trends that were consistent with the macroscopic observations. First, comparing Fig. 6(a) to 6(b), it was clear that subsurface damage was increased for both specimens at the higher indentation load. This result agreed with the general stress-strain behavior and profilometry results for both samples. It was also clear that the A5_G10 sample experienced a greater amount of subsurface deformation in the form of cracking and pore collapse compared to the A5 sample. Furthermore, recall that a negligible residual impression was observed for the A0_G10 sample, which correlated with large glassy pockets isolated from porosity remaining undamaged after indentation.²² Thus we find that the porous LPS alumina responded with a greater fall-off from the elastic stress-strain response, an increased residual indentation impression and a larger amount of subsurface microstructural damage than its pure alumina counterpart at similar porosity.

Mechanistically, this behavior is important to understand in light of the previous work on dense alumina by Guiberteau *et al.*¹⁸; i.e., dense fine grained alumina exhibited a solely brittle response to Hertzian contact, whereas larger grain sizes deformed by a twin faulting mechanism. From that result, one would not have expected a significant quasi-ductile response from the fine

grained ($<10\ \mu\text{m}$) LPS alumina in this investigation. Furthermore, none of the alumina samples tested revealed evidence of twin faulting to any extent, except for the most severe loading condition and only with the LPS material (A5_G10). Therefore, one may assume that the primary mechanism responsible for quasi-ductility in this system was not based on the classic shear/twin faulting model (Fig. 1(a)) as found in most quasi-ductile ceramics.

Previous work by the authors²² served to locate the origin of fracture at pore spaces within the microstructure, and furthermore showed that the cracks proceeded intergranularly where the glass phase was expected. It was concluded that the pore was acting as a stress concentrator and ultimately as an area of strain accommodation. The lower fracture toughness of the glass phase with respect to individual fracture toughness of the alumina grains provided a weak path for short crack propagation. Specifically, the intergranular fracture toughness of pure alumina is higher than glass⁴² and thus the stress concentrating pore mechanism would require a higher indentation load to initiate intergranular fracture.

The issue of pore size in the comparison of the A5 and A5_G10 samples should be considered. Although enhanced by a small degree of grain pull-out, the pores in the A5_G10 sample are larger than those in the A5 sample. The stress concentrating effect of an idealized pore can be estimated from Eq. (6). Assuming a similar fixed flaw size l at a given set of compressive stresses (σ_1, σ_2), the mode I stress intensity factor will be higher for larger diameter voids in relation to smaller ones. Thus, the pores in the A5_G10 samples would experience a larger stress concentrating effect at a given indentation stress than the A5 sample. Both the increased pore size and the presence of glass at grain boundaries would make quasi-ductile damage more likely at a lower indentation stress. It may be assumed that this factor was responsible for the observation of the residual impression on the surface *before* the appearance of

ring cracking for the LPS alumina because the ring-crack initiation stress is not strongly dependent on the surface flaw distribution.¹

A note of caution is required regarding ring-crack formation because the amount of quasi-ductile deformation that occurs under the Hertzian indenter can influence the surface tensile stress that propagates ring and subsequent subsurface cone cracks – lessening the surface tensile stress for larger quasi-plastic responses.⁴³ For example, in a similar LPS alumina system, a porosity of ~18% was shown to eliminate the formation of cone-cracks during indentation.²¹ However, in our material (A5_G10) and similar material produced by Latella *et al.*²¹ (~7% porosity), cone-cracks were observed. The most important aspect of the deformation was the order in which the damage developed -- residual impression versus ring-cracking -- rather than the absolute magnitude at which it occurred.

The results obtained from this investigation suggest clearly a critical relationship between the porosity and glass phase in the alumina microstructure, which controls damage induced by a Hertzian indenter. With respect to microelectronic substrates, the susceptibility to contact damage and wear is evident. Because both the porosity and glass phase are practical service requirements, future research should address ways of manipulating these parameters to reduce contact damage. To address the mechanistic role of the pores quantitatively, further investigations concerning pore size and pore arrangement were performed and will be reported in a subsequent publication.

Conclusions

Porosity in alumina was found to provide a mechanism for quasi-ductility under Hertzian contact through macroscopic and microscopic observations of deformation. It is believed that the pores act as stress concentrators allowing distributed microfracture to initiate and proceed intergranularly. The presence of the weaker intergranular glassy phase facilitates this process, leading to enhanced quasi-ductile behavior at a given porosity. Furthermore, twin faulting, which has been previously established as a deformation mechanism giving rise to quasi-ductility, was not observed; a result of the fine grain size of the specimens studied ($\leq 7 \mu\text{m}$). Thus, porosity may be viewed as an additional mechanism leading to quasi-ductility under Hertzian contacts, especially in the presence of weak grain boundary phases. The results have important implications for tailoring the response of commercial LPS alumina products subjected to Hertzian-like contacts during fabrication and service.

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Table 1 Alumina Sample Characteristics

Sample Designation	Forming Method [‡]	Forming Temperature (°C)	Time (h)	Glass (vol%)	Porosity (%)	Average Grain Size (μm)
A0	AS	1600	1	-	<0.5	1
A1	AS	1325	5	-	1	2
A5	HP, 30 MPa	1450	0.67	-	5	7
A8	AS	1300	0.5	-	8	<1
A0_G10	HP, 50 MPa	1250	0.5	10	~0	3
A5_G10	AS	1600	10	10	5	5
A6.5_G10	AS	1600	2	10	6.5	4
AD96R	AS	*	*	7	3	4

‡ - AS = air sintering and HP = hot pressing.

* - unknown commercial parameters

Figure Captions

Figure 1 Quasi-ductile mechanisms. (a) Shear fault with wing-cracks propagating from ends. (b)

Idealized pore/crack mechanism under compressive stress. Cracks are symmetric with length = l and pore radius = r .

Figure 2 Indentation stress-strain curve for AD96R alumina. Deviation from Hertzian behavior

is correlated with the observation of residual surface deformation under Nomarski LOM. Ring cracking in AD96R and A5-G10 occurs at higher loading with increased fall-off in the stress-strain curve. Error bars calculated from Eq. (7).

Figure 3 Nomarski optical micrograph of the indented surface. $P = 800$ N, $R = 1.59$ mm. (a)

AD96R; slight ring-cracking is visible at the perimeter of contact on the upper-right of the impression. Higher magnification reveals a fine ring-crack surrounding the entire indent. (b) Similar indentation behavior was observed for A5_G10.

Figure 4 Profilometry results from the residual impressions corresponding to Figure 3. (a) 3D

surface profilometry of AD96R. A single trace reveals a residual depth around 1 micron (bottom). The average residual depth for four indents was 1.5 microns. (b) 3D surface profilometry of A5_G10. A single scan from the center of the impression reveals a maximum depth of approximately 1 μm . The average residual depth for three indents was 1.1 μm . Y-axis units $\times 1000$ angstroms.

Figure 5 Plot of the residual depth of indentation in alumina at a fixed 800 N load and indenter

radius ($R = 1.59$ mm) for increasing sample porosity. Note both curves contain a data point at the origin. Lines are provided as a guide to the eye.

Figure 6 Comparison of subsurface damage between A5 and A5_G10. (a) 6 GPa indentation

stress. A5 shows no visible damage whereas the A5_G10 sample produced visible

cracking within the microstructure in the high-shear region beneath the indenter. (b) 10 GPa indentation stress. Only marginal amounts of damage was observed within the high shear subsurface region of A5. Cracking could be observed within grains (arrow). In the case of the A5_G10 sample, grain rearrangement and pore collapse was evident. Intragranular fracture was also seen (arrow).

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Figure 6 Comparison of subsurface damage between A5 (*left*) and A5_G10 (*right*). (a) 6 GPa indentation stress. A5 shows no visible damage whereas the A5_G10 sample produced visible cracking within the microstructure in the high-shear region beneath the indenter. (b) 10 GPa indentation stress. Only marginal amounts of damage were observed within the high shear subsurface region of A5. Cracking could be observed within grains (*arrow*). In the case of the A5_G10 sample, grain rearrangement and pore collapse was evident. Intragranular fracture was also seen.