

## Fatigue Behavior of 2 and 4 mol% $Y_2O_3$ -Containing Tetragonal Zirconia Polycrystals (Y-TZP)

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*The fatigue behavior of 2 and 4 mol%  $Y_2O_3$ -containing tetragonal zirconia polycrystals (Y-TZP) was studied by measuring their fracture strength  $\sigma_f$  as a function of stressing rate  $\dot{\sigma}$  (dynamic fatigue technique). The average grain size of 2 mol%  $Y_2O_3$ -containing tetragonal zirconia polycrystals (Z2Y) was 1.0-1.5  $\mu\text{m}$ , while Z4Y was composed of 2.0-2.5  $\mu\text{m}$  grains in the most part and above 3  $\mu\text{m}$  grains in the other part. Stress-deflection relationship of Z2Y at 20°C and Z4Y at 20° and 250°C showed elastic behavior prior to failure. Stress-deflection relationship of Z2Y at 250°C showed elastic behavior at the stressing rate over 3.02 MPa/s, too. On the other hand, stress-deflection relationship of 0.301 MPa/s (crosshead speed 0.005 mm/min) showed inelastic behavior. The  $\log \sigma_f$ - $\log \dot{\sigma}$  relationships of Z2Y at 20° and 250°C were linear. Their crack growth parameter  $N$ 's were 67.3 at 20° and 8.7 at 250°C. The  $\log \sigma_f$ - $\log \dot{\sigma}$  relationship of Z4Y at 20°C was linear, too. The  $N$  was 45.3. On the other hand, the fracture strength of Z4Y at 250°C increased below the stressing rate of 3.02 MPa/s and kept a constant value over 3.02 MPa/s, if stressing rate increased. The  $N$  value obtained from the linear relationship below 3.02 MPa/s was 4.7. The fracture surface of Z2Y and Z4Y at 250°C tested at lower stressing rates showed fatigue fracture surface in the vicinity of the tensile surface side. The area of the fatigue fracture surface in Z2Y at stressing rate of 3.01 MPa/s and in Z4Y at 0.302 MPa/s occupied about one-fourth and one-half the fracture surface, respectively. Many cracks were observed on the tensile surface of Z2Y which showed inelastic behavior. The fracture surface of those specimens showed larger fatigue fracture surface than the specimens which showed the elastic behavior.*

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## A Quantitative Evaluation of Diamond and CBN Grains by Overcut Fly-Milling Test

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*An overcut fly-milling test with inclined workpiece was performed to accurately evaluate the cutting and wear characteristics of diamond and CBN grains. Namely, a quantitative evaluation of the chipping and tip wear of a single grain was made by tracing across the grooves with a stylus instrument. The milling experiment was carried out on a surface grinder, in dry, at cutting speeds 12.7 and 38.0 m/s, and feeds 1.3 and 4.0 m/min. The work materials used were hardened SUJ 2 steel, P 20 carbide, alumina,  $Si_3N_4$ , and silicon. The test grains were natural diamond, CBN, green SiC, and fused alumina, where grit size was #12 with an exception of CBN grain (#14-20). The CBN grain had the most high resistance to chipping and attritious wear for cutting SUJ 2 steel. However, the CBN grain as well as A and GC grains could not cut the other work materials due to grain fracture. The high chipping probability of the diamond grains was found on a  $Si_3N_4$  ceramic. Also, the most fine chipping appeared on this work material. The attritious wear of the diamond grain was most remarkable on the P 20 carbide.*

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## Fracture Strength of Silicon Nitride Ceramics with Spherical Voids

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*Void size dependency of the fracture strength of silicon nitride ceramics was studied to develop a failure model from a void. Fracture strength of silicon nitride ceramics including spherical voids generally decreased with increasing void size. A model of a spherical void with a circumferential crack was applied to estimate the crack extension. The length of the circumferential crack was calculated to be about 30  $\mu\text{m}$ , which corresponded to 10-15 times of the average grain diameter. Thus, it was*

assumed that the microcrack extension from a void occurred during loading before the maximum load was reached. Considering the transition of the fracture toughness from single-crystal to polycrystalline, it was concluded that the critical crack extension from the void was attributed to the dependence of the fracture toughness on crack size/grain size ratio.

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### Effects of $ZrO_2$ Ultrafine Particle Additive on the Formation of $Al_2O_3$ from Sulfate

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An aqueous solution of aluminum sulfate containing ultrafine  $ZrO_2$  particles was dehydrated at  $300^\circ C$  to prepare the starting material of  $ZrO_2$ - $Al_2O_3$  composite. The effects of  $ZrO_2$  additive on the decomposition of anhydrous sulfate into  $\eta$ - $Al_2O_3$ , on the  $\eta \rightarrow \alpha$  phase transformation and on the sinterability of produced  $ZrO_2$ - $Al_2O_3$  composite powder were studied. The apparent crystallite size of the anhydrous sulfate decreased with increasing  $ZrO_2$  content from 42 nm (no additive) to 18 nm (20 vol%  $ZrO_2$ ). The composite oxide powder, in which aggregated particle (50-100 nm in diameter) of tetragonal  $ZrO_2$  dispersed in the secondary grains of  $\eta$ - $Al_2O_3$ , was obtained by decomposing the anhydrous sulfate. In the composite powder containing 20 vol%  $ZrO_2$ ,  $\alpha$ - $Al_2O_3$  was found above  $1100^\circ C$ , while it formed above  $1200^\circ C$  for the single component  $Al_2O_3$  powder. The grain growth of  $Al_2O_3$  was prevented by  $ZrO_2$  particles dispersed in the  $\eta$ - $Al_2O_3$  grains; that is, the primary particle size of  $Al_2O_3$  decreased with increasing  $ZrO_2$  content from 120 nm (no additive) to 60 nm (20 vol%  $ZrO_2$ ) after calcination at  $1250^\circ C$  for 1h. The composite powders had very low green densities and they were difficult to sinter. But ball-milling of the composite powders yielded high densities up to 94% for 10 vol%  $ZrO_2$  bodies fired at  $1600^\circ C$  for 1h in air. The  $ZrO_2$  particles were located at the boundary of  $Al_2O_3$ , and the grain size of  $Al_2O_3$  decreased from  $3.0 \mu m$  (no additive) to  $1.2 \mu m$  (10 vol%  $ZrO_2$ ) as the content of  $ZrO_2$  increased.

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### Low Temperature Monoclinic to Tetragonal Transformation of Ultrafine $ZrO_2$ Contacting with Stabilizers

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The monoclinic to tetragonal transformation in  $ZrO_2$  at lower temperatures in ultrafine particles contacting with stabilizers was studied using several powders prepared by hydrolysis of  $ZrOCl_2$  at  $97^\circ$ - $200^\circ C$  and preheat-treated before adding of stabilizers.  $ZrO_2$  particles in the powders were aggregates less than  $1000 \text{ \AA}$  of primary particles of 30-50  $\text{ \AA}$ . The powders, dried under an IR lamp or preheat-treated at  $150^\circ$ - $500^\circ C$ , were mixed with oxides, nitrates or chlorides of Mg, Ca and Y, and heat treated at various temperatures. Identification and determination of monoclinic and tetragonal  $ZrO_2$  were carried out by high temperature and room temperature XRD. Tetragonal  $ZrO_2$  was detected even at  $300^\circ C$  for dried powders and its amount increased with increasing heat treatment temperatures. Preheat treatments without the stabilizers above  $150^\circ C$  resulted in the powders less active in the transformation. The preheat treatments at  $150^\circ$ - $400^\circ C$  decreased the water content and interplanar distance  $d_{111}$  of monoclinic crystal, whereas they did not change specific surface area. Additives other than the stabilizers were not effective for the transformation. The transformation at low temperatures would be due to the large surface energy effect and free energy change by alloying with stabilizers. The surface energy effect is considered to stabilize the tetragonal form at low temperatures when it is alloyed with even a small amount of stabilizers. The alloying at such low temperatures as  $300^\circ C$  would be effected due to large surface area of the  $ZrO_2$  powder, existence of  $H_2O$  and  $OH$  in the crystal and its lattice expansion.

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