Machinability and Contact Damage of Al$_2$O$_3$/BN Composites Fabricated through Chemical Processing

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The machinability and contact damage of Al$_2$O$_3$/BN nanocomposites fabricated through the chemical method were investigated in the present work. The Al$_2$O$_3$/BN microcomposites indicated considerably low fracture strength by adding BN particles, although machinability was improved. However, the nanocomposites showed both high fracture strength and good machinability. Hertzian contact test was performed to evaluate the contact damage. From the result of SEM observation of the samples after this test, it was found that the damage of the monolithic Al$_2$O$_3$ and Al$_2$O$_3$/BN microcomposites have a classical Hertzian cone fracture and many large cracks, respectively, whereas the damage observed in the nanocomposites appeared to be quasi-plastic deformation.

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1. Introduction

Among ceramic materials, Al$_2$O$_3$ is one of the most important engineering materials in the field of structural materials because of high fracture strength and high Young’s modulus in spite of an inexpensive material. In order to extend the application of Al$_2$O$_3$ ceramics, many researchers have studied that the incorporation of second phase dispersoids into Al$_2$O$_3$ matrix, have reported the enhancements of mechanical properties such as fracture toughness, fracture strength and creep resistance. However, there are a few researches of Al$_2$O$_3$ ceramics about the improvement of machinability. Since Al$_2$O$_3$ as well as other structural ceramics possesses the properties of brittle fracture and hard machinability, its commercialization needs high cost manufacturing to machine sintered materials to the form of products.

Some researchers have studied improvement of machinability of hard ceramics by adding hexagonal BN (h-BN) which is weak and soft material due to extremely low Young’s modulus and cleavage property. However, by the addition of micro-sized h-BN particles as a second phase, it has been reported that fracture strength decrease remarkably with an increase in h-BN content due to the aggregation of BN particles caused by conventionally mixing commercially powders, even though its machinability was somewhat improved. Also, large grain size h-BN particle maybe initiate fracture, because Young’s modulus of h-BN is lower than 1/6 of that of monolithic Al$_2$O$_3$. Therefore, for h-BN dispersed ceramic composites, the nanocomposite structure, which is the homogeneous dispersion of nano-sized h-BN particles into Al$_2$O$_3$ matrix, is probably effective to enhance the fracture strength of machinable Al$_2$O$_3$/BN composites.

Recently, the authors have investigated Si$_3$N$_4$/BN nanocomposites in which the nano-sized h-BN particles are dispersed within Si$_3$N$_4$ matrix grains and/or at the grain boundaries. As a result, it has been reported that the nanocomposites indicate good machinability and strongly improved fracture strength compared to the microcomposite. It is interesting whether the same result as Si$_3$N$_4$/BN nanocomposites is obtained with Al$_2$O$_3$ matrix.

Furthermore, the investigation of contact damage of this nanocomposite is also interesting, due to the unique microstructure in which extremely soft and weak second phase particles were homogeneously dispersed in hard ceramics matrix. Hertzian contact test has been introduced as a method to clarify the deformation mechanisms by mechanical damage. It has been reported that the contact damage by Hertzian indentation for homogeneous brittle materials, such as glasses and single crystals, was the Hertzian cone crack, which is common for brittle ceramics. However, Lawn and co-workers have shown in their recent work that the nature of this contact damage changes fundamentally, as the ceramic becomes coarser and more heterogeneous. Some cone fracture observations have been reported on low-toughness fine-grain monolithic ceramics, for example Al$_2$O$_3$ with fine grain size. However, examination of Hertzian indentation in Al$_2$O$_3$ ceramics with large grain size or relatively high porosity has revealed a fundamental transition from classical Hertzian cone fracture to quasi-plastic deformation with the accumulated damage. Since this deformation mechanism has a potential to disperse and absorb mechanical shock, the relation between machinability and quasi-plastic deformation is described in some papers.

The purposes of this contribution is to evaluate the machinability of Al$_2$O$_3$/BN microcomposites and nanocomposites by drilling and observation of drilled surface, and to investigate the effect of microstructure and nanostructure on the contact damage using the Hertzian contact test.

2. Experimental

2.1 Fabrication of test specimens

For fabrication of Al$_2$O$_3$/BN nanocomposites, the process presented in previous paper was adopted. The starting powders for the nanocomposites were the commercially available α-Al$_2$O$_3$ powder (AKP53, Sumitomo Chemicals, Tokyo) and boric acid (Reagent grade, Wako Pure Chemi-
nal Industries Ltd., Tokyo) and urea (Reagent grade, Wako Pure Chemical Industries Ltd., Tokyo) as the BN precursors. The BN content was adjusted to be 20 and 30 vol% in this process, where Al2O3 powder, boric acid and urea were mixed by the conventional wet ball milling method with ZrO2 balls and ethanol for 24 h. The dried mixtures were reduced at 400°C and 1000°C in hydrogen gas, and then heated at 1500°C and 1600°C in nitrogen gas to produce the Al2O3-BN composite powders. The composite powders were ball milled in ethanol for 72 h again. After second step of ball milling, the slurry was dried, and then the powders were dry ball milled for 6 h to eliminate hard agglomerations. For comparison, the commercially available BN powder with an average grain size of 9 μm (GF grade, Denka Co., Ltd., Tokyo) was also used to fabricate Al2O3/BN microcomposites.

Hot-pressing for the composites containing 0, 20 and 30 vol% of BN was performed at 1400, 1650 and 1700°C for 1 h in nitrogen atmosphere under 30 MPa of applied pressure, respectively.

2.2 Evaluation of machinability

Machinability of each composite was tested using tungsten carbide (WC/Co) drill bit of 1–2 mm in diameter. The drilling test was done using a standard drill press operating at 660 rpm. The composite specimens were mounted on a load cell and tested by manually applying a constant normal force to the drill at a load of 50 N.

2.3 Hertzian contact test

Ball indentation measurement

Ball indentations were made using WC/Co spheres of radii r = 1, 1.5 and 2 mm, with a load range from P = 0 to 1960 N using an universal testing machine (Autograph, AG–10TC, Shimadzu Co., Ltd.), as shown in Fig. 1. Contact radius (a) at each applied load was determined by residual traces in the gold layer after ball indentation, and hence contact pressure (P0) as a function of indentation strain (a/r) was plotted (see Fig. 4).

Damage observations

Examination of sub-surface contact damage by ball-indentation was made using a bonded-interface technique, consisting of two polished half blocks joined together by adhesive. Indentations were made symmetrically across the traces of the interface at loads from 1470 and 1960 N with a tungsten carbide (WC) sphere of radius 1.5 mm. After testing, the bonded materials were separated and the surfaces and sections cleaned with acetone. The polished specimens were viewed in an optical microscope using Nomarski interference contrast to reveal the macroscopic damage patterns, as also represented in Fig. 1.

3. Results and discussion

All the specimens consisted of α-Al2O3 and h-BN. The measured results of mechanical properties for the sintered materials are included in Table 1. As shown in this table, Young’s modulus of both microcomposites and nanocomposites decreased with increasing h-BN content according to the rule of mixtures, but the fracture strength of the nanocomposites was significantly improved, compared with the conventional microcomposites.

3.1 Machining

It was impossible to machine Al2O3/BN composites containing less than 10 vol% BN by using a WC/Co drill. Al2O3/15 vol% BN composites were somewhat difficult to machine. However, as shown in Fig. 2, good machinability was observed in both Al2O3/BN microcomposites and nanocomposites containing more than 20 vol% BN. Especially the nanocomposites represented excellent machinability like metals with retaining high strength. The difference

![Fig. 1. Hertzian test geometry for bonded-interface specimen. Sphere, radius r, delivers load P over contact radius a. Specimen consists of two polished halves bonded across the interface.](image1)

![Fig. 2. Machining of the nanocomposites using WC/Co bits: (a) drilling of Al2O3/15 vol% BN and (b) turning of Al2O3/30 vol% BN.](image2)

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<th>Table 1. Characteristics of Hot-Pressed Materials</th>
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<td><strong>Materials</strong></td>
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<td>**** Microcomposite</td>
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<td>Strength (MPa)³</td>
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<td>Young’s modulus (GPa)²</td>
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<td>Hardness (GPa)⁴</td>
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³Three point bending test.
²Resonance vibration method with first-mode resonance.
⁴Vickers hardness test with 98N load.
of machinability between 15 and 20 vol.% BN content is attributed to the BN particles dispersed at grain boundaries. The intergranular BN particles, rather than the intragranular BN particles, are effective for machinability, because the intergranular fractures were frequently observed in the drilled surface of the Al₂O₃/BN composites containing over 20 vol.% of BN. It has been reported that machinability is caused by the weak interface between matrix grains and second phases at grain boundaries. Morgan et al. and Hikichi et al. reported, even considerably hard material, ZrO₂, exhibited machinability due to easy grain detachment caused by incorporating rare-earth phosphate, specially weak interface between ZrO₂ and rare-earth phosphate, into the microstructure. The machinability observed in the nanocomposites with high strength were probably achieved by grain detachment promoted by weak interface formed between Al₂O₃ matrix grains and intergranular fine h-BN particles with cleavage property.

3.2 Damage by machining

Figure 3 presents the drilled surfaces of the microcomposite and nanocomposite materials containing 30 vol.% BN dispersion. In the drilled surface of all samples, it seems the intergranular fracture of Al₂O₃ grains is dominant, and the surface was partly covered with the debris of h-BN. From these SEM micrographs, the significant different micromechanism of material removal was recognized between the microcomposites and the nanocomposites. In the microcomposite, the damage caused by drilling was very severe because of lager crack path along micro-sized BN grain and its aggregation. As shown in Fig. 3(d), the large chippings of drilling track were observed in the drilled surface of the microcomposites. In comparison with the microcomposite, the damage in the nanocomposites, characterized by finer h-BN dispersion, was considerably minor. Furthermore, as can be seen from Figs. 3(a) and (b), the drilling tracks in the nanocomposite were obvious and shallow grooves without breakdown. These grooves formed by sharp edge of drill bit demonstrates good machinability that might be possible to shape the nanocomposites into complex shape. In the microcomposites, such sharp grooves were not evident because of heavy damage in the drilled surface. The superior damage property and machinability of the nanocomposites presumably results from the homogeneous dispersion of finer h-BN particles, which is expected to absorb and to dissipate the damage force during the machining operations by many finer cracks formed at the grain boundary.

3.3 Indentation stress-strain curve

Hertzian contact test studied by Lawn et al. is a simple experiment to investigate the potentials to absorb the mechanical shock. In this work, this test was performed in order to demonstrate the damage mechanisms of each sample. Figure 4 showed indentation stress-strain curve for the monolithic Al₂O₃ and Al₂O₃/BN nanocomposites including 20 and 30 vol.% BN dispersions. The responses were obtained by monitoring mean contact pressure \( p_0 \)

\[
p_0 = P/\pi a^2
\]

as a function of the geometrical ratio \( a/r \), where \( P \), \( a \) and \( r \) are the indentation load, the radius of contact and the sphere radius, respectively. The monolithic Al₂O₃ behaved in an ideal brittle manner, with near linear response up to \( p_0 = 8.5 \) GPa, but above that stress, the curve slightly deviated below the Hertzian line due to deformation of the WC/Co spherical indenter. In contrast, the curves of the Al₂O₃/BN nanocomposites including 20 and 30 vol.% BN dispersions showed dramatically deviated from the linear response at \( p_0 = 6 \) and 5 GPa. Considering these responses of the monolith and the nanocomposites, it is supposed that some deformation occurred at lower indentation stresses with increasing in BN content. These results imply that the deformation can be promoted by homogeneous dispersion of nano-sized BN.

3.4 Observations of subsurface damage

Figure 5 shows the half-surface (upper) and side section (lower) views of Hertzian contact damage for Al₂O₃ monolith and Al₂O₃/BN microcomposite and nanocomposite containing 20 vol.% BN dispersion, respectively. The damage in the monolithic Al₂O₃ showed typical Hertzian cone fracture, in which the cone cracks propagate downwards and outwards into the specimen (Fig. 5(a)), and the detectable deformation except the cone crack could not be observed beneath the contact in the monolithic Al₂O₃. This fracture pattern in the monolithic Al₂O₃ is indicative of a highly brittle solid. On the other hand, there are many large cracks propagating in the perpendicular of hot press direction in the subsurface deformation zone of the Al₂O₃/20 vol.% BN microcomposite (Fig. 5(b)). These large cracks were obviously different from the classical Hertzian cone crack observed in the monolithic Al₂O₃, and may be attribut-
ed not only to the dispersion of larger $h$-BN particles but also to the microstructure with the orientation of platelike $h$-BN perpendicular to the hot-pressing direction. The platelike $h$-BN particles with crystallographic anisotropy easily oriented perpendicular to the hot-pressing direction during hot-press sintering. Platelike $h$-BN constituted by stacking of BN hexagonal network has cleavage plane between hexagonal networks of boron and nitrogen. Because of this cleavage property, the strength perpendicular to the hot-pressing direction of bulk specimen is very weak, and, hence, cracks propagate easily in this direction. As a result, it is presumed that such large oriented cracks were observed in the microcomposite, as shown in Fig. 6.

In contrast to previous two materials, in the nanocomposite, the damage observed in subsurface deformation zone appears to be quasi-plastic deformation, like the plastic deformation zone in ductile metals. And also, this deformation accompanied the residual surface depression instead of surface ring crack, and this fact represents the transition from brittle to ductile by finer $h$-BN dispersion. The nanocomposite has the microstructure in which fine $h$-BN particles were homogeneously dispersed within $\text{Al}_2\text{O}_3$ grain as well as at the grain boundary. Therefore, it is thought to be possible to disperse and absorb the stresses from the ball-indentation by deformation and/or fine cracks originated from soft $h$-BN particle dispersed at the grain boundary.

Figures 7 (b) and (c) show SEM photographs of the undeformation surface and the quasi-plastic deformation zone in the ball-Indented specimen of the nanocomposite. Significant grain boundary detachments were frequently observed in the damage zone. These observations suggested that the nano-sized intergranular BN particles weakened the bonding strength of the grain boundary. The quasi-plastic deformation zone, in which microcracks concentrated, was driven by a strong shear component at the grain boundary. It is expected that the quasi-plastic deformation observed in the nanocomposites has affected good machinability not a little.

4. Conclusion

The $\text{Al}_2\text{O}_3$/BN nanocomposite containing more than 20 vol.% of BN dispersion attained high fracture strength and good machinability simultaneously. The damage by drilling in the nanocomposites is minor compared with the microcomposites. Although the contact damage of the monolithic $\text{Al}_2\text{O}_3$ was the classical Hertzian cone crack, indicative of a highly brittle material, the damage form of the nanocomposites indicated the quasi-plastic deformation which is expected to absorb and to disperse the damage. This deformation zone was formed by the microfractures, which caused by the weak interface between $\text{Al}_2\text{O}_3$ and $h$-BN at the grain boundary.

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