

Figure 1 Possible stress–separation relationships for a tied crack.

failure mechanism it is reasonable to suggest that a relatively large force is required to start the fibres or particles slipping and thereafter a smaller “frictional” force resists the subsequent slipping, i.e. a stress–separation relationship of the form shown in Fig. 1b. Since “fracture mechanics” is principally concerned with the area under this curve, i.e. the work required to propagate the crack, we can approximate Fig. 1b with 1c, in which the stress parameter  $f_t$  is no longer the strength of the material, but is some lower arbitrary stress. This interpretation suggests that the good fit obtained by putting  $f_t = 5 \text{ MN m}^{-2}$  is no longer inconsistent with the fact that direct tensile strengths of up to  $12.5 \text{ MN m}^{-2}$  were experimentally measured.

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## Thermal shock resistance of silicon nitride

Silicon nitride ( $\text{Si}_3\text{N}_4$ ), silicon carbide (SiC), sialon and aluminium nitride (AlN) have been recognized as leading candidates for high-temperature gas turbine materials [1–9]. Of all the ceramic components in gas turbines, rotor and stator blades have to receive and withstand the most severe mechanical and thermal stresses. Fully densified  $\text{Si}_3\text{N}_4$  and SiC would be limited materials because of the requirement that components should possess high strength at operating temperatures.

Recent developments have produced a hot-pressed high-strength  $\text{Si}_3\text{N}_4$  with 5 wt% yttrium oxide ( $\text{Y}_2\text{O}_3$ ) and 2 wt% aluminium oxide ( $\text{Al}_2\text{O}_3$ ) by applying a new sintering method, the grain-boundary crystallization (GBC) method [9].

A major factor determining the importance of ceramics is their usefulness at high temperatures. Since this often involves rapid heating and cooling of the material, good thermal shock resistance is advantageous. Thermal shock tests currently in use are essentially of two types: determination of the minimum shock to nucleate cracking, and determination of the amount of damage sustained

by a fixed shock or series of shocks.

Initiation of fracture determines materials usefulness in a gas turbine, where a single crack can cause a catastrophic fracture by virtue of the reduction in strength, whereas for a refractory fire brick, the degree of damage is a more important factor.

Cracking is nucleated when the thermal stress,  $\sigma_t$ , reaches fracture stress under shock condition,  $\sigma_f$ . Thermal stress is given by

$$\sigma_t = AE\alpha\Delta T/(1 - \nu), \quad (1)$$

where  $E$  is Young's modulus,  $\alpha$  the coefficient of linear expansion,  $\Delta T$  the fall in temperature and  $\nu$  Poisson's ratio [10].  $A$  is a number between 0 and 1. For an infinitely fast quench, the maximum tensile stress is developed instantaneously and  $A$  equals unity.

In the present work, the thermal shock resistance of  $\text{Si}_3\text{N}_4$  was evaluated by the measurement of critical thermal shock temperature difference  $\Delta T_c$  by water-quenching method, and for this temperature difference, an abrupt strength reduction occurs in accordance with the crack initiation.  $\Delta T_c$  measured by the water-quenching method is convenient for ranking materials under rapid heat-transfer conditions.

3 mm × 3 mm × 30 mm bar specimens were used for the strength measurement. The surface of each specimen was finished with diamond wheel (40 μm grit size). Round edges were obtained with use of SiC water-proof abrasive paper (40 μm). The strength measurement for quenched specimens was

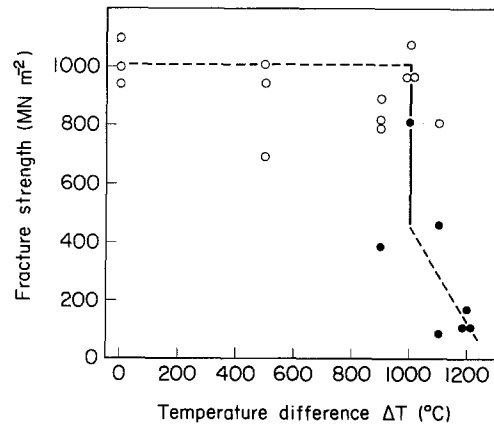


Figure 1 Fracture strength of the specimen after water quenching; ○ no cracks after quenching, ● cracked after quenching.

carried out by means of three-point bend test with a span length of 20 mm and a stress rate of about  $33.3 \text{ MN m}^{-2} \text{ sec}^{-1}$ .

Fig. 1 shows the fracture strength of the GBC- $\text{Si}_3\text{N}_4$  quenched from various temperatures into flowing water kept at 20°C. Strength dropped suddenly, accompanied by crack generation, at the temperature difference of 1000°C. The critical thermal shock temperature,  $\Delta T_c$ , was thus determined to be 1000°C. Exceptional cracking was observed at a temperature difference of 900°C, however the crack ran longitudinally to the specimen and was extraordinarily large compared to the other ones. These aspects seem to show that the crack was due to a local inhomogeneity in the specimen microstructure attributed to the

TABLE I Measured and calculated resistances of various ceramics to thermal fracture under severe quenching

Materials	$\sigma_t \times 10^8$ ( $\text{N m}^{-2}$ )	$E \times 10^{11}$ ( $\text{N m}^{-2}$ )	$\sqrt{\quad}$	$\alpha \times 10^{-6}$ ( $^{\circ}\text{C}^{-1}$ )	$R$ ( $^{\circ}\text{C}$ )	$\Delta T_c$ ( $^{\circ}\text{C}$ )	Description
SiC-1*	1.161	2.361	0.14	4.8	88	265	Recrystallized
SiC-2*	2.128	3.593	0.15	4.0	126	305	Reaction sintered
$\text{Si}_3\text{N}_4$ -1*	1.225	1.258	0.27	3.2	222	460	Reaction sintered
$\text{B}_4\text{C}$ -1*	4.515	4.109	0.26	2.4	339	160	Hot-pressed
$\text{B}_4\text{C}$ -2*	4.515	4.012	0.24	2.4	356	200	Hot-pressed
SiC-3*	6.127	4.160	0.15	3.4	368	415	Hot-pressed
$\text{Si}_3\text{N}_4$ -2*	5.160	2.973	0.25	2.8	465	>900	Hot-pressed
AlN†	7.42	2.79	0.25	4.9	407	250	Hot-pressed
GBC- $\text{Si}_3\text{N}_4$	10.10	3.13‡	0.26‡	2.6	918	1000	(This work)

Dimensions; \*6.35 mm × 3.175 mm × 6.35 mm; †and this work 3 mm × 3 mm × 30 mm.

\*C. C. Seaton [11].

†K. Komeya and F. Noda [3].

‡H. Iwasaki (to be published).

§A. Tsuge *et al.* [12].

sintering processes. It should be noted that no cracks were initiated in any of three specimens subjected to thermal shock of  $\Delta T = 1100^\circ\text{C}$ . This fact suggests the possibility of a  $\Delta T_c$  of over  $1100^\circ\text{C}$  in the GBC-Si<sub>3</sub>N<sub>4</sub>.

Values of  $\Delta T_c$  for the present GBC-Si<sub>3</sub>N<sub>4</sub> and some various structural ceramics [3, 11] measured by a similar method, are shown in Table I. It is clear that  $\Delta T_c$  values of Si<sub>3</sub>N<sub>4</sub> ceramics, especially GBC-Si<sub>3</sub>N<sub>4</sub>, are higher than others, and that the GBC-Si<sub>3</sub>N<sub>4</sub> is an excellent thermal shock-resistant material. This is based on both high strength value and low thermal expansion coefficient [9].

Calculated thermal resistances of some structural materials are also listed in Table I, where resistance  $R$  is calculated using [11].

$$\Delta T_c \propto R = \frac{\sigma_f(1-\nu)}{E\alpha} \quad (2)$$

Except for the case of GBC-Si<sub>3</sub>N<sub>4</sub>, where measured  $\Delta T_c$  is approximately equal to the calculated  $R$ , the relationship between  $\Delta T_c$  and  $R$  is not precise. However, it seems to be apparent that, whereas  $\Delta T_c$  values of both B<sub>4</sub>C and AlN are smaller than respective  $R$  values, SiC and Si<sub>3</sub>N<sub>4</sub> have larger  $\Delta T_c$  than  $R$  values respectively. It should be noted that  $R/\Delta T_c$  ratio approaches 1, i.e.  $R = \Delta T_c$ , with increasing  $\Delta T_c$ , but the explanations of this phenomenon is not yet available.

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