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CHARACTERIZATION OF SUBSURFACE DAMAGE OF EXPLOSIVELY INDENTED SILICON NITRIDE CERAMICS

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In this study, explosive indentations were used to characterize the dynamic damage behavior of silicon nitride ceramics with different microstructures. Three grades of silicon nitrides with different grain size and shape were prepared. Subsurface damage of specimens was characterized extensively using optical and electron microscopy. It was found that the dynamic damage response depends strongly on the microstructure of specimens, particularly on the glassy grain boundary phase.

Keywords: Silicon nitride; explosive indentation; dynamic indentation.

1. Introduction

Silicon nitride has received much attention for structural ceramics, due to its high hardness, high strength, low thermal expansion coefficient, and high fracture toughness. In general, ceramics materials have limitation to apply for structural parts because of their brittleness. The effect of microstructure on mechanical properties is crucial importance for performance and reliability of ceramic components. Hence silicon nitride has been studied to overcome the brittleness of silicon nitride by tailoring microstructure in the past two decades.[1] In the recent study about sphere indentation on various silicon nitrides has elucidated the effect of microstructure on mechanical properties: especially flexural strength, yield strength, and R-curve behavior.[2] In recent, newly developed silicon nitrides have comparably high hardness, toughness, and modulus, relative to those of ceramics that has been successfully applied to armor systems. Thus silicon nitride is expected to provide effective resistance to impact damage. Some studies have been devoted to the dynamic fracture behavior of silicon nitride with the comparison of damages between static and dynamic indentation experiments. Previous studies have

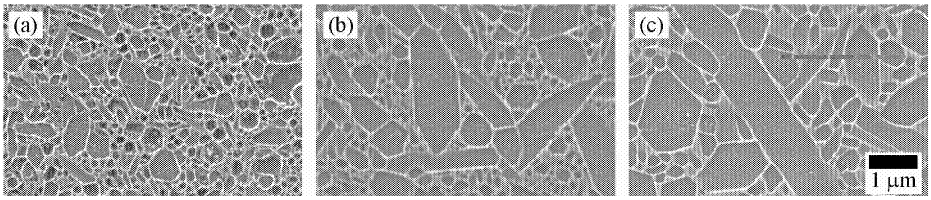

Fig. 1. SEM micrographs of polished and plasma etched silicon nitride specimens sintered at (a) 1600 °C, (b) 1700 °C, and (c) 1800 °C.

provided the evidence of an interrelationship between the static and dynamic fracture properties of silicon nitride and clue to ballistic efficiency as a armor ceramics.[3,4]

In this study, three grades of silicon nitride were fabricated by hot-press sintering technique at different sintering temperature. The 'bonded-interface' specimen that enables the observation of damaged subsurface, is prepared for testing. Explosive indentation on prepared silicon nitride was conducted to elucidate the damage evolved to dynamic fracture. The subsurface of indented specimen was observed with optical and scanning election microscopy. Extensive transmission election microscopy was applied to characterize the micromechanical failure of dynamically damaged specimens. The purpose of this study is to reveal the effect of microstructure on explosive indentation damage and investigate the primary failure mode of silicon nitride.

2. Experimental method

2.1. *Specimen preparation*

Three grades of silicon nitrides were prepared by hot pressing sintering technique using starting powder consisted of a silicon nitride (UBE-SN-E10, Ube Industries, Tokyo, Japan) with following additives: 5 wt% Y_2O_3 (Fine Grade, H. C. Starck GmbH, Goslar, Germany), 2 wt% Al_2O_3 (AKP50, Sumitomo Chem. Co. Ltd., Tokyo Japan), and 1 wt% MgO (High Purity Baikowski Co, NC, USA). Densification was achieved in nitrogen atmosphere at 1 atm under uniaxial pressing pressure 25 MPa, at 1600, 1700, 1800 °C for 1 hour in a hot press (Astro Industries Inc., CA, USA). The sintering temperature produced fine equiaxed (F) at 1600 °C, intermediate (M) at 1700 °C, and coarse elongated (C) microstructure at 1800 °C.

Specimen surfaces normal to the hot pressing direction were polished to 1 μm. finish to enable optical microscope observation. The surfaces were plasma etched to reveal the grain structures. The microstructure of the specimens was observed by scanning electron microscope. Prior to explosive indentation testing, the specimens were machined into 6 mm x 8mm x 20 mm rectangular-shaped bars. Side surfaces normal to top surface were polished to observe the subsurface damage. The opposing sides were adhered together using adhesive to fabricate 'bonded-interface' specimens.[5] In the preparation of bonded-interface specimen, the gaps between two bars were maintained below 10 μm to minimize the additional free surface effect. After explosive indentation experiment, these bonded interface specimen were separated and side surface was cleaned by acetone.

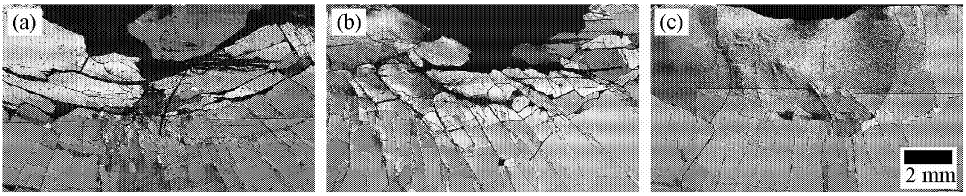

Fig. 2. The optical micrographs of the subsurface damage of explosively indented silicon nitride sintered at (a) 1600 °C, (b) 1700 °C, and (c) 1800 °C.

2.2. *Explosive indentation and damage observation*

Explosive indentation testing was made on the prepared bonded-interface specimens to reveal the dynamic deformation of specimens. These specimens where clamped with auxiliary fixture to inhibit shattering of specimen during impact loading. Explosive indentation was performed using the electric bridge wire (EBW) detonator. The small explosive detonator encased in a stainless steel cylinder and 5 mm diameter bottom plate. The detonator propels the bottom stainless steel flyer toward specimen top surface having ~1 km/s velocity to induce impact shock.

Optical and scanning electron microscopy (SEM) was used to observe the subsurface damage of explosively indented specimens. Specimens were gold-coated for viewing in Normarski interference optical microscope which emphasizes the feature of micro-cracks and deformation. Observation of the subsurface damage zone in higher magnification was conducted by SEM. Dual focused ion beam milling (FEI, Netherlands) was adopted to prepare the specimens for transmission electron microscopy (TEM) observation. This technique enables the investigation of intrinsic fracture nature of dynamically indented specimens.

3. Results and Discussion

3.1. *Preliminary characterization*

Results of the microstructural characterization are shown in Fig 1. Fig. 1 shows scanning electron microscopy micrographs of the different silicon nitride microstructure for each sintering temperature. At 1600 °C, the material contains only mainly equiaxed α grains of mean grain size ~0.2μm, but some elongated β grains are found. At 1700 °C, these β grains have continued their grain growth and β phase is dominant. At 1800 °C, the β grains enlarge further and mean grain size is much increased. The starting powder contains only α phase silicon nitride. In the sintering process, an α to β transformation takes place rapidly and there is no detectable α phase above 1700 °C. The variation of mean grain sizes and aspect ratios was evaluated at each sintering temperature. Overall grain sizes increases steadily with sintering temperature from 0.2 to 1 μm. It is notable

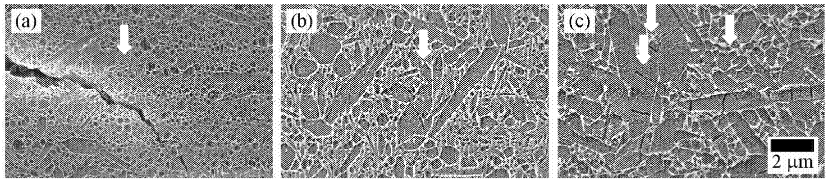

Fig. 3. SEM micrographs of quasi-plastic deformation zone of silicon nitride sintered at (a) 1600 °C, (b) 1700 °C, and (c) 1800 °C.

that the population of elongate β grains is most dominant in silicon nitride sintered at 1700 °C. The development of microstructure shows same results with previous study and we can expect similar mechanical properties, such as Young's modulus, hardness and R-curve behavior.[2]

In the previous study, Hertzian indentation tests were conducted to evaluate the contact damage resistance of silicon nitride ceramics.[2-4] In brief, the examination of materials indicates a competition between brittle and quasi-plastic damage modes: in structures with relatively equiaxed grains, the damage takes the form of classical cone cracking; in structures with large elongate grains, the damage is distributed beneath the contact as grain-localized microfailures within a subsurface yield zone.

3.2. *Explosive indentation damages*

Figure 2 shows the subsurface side views of explosively indented specimens. As shown in the micrographs, radial and lateral cracks are dispersed through the entire area of the specimens with partial fragmentations. The macroscopic cracking and fragmentation appear to represent post-shock damages, because the reflected wave breaks the specimen into several pieces. This can be inferred from the observation that the orientation of the macroscopic cracks corresponds with the normal of the compressive wave. In contrast to previous study about static sphere indentation, brittle and quasi-plastic transition is not clearly shown in the explosive indentation. The amount of spallation dominant damage is apparent in a sequence of sintering temperature 1600→1700→1800 °C. Interestingly, the silicon nitride at 1600 °C shows a quasi-plastic deformation just below the spallation region. This implies that all specimens would contain quasi-plastic damages. Like the previous study about explosive indentation on silicon carbide, the role of the weak grain boundary phase on the evolution of subsurface damages appears to be primary failure site during dynamic fracture. The analogous fracture mode could operate in silicon nitride specimens because the glassy phase is distributed on the entire grain boundary surface. The other literature also reports the weak grain boundary attacking mechanism with regard to the dynamic failure of several armor ceramics.[6,7]

3.3. *Subsurface damage observation with SEM and TEM*

The quasi-plastic zone of damaged silicon nitride specimens were examined via extensive scanning electron microscopy to reveal the fracture mode of explosively indented

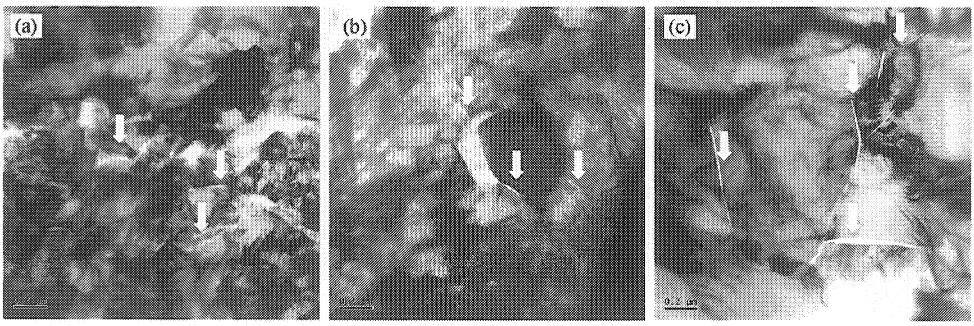

Fig. 4. TEM micrographs of quasi-plastic deformation zone of silicon nitride sintered at (a) 1600 °C, (b) 1700 °C, and (c) 1800 °C bright field images.

specimens. The results for each specimen are shown in Fig. 3. Extensive examination of the quasi-plastic zone revealed strong evidence of major grain boundary attacking and minor transgranular fracture. These failures mechanism is also suggested by Meyer's principal damage initiation.[8] As shown in the micrographs, grain boundary debonding and voids initiation are the dominant fracture mode. Also elongate abnormal grain beaks into several pieces. Two mixed fracture mode are mainly due to the glassy grain boundary phase and shows trends corresponding to comminuted zone of impact damaged silicon carbide in Hauver's work.[9] In Fig. 4, the observation of explosively indented silicon nitrides were shown. The transmission election microscopy bright field images at high magnification clearly indicate the intergranular fracture of silicon nitrides. The presence of cracking along grain boundaries was observed and it is believed that these defects were incorporated in the process of impact damage. Grain boundary debonds and failure is clearly shown in all samples and seems to play a primary role in the resistance to dynamic fracture.

4. Conclusion

The explosive indentation on the hot pressing silicon nitride was conducted to study the effect of microstructure on the dynamic fracture. It was found that silicon nitrides have brittle fracture dominant behavior in fine grain specimen and quasi-plastic deformation dominant in the coarse grain specimen. However the quasi-plastic deformation failure is primary fracture mode in all microstructures. Extensive SEM and TEM observation of the damaged region confirmed the nature of dynamic fracture mode: the weak grain boundary attacking and transgranular fracture. The weakness of glassy grain boundary phase in the silicon nitride ceramics is considered as an important factor to determine impact damage resistance.

Acknowledgments

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