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Study on thermal shock in Si_3N_4/SiC platelets

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Intensive investigations have been carried out to improve the mechanical behavior of silicon nitride. Studies show an increase of mechanical resistance and creep resistance through the incorporation of reinforcement compounds such as fibers, whiskers and platelets [1–6], as well as through the use of additives, which lead to a partial crystallization of the grain boundary vitreous phase [7–9].

In recent years, platelets have been used more than whiskers due to the toxicity of the latter. Alumina and silicon carbide platelets have been used to reinforce monolithic ceramic matrices [1–3, 10–12] and vitreous ceramics [13]. In both cases, the mechanical properties showed an improvement over the base material.

Silicon nitride is a material with great potential under high temperatures. It is therefore important that it should resist thermal gradients. While there are several works in the literature related to thermal shock resistance of pure silicon nitride, none report the effects caused by sudden temperature changes on the residual mechanical resistance of silicon nitride reinforced by silicon carbide platelets.

The purpose of this work is to investigate the damage occurring in this composite material when submitted to thermal shock.

The material studied was fabricated by conventional hot-pressing of a silicon nitride powder (SNE 10, Ube Corp., Japan) containing as sintering additives 5 and 2 wt % of aluminum and yttrium oxide, respectively. As the reinforced phase, 20 v/o of monocrystalline silicon carbide platelets were added (32–50 μm, American Matrix, Knoxville, USA). Fig. 1 shows a scanning electronic microscope (SEM) micrograph of *monocrystalline* silicon carbide platelets used in this work. One can easily notice that the platelets present a hexagonal format. The powder mixture of silicon nitride and silicon carbide platelets was pressed at 1800 °C in graphite dies. Details on the preparation of this composite have been described elsewhere [1, 14].

Thermal shock treatment was undertaken by heating the samples in a vertical tubular furnace to a particular temperature. The temperature was measured by a cromel–alumel thermocouple beside the samples. The samples (having dimensions of $4 \times 4 \times 50\ \mathrm{mm}^3$) considered for mechanical resistance measurement were ground and polished and had rounded edges. After 10 min under constant temperature, the samples were released and allowed to fall into a water reservoir at 25 °C.

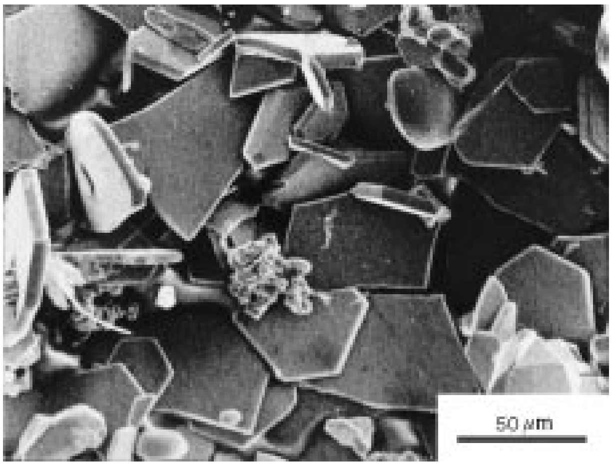

Figure 1 SiC platelets.

After thermal shock, residual flexure strength tests by the four-point bending test were performed using a cross-head speed of 0.05 mm min^{-1} and a standard testing machine (Instron), with inner and outer spans of 20 (*e*) and 40 mm (*l*), respectively (Fig. 2). To evaluate the thermal shock behavior of the composite, two parameters, R and R^{IV}, were calculated. The determination of these two parameters are based on Hasselman's theory [15].

After the rupture, the width and length of the samples were measured in the region of the fracture.

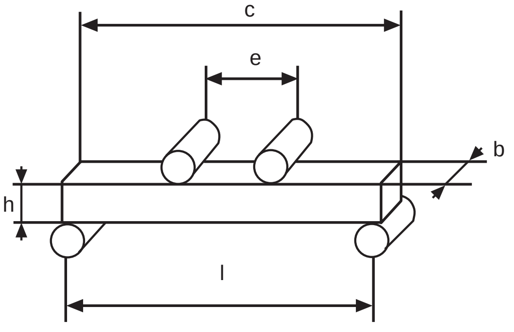

Figure 2 Schematic of loading geometry for strength measurements.

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The residual bending strength σ is given by the relationship

$$\sigma = 3/2[P(1-e)/bh^2] \qquad (1)$$

where P is the fracture load and b and h are dimensions of the sample, according to Fig. 2.

Specimens for SEM observation were prepared by polishing and plasma etching.

Figs 3 and 4 show the microstructure of the investigated composite. Fig. 3 shows a region containing only silicon nitride grains, and it can be seen that those grains present the usual "needle" grain structure. The platelets show a preferred orientation perpendicular to the direction of hot-pressing. It can also be observed that the platelets are equally spaced and uniformly distributed in the matrix (Fig. 4).

Fig. 5 shows the values of residual strength after thermal shock of silicon nitride reinforced by silicon carbide platelets in comparison with the literature data for pure silicon nitride samples [16].

The shaded region corresponds to the range of values for hot-pressed silicon nitride (HPSN). It can be seen that ΔT_c (the critical value of ΔT) for platelet reinforced–silicon nitride is in the range (right side of the dashed region) of values found for the pure silicon nitride. Therefore, no improvement is observed on adding the silicon carbide platelets.

Fig. 5 also shows a three-stage behavior of the bending strength. In the first stage ($\Delta T < 600$ °C), the thermal shock treatment does not affect this mechanical property. The second stage, in the range of ΔT_c ($\approx$600 °C), corresponds to a significant and sudden decrease of the bending strength. In the third stage, the flexure resistance holds a constant level for $\Delta T > 600$ °C but significantly lower than the level of the first stage. The presence of these three stages is characteristic of the Hasselman's model [15] for the thermal shock.

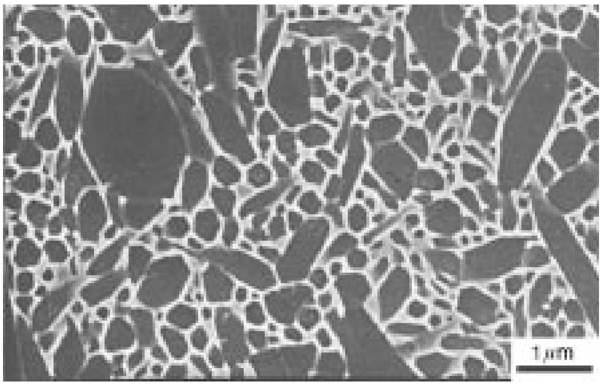

Figure 3 Plasma-etched specimen showing only silicon nitride grains.

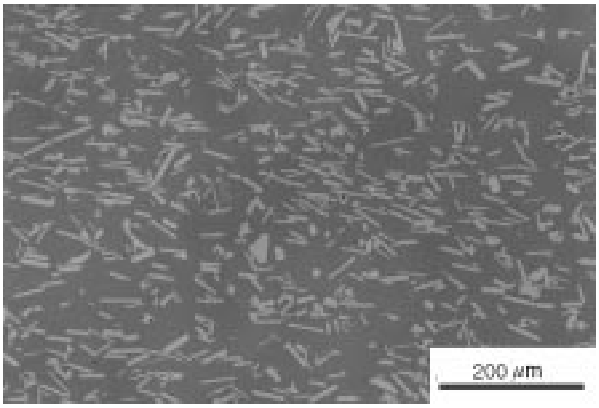

Figure 4 Optical micrograph of Si_3N_4/SiC platelets.

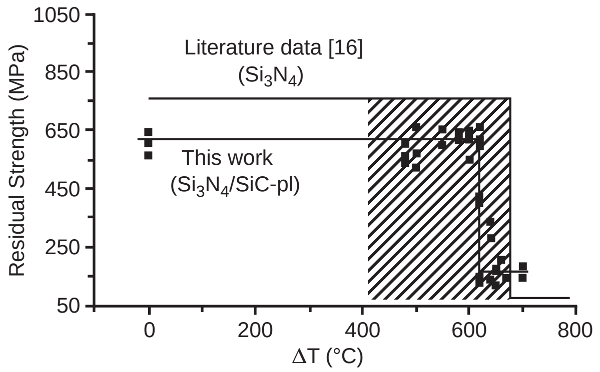

Figure 5 Bending strength of Si_3N_4/SiC platelets and pure silicon nitride [16] after thermal shock.

The thermal stress fracture resistance parameter R and the thermal stress damage resistance parameter R^{IV} were calculated by

$$R = \sigma(1-\mu)/E\alpha \qquad (2)$$

$$R^{IV} = E\gamma_f/[\sigma^2(1-\mu)] \approx (K_{IC}/\sigma)^2/(1-\mu) \qquad (3)$$

where K_{IC} is the fracture toughness of the material, E is the elastic modulus, α is the thermal expansion coefficient, μ the Poisson's modulus (assumed to be 0.27), γ_f the fracture surface energy and ΔT the temperature difference between the surface and the center of the sample. The parameter R^{IV} represents the resistance of the material to catastrophic crack propagation.

It can be seen from Equations 2 and 3 that an increase in R causes a decrease in R^{IV} and vice versa. Therefore, either the material is resistant against crack nucleation or crack propagation.

Table I compares the values of R and R^{IV} (calculated from Equations 2 and 3) found in this work with data from the literature [1, 16–19]. The values of R^{IV} were calculated for pure silicon nitride through the use of $\mu = 0.23$. By comparing the values of R and R^{IV}, one sees that the platelet-reinforced silicon nitride is less resistant to thermal shock (R) than the pure silicon nitride but presents a higher resistance to thermal shock damage (R^{IV}).

These results indicate that the crack propagation can be improved by adding silicon carbide platelets.

TABLE I Values of the parameters R and R^{IV}

R (°C)	R^{IV} (μm)	Literature
412	200	This work
625	—	[18][a]
650	—	[19][a]
687	121	[17][a]
330	164	[17][b]
300–780	98–102	[16][a]

[a] pure silicon nitride.

[b] Si_3N_4/SiC whisker.

In contrast, the use of platelets decreases the fracture strength at room temperature and also the resistance to crack initiation. Similar behavior was also found by Jia *et al.* [17] (Table I), who observed that the use of silicon carbide whiskers reduced the value of R and increased R^{IV}. The lower values of R may be based on the fact that the original cracks in the platelet composite might be larger than the cracks caused by thermal shock before the critical temperature difference.

It can also be seen from Table I that the value of $R = 412\ ^{\circ}\mathrm{C}$ is smaller than the observed critical temperature difference $\Delta T_{\mathrm{c}} = 600\ ^{\circ}\mathrm{C}$ (Fig. 5). This behavior was also found by Jia *et al.* [17] and was attributed to anisotropic thermal expansion of the hot-pressed composite and to water-quenching speed.

The results presented in this work show that the silicon carbide platelet reinforced with silicon nitride is a composite material that can be used for thermal shock applications in which materials with a high resistance against crack growth but not to crack initiation are needed.

The results lead to the following conclusions:

1. The platelet composite material has a value of ΔT_{c} comparable to that of pure silicon nitride.
2. The thermal shock behavior of the platelet composite can be described by Hasselman's model [15].
3. The addition of silicon carbide platelets causes a decrease in the resistance against the crack initiation (R) and an improvement in the resistance against crack growth (R^{IV}).

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