

## Metallizing of Silicon Nitride Ceramics

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It was found that a metallized layer can be formed on  $\text{Si}_3\text{N}_4$  ceramics using the paste containing  $\text{Li}_2\text{MoO}_4$  and  $\text{TiO}_2$ . X-ray diffraction pattern and SEM observations revealed that the metallized layer consisted of Mo, TiN,  $\text{Y}_2\text{O}_3 \cdot 2\text{SiO}_2$ , etc., and that the thickness was around  $7\text{ }\mu\text{m}$ . Metallized  $\text{Si}_3\text{N}_4$  specimens were bonded to steel by Ag-Cu solder after Ni plating. The shear strength of the bonded specimens was about 130 MPa at room temperature, 100 MPa at  $300^\circ\text{C}$  and 50 MPa at  $500^\circ\text{C}$ .

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## Oxidation Resistance of AlN Coated Graphite Prepared by Plasma Enhanced CVD

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Uniform and adherent aluminum nitride (AlN) films were coated on graphite substrate by plasma enhanced chemical vapor deposition using a reaction gas mixture of  $\text{AlBr}_3$ ,  $\text{N}_2$ ,  $\text{H}_2$  and Ar. The oxidation resistance test of AlN coated graphite specimen ( $15 \times 10 \times 1\text{ mm}$ ) was carried out in air (relative humidity :  $\sim 50\%$ ) in the temperature range from room temperature to  $1200^\circ\text{C}$ . Thermogravimetric analysis of the specimen showed that the oxidation resistance depends upon the preferred orientation and the film thickness of the AlN film. No oxidation of the graphite substrate occurred even at  $1200^\circ\text{C}$ , when the specimen was coated uniformly by the thick AlN film (thickness  $> 15\text{ }\mu\text{m}$ ) with a high preferred orientation to the c axis. At elevated temperatures ( $1050^\circ\text{C}$ – $1200^\circ\text{C}$ ), these films followed the parabolic oxidation law. The oxidized surface layer of  $\alpha\text{-Al}_2\text{O}_3$  was confirmed to act as a passivation film for further oxidation of AlN film. However, the AlN films having low preferred orientation, were oxidized at  $1200^\circ\text{C}$  approximately linearly with the oxidation time. The latter film was unfavorable for the oxidation-resistant film of graphite.

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## Thermal Shock Testing of Dense SiC by Water-Quenching

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For the evaluation of thermal shock resistance of ceramics, water quench test in which a critical temperature difference,  $\Delta T_c$ , is measured by degradation of strength has often been applied, although

many experimental conditions may influence the result. The main reason for using this method is that the test can be easily carried out. In this report, the effect of variation of heat transfer coefficient on the result was analyzed. Reaction-sintered (RB-) and pressureless-sintered (S-) SiC were used. A semicircular crack was introduced by Knoop hardness indentation on a polished surface of the specimen of  $5 \times 36 \times (2-5)$  mm. Each specimen was quenched in a 50 cm-deep water bath kept at  $290 \pm 2$  K and then its bending strength was measured in 4-point bending. The value of  $\Delta T_c$  was determined from a diagram of bending strength and quench temperature difference. Generally RB-SiC showed larger resistance than S-SiC, mainly because of much higher thermal conductivity of RB-SiC. Thermal stress generated by quenching at the indentation site and the surface heat transfer coefficient  $h$  was calculated from the following relation,

$$\sigma = \frac{E\alpha\Delta T}{1-\nu} f(\beta)$$

where  $\sigma$  is the stress generated,  $\alpha$  the thermal expansion coefficient,  $E$  Young's modulus,  $\Delta T$  the temperature change,  $\nu$  Poisson's ratio and  $\beta(=ah/k)$  is Biot's modulus, with  $k$  the material's thermal conductivity and  $a$  the characteristic length. Contrary to the above, the thermal stress generated was calculated as a function of quench temperature difference using the  $h$  value. Consequently Knoop indented specimen gives more than two  $\Delta T_c$ 's in some case and no well defined  $\Delta T_c$  in other case.

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## Thermal Expansion of Chemically Vapor-Deposited Si<sub>3</sub>N<sub>4</sub>

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The thermal expansion of chemically vapor-deposited (CVD) Si<sub>3</sub>N<sub>4</sub> was investigated from 20° to 1000°C. X-ray and TEM analyses revealed that CVD-Si<sub>3</sub>N<sub>4</sub> samples prepared from a mixture of SiCl<sub>4</sub>, NH<sub>3</sub> and H<sub>2</sub> were only  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> and free from impurity phases even at grain boundaries. The thermal expansion was measured using dilatometry, and X-ray diffraction techniques. The bulk CVD-Si<sub>3</sub>N<sub>4</sub> specimens with (110) and (210) orientations indicated a lower coefficient of thermal expansion than those for the specimens with (222) orientation. This difference in thermal expansion may be the effect of the crystallographic anisotropy. In fact, X-ray diffraction technique revealed that the coefficient of thermal expansion of  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> is lower in the  $a$  axis than in the  $c$  axis.

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## Thermal Conductivity of Pressureless-Sintered SiC with B<sub>4</sub>C and C

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The effects of amount of additives B<sub>4</sub>C and C on the thermal conductivity and bulk density, and the relation between the thermal conductivity and bulk density were described. SiC con-