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MICROSTRUCTURE/MECHANICAL–PROPERTY RELATIONSHIPS AND R–CURVE BEHAVIOR IN $Si_3N_4/Si_3N_4(w)$ COMPOSITES

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Abstract

Fracture toughness of Si_3N_4–whisker–reinforced Si_3N_4–matrix composites were evaluated with various whisker contents. Toughness was observed to increase with increasing whisker content, reaching a maximum value of 8.8 MPa√m at 5 vol.% whisker content. Additional whisker content caused reduced toughness. A model based on microstructural features indicates that the observed dependence of toughness on whisker content is due to both whisker–toughening and matrix–grain–size effects. Composites with 5 vol.% whisker content showed a rising crack growth resistance (R–curve) behavior. This is believed to be a combined effect of crack bridging and crack deflection due to both elongated grains and reinforcing whiskers.

Key Words: Ceramic Materials; Fracture Toughness; Composites; Microstructure; R-Curve; Silicon Nitride; Whiskers.

Introduction

Silicon nitride (Si_3N_4) is one of the most widely studied structural ceramic material because of its potential for high strength and toughness. Several studies have been conducted to improve its toughness by SiC–whisker additions [1–3]. However, in many cases, satisfactory improvements in properties were not achieved because of the adverse interfacial reactions between the whiskers and matrix. Contradictable results for strength and toughness have been observed [1–4]. In view of these problems, single phase, Si_3N_4–whiskers were used to reinforce Si_3N_4–matrix in an effort to improve its toughness. Room temperature fracture toughness was evaluated and correlated with microstructure. A study was conducted to investigate the rising crack growth resistance (R–curve) behavior, i.e., increase in critical stress intensity with increasing crack length in these composites.

Experimental Procedure

Si_3N_4 composites were fabricated by mixing α–Si_3N_4 powder (Grade: UBE SN–E10, UBE Chemical Industries, Japan) with various amounts of β–Si_3N_4 whiskers and subsequently hot pressing the composite powder mixture. Initially, α–Si_3N_4 powder was mixed with 3 wt.% MgO, in the form of $Mg(NO_3)_2.6H_2O$ as a sintering aid, in an ethanol suspension. This mixture was then milled for approximately 16 hours using high density, high purity alumina milling media. Subsequently, 5–30% β–Si_3N_4 whiskers (0.6 µm diameter and 45 µm length) were added and milled for an additional 3 hours. The slurry was dried in a pyrex pan on a low heat. The dry composite powder was hot pressed into approximately 1.6 in. diameter disks in a boron nitride coated graphite die at 1650-1750°C and 32 MPa for 2h in a flowing nitrogen atmosphere.

Density of the fabricated specimens measured using Archimedes' method were over 96% of the theoretical density. Grain size was evaluated by transmission electron microscopy (Model# CMX-100, JEOL, Japan) using linear intercept method. Elastic modulus evaluated using ultrasonic velocity measurements using a pulse–echo technique[5] ranged from 310–330 GPa. Vickers hardness was measured by means of hardness tester (Model # V-100A, Leco Corp., Warrendale, PA, USA) with an indent load of 10 kg. Fracture toughness was evaluated by indentation techniques [6,7] for each whisker content. Two independent methods: indentation[8] and modified indentation–bend[9] were employed to investigate R–curve behavior for the composites. First, three indentations at identical loads were made within the loading span of the flexure specimen and their crack lengths measured. Subsequently, specimens were loaded in four-point-bend mode on a universal testing system (Instron Corp., Canton, MA, USA) and fractured. This procedure was carried out at different indentation loads. R–Curve behavior was established using the two methods from indentation loads, corresponding intial crack lengths and fracture stresses.

Results and Discussion

Figure 1 shows a typical transmission electron micrograph of Si_3N_4--5 vol.% Si_3N_4(w) composites showing the grain microstructure. It was observed that average matrix grain size decreased from ~ 0.325 μm to 0.125 μm with 0 to 15 vol.% whisker additions, respectively. The decrease in grain size is believed to be due to pinning of matrix grains by whiskers[10].

Figure 2 shows the variation in the fracture toughness as a function of whisker content. With an addition of 5 vol% whiskers, fracture toughness increased by 35% from 6.5 to 8.8 MPa√m. As whisker content was further increased fracture toughness decreased. This behavior can qualitatively be explained as follows. The toughness of composites depends on both toughness of matrix and the toughening contribution from whiskers. The toughening due to whiskers results from whisker pullout and crack deflection mechanisms, as seen in the SEM micrographs (Fig. 3 A&B). Both whisker pullout[11] and crack deflection[12] mechanisms provide a positive dependence of fracture toughness on whisker content. On the other hand, the increase in whisker content was observed to decrease matrix grain size. The effect of change in matrix grain size from D° to D on the change in fracture toughness (K_{Ic}) can be predicted by[13]:

$$\Delta K_{Ic} = C \; K_{Ic}^{o}\left(\frac{D}{D^{o}} - 1\right) \tag{1}$$

where C is a geometrical factor and K_{Ico} is the fracture toughness of the material with grain size equal to D°. This equation suggests that a decrease in grain size associated with an increase in whisker content is expected to result in a decrease in fracture toughness. A schematic representation of these two competing effects (grain size and whisker toughneing effects) and the resulting toughness distribution are shown in Figure 4. This resulting toughness distribution is similar to the measured dependence of toughness on whisker content (Fig. 2).

In view of the fact that composites with 5 vol.% whisker reinforcements exhibited the largest increase in fracture toughness; therefore, it was appropriate to select these composites for the evaluation of crack growth resistance (R–curve) studies. Rising crack growth resistance (K_R) behavior was first obtained from as–indented crack lengths based on the following equilibrium relationship[8]:

$$K_R = \frac{\chi P}{c_o^{3/2}} \qquad (2)$$

where P is the indentation load and c_o is the corresponding equilibrium crack length, χ is a dimensionless constant and was determined as 0.105 from elastic modulus (312 GPa) and hardness (17.7 GPa) of the composite[8,14]:.

In addition, R–curve behavior can be described by an empirical power–law form as follows[9]:

$$K_R = Ac^n \qquad (3)$$

where K_R is the critical stress intensity, c is the crack length, and A & n are constants dependent on the material. In the present study, parameters A & n were evaluated from modified indentation–strength method in which specimens were first indented at a load, P, and then tested in four–point–bending mode to obtain fracture strength (σ_f). Krause[9] has shown that for materials exhibiting R–curve behavior (as given by Eqn. 2) it is possible to correlate fracture strength, σ_f, to indentation load, P, as follows[14]:

$$\sigma_f = \frac{A\pi^{1/2}(3+2n)}{8\,\Omega}\left[\frac{4\chi P}{A(1-2n)}\right]^{\frac{2n-1}{2n+3}} \qquad (4)$$

where, Ω is a surface correction factor taken to be 1.21 for indents much smaller compared to specimen dimensions[15], χ is same as described above. Thus, R–curve parameters n and A, can be estimated from the slope and intercept of a log σ_f vs log P plot. It should be noted that Eqn. 4 is valid only for the cases where $n<0.5$. Figure 5 shows the variation of fracture stress as a function of the indentation load in 5 vol.% Si_3N_4 whisker-reinforced Si_3N_4 matrix composites. Parameters n and A were obtained as 0.126 and 24.3 MPa $m^{0.5-n}$, respectively.

R–curve behaviors of Si_3N_4–5 vol.% Si_3N_4(w) composites obtained from the two techniques are shown in Figure 6. Solid line represents power–law variation of crack growth resistance as given from Eqn. 2 using parameters A and n obtained from indentation–strength technique. Whereas, symbols represent data obtained from as–indented crack length measurements. Results from both techniques agree well. Modest increases in toughness were observed from 7.9 MPa$\sqrt{m}$ to 9.0 MPa$\sqrt{m}$ as crack length increased from 155 μm to 365 μm . Increase in fracture toughness with increasing crack length is attributed to various crack wake mechanisms such as whisker and grain bridging of crack faces[15]. These bridging mechanisms lead to crack–closure stresses which act in shielding the crack tip from the applied stresses[11,14,15]. Figure 7 shows clear evidence of grain bridging mechanism in operation. Moreover, it is also possible that toughening mechanisms such as crack deflection will also contribute towards toughness enhancement. This is more so in this material because of the large elongated grains of silicon nitride matrix. Therefore, these results suggests that crack growth resistance (R–curve) behavior is important and needs to be accounted in reliabiltiy studies for design of structural components with these composites.

Conclusions

The fracture toughness of $Si_3N_4/Si_3N_4(w)$ composites was found to increase from 6.5 MPa$\sqrt{}$m to 8.8 MPa$\sqrt{}$m as a result of 5 vol.% whisker additions. The toughness of the composites is believed to be controlled by the whisker strengthening and the matrix grain size. Crack growth resistance behavior was observed for composites with 5 vol.% whiskers. Increase in critical stress intensities from 7.9–9.0 MPa$\sqrt{}$m were observed as crack lengths increased from 155–365 µm. This behavior is attributed to whisker and grain bridging and as well as crack deflection mechanisms.

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Figure 1. Microstructure of Si3N4-5 vol.% Si3N4(w) Composites.

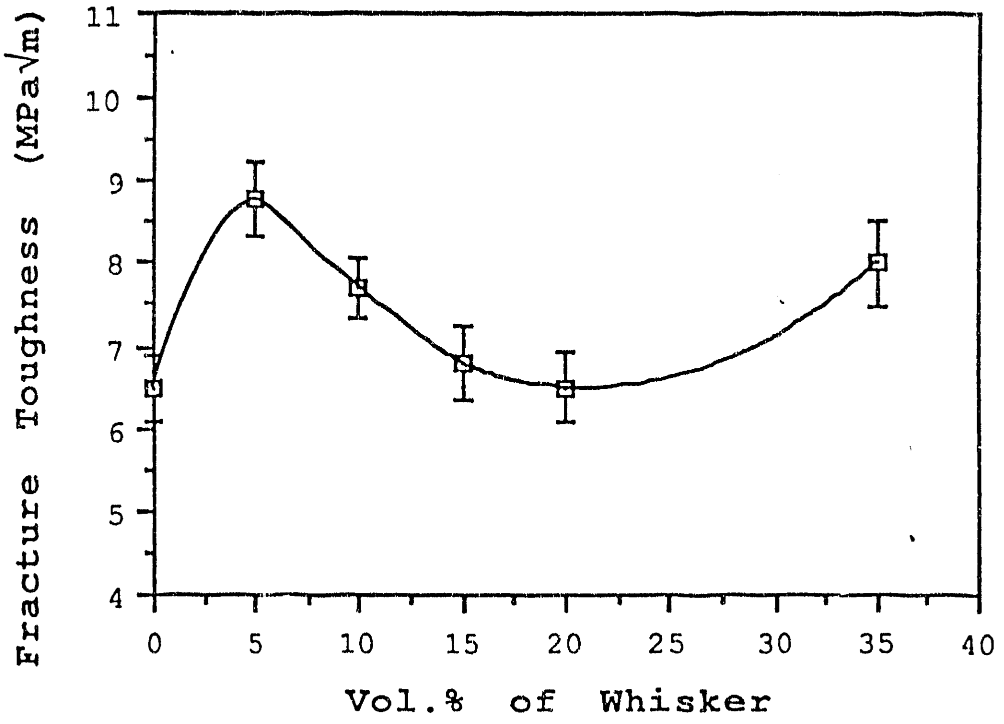

Figure 2. Variation of Fracture Toughness of Si3N4-Whisker-Reinforced Si3N4 Composites as a Function of Si3N4 Whisker Content.

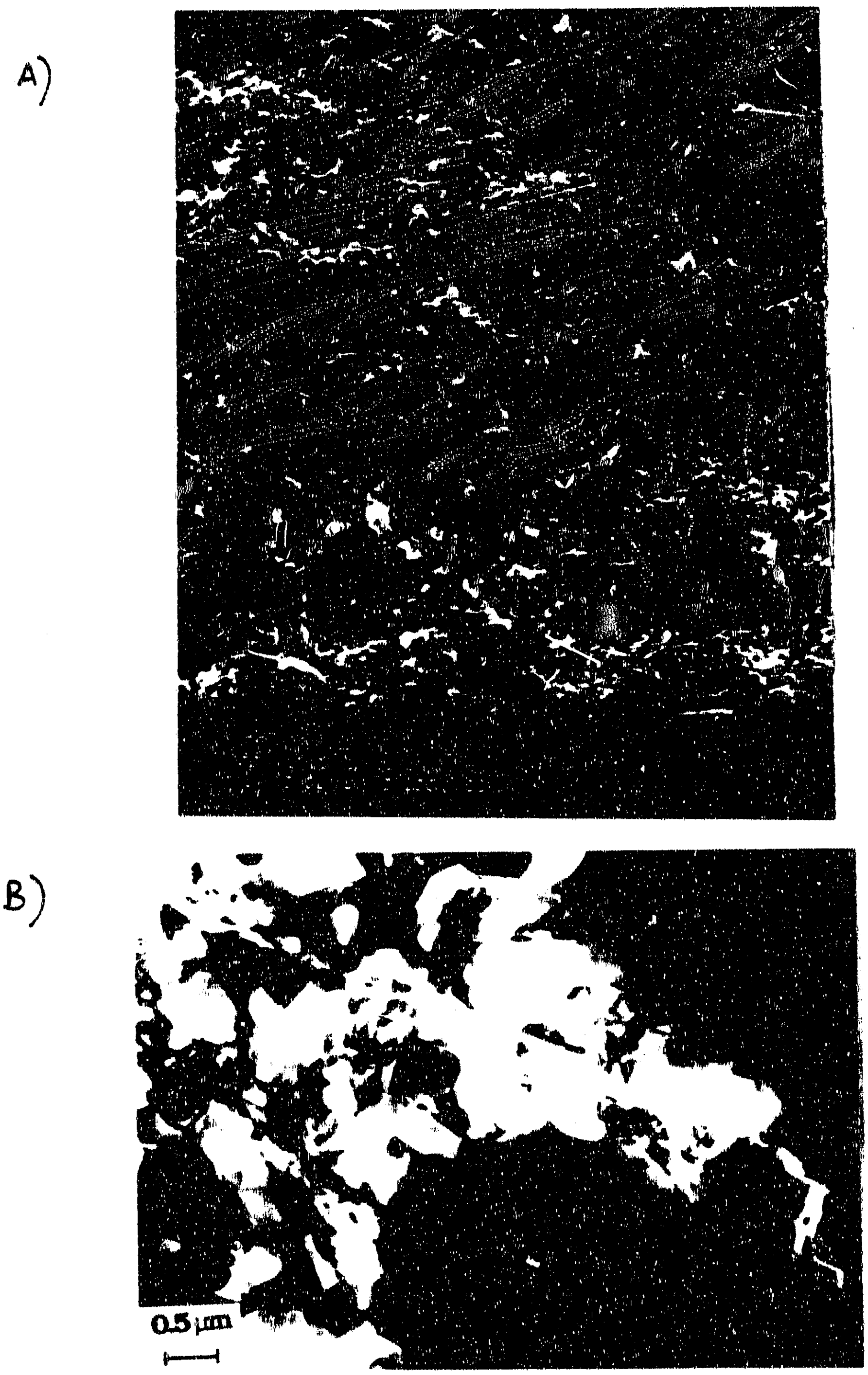

Figure 3. Micrographs of the Si_3N_4/Si_3N_4(w) Composites Showing (A) Whisker Pullout.(B) Crack Deflection.

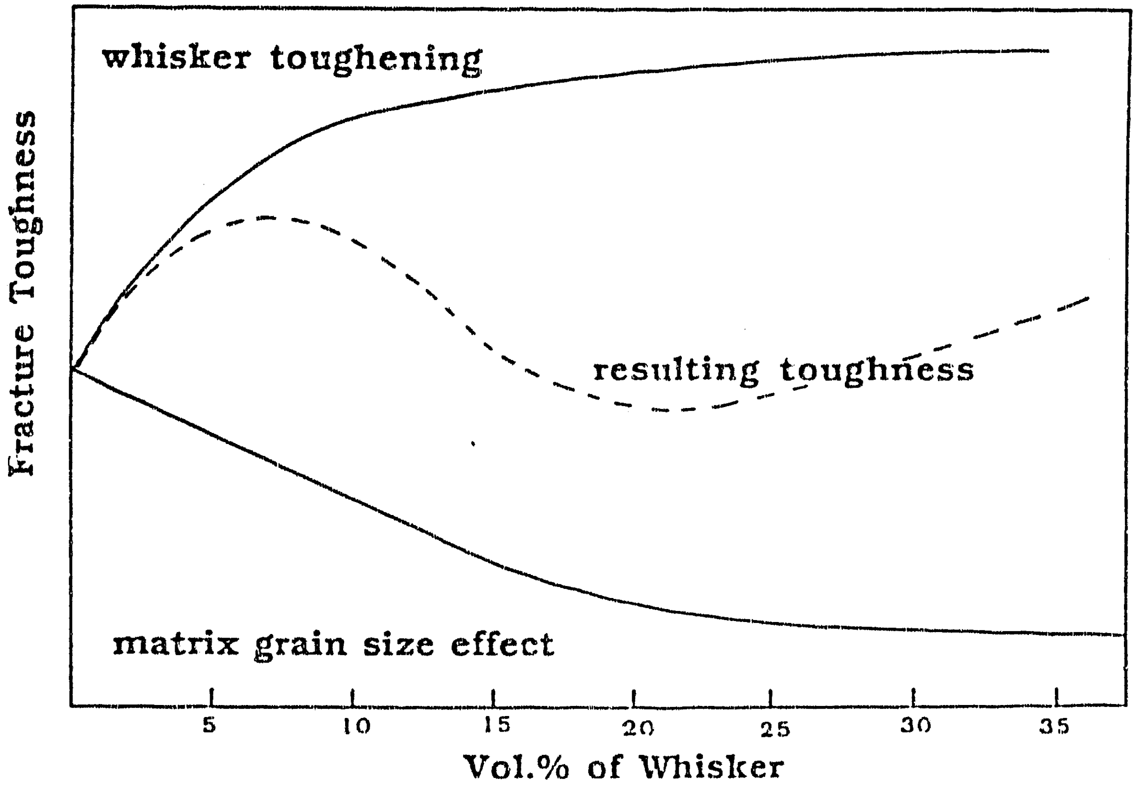

Figure 4. Schematic Representation of the Toughening Behavior of the Si_3N_4-Whisker-Reinforced Si_3N_4 Composites.

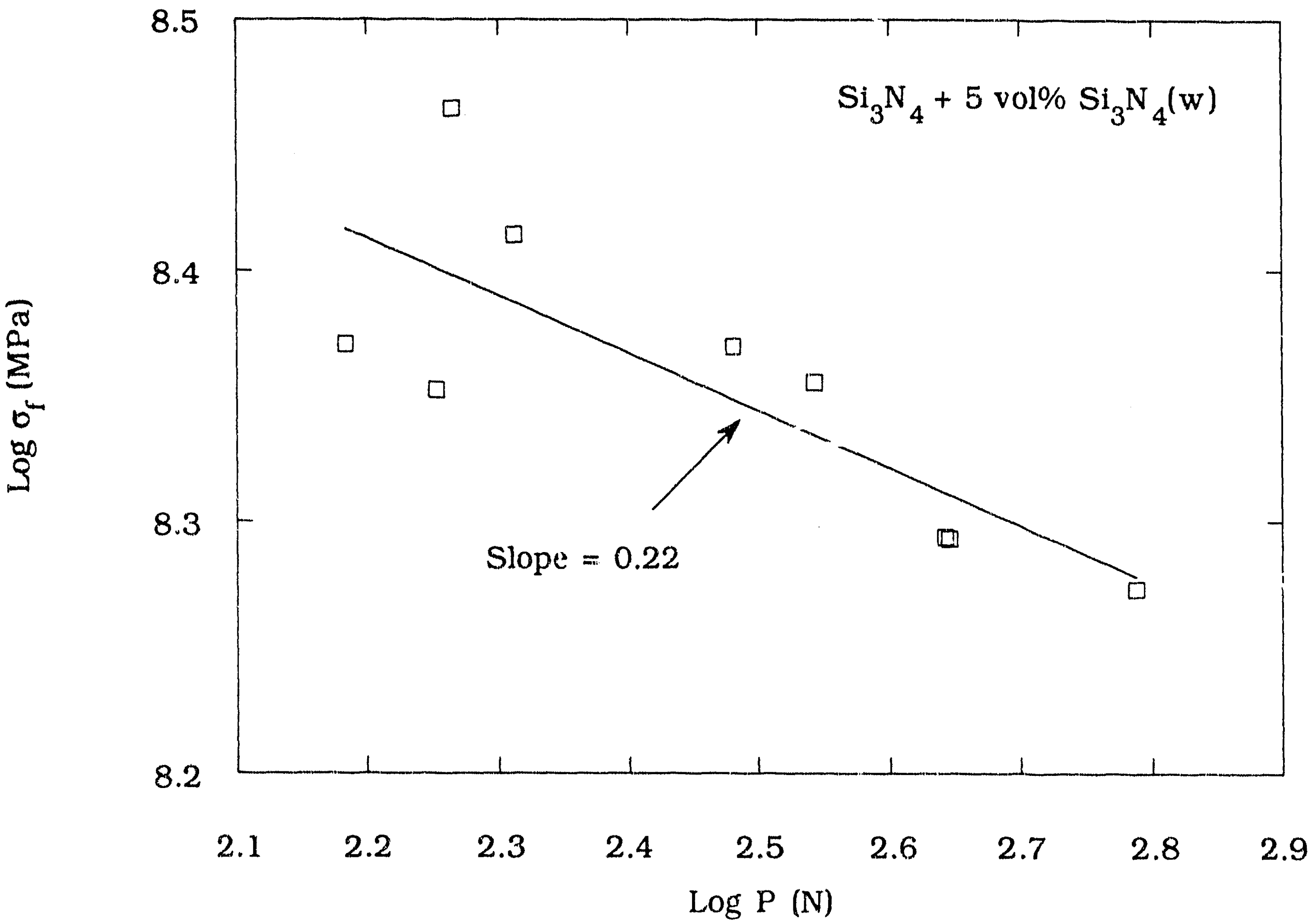

Figure 5. Variation of Fracture Strength With Indentation Load for Si_3N_4-5 vol.% Si_3N_4(w) Composites Composites.

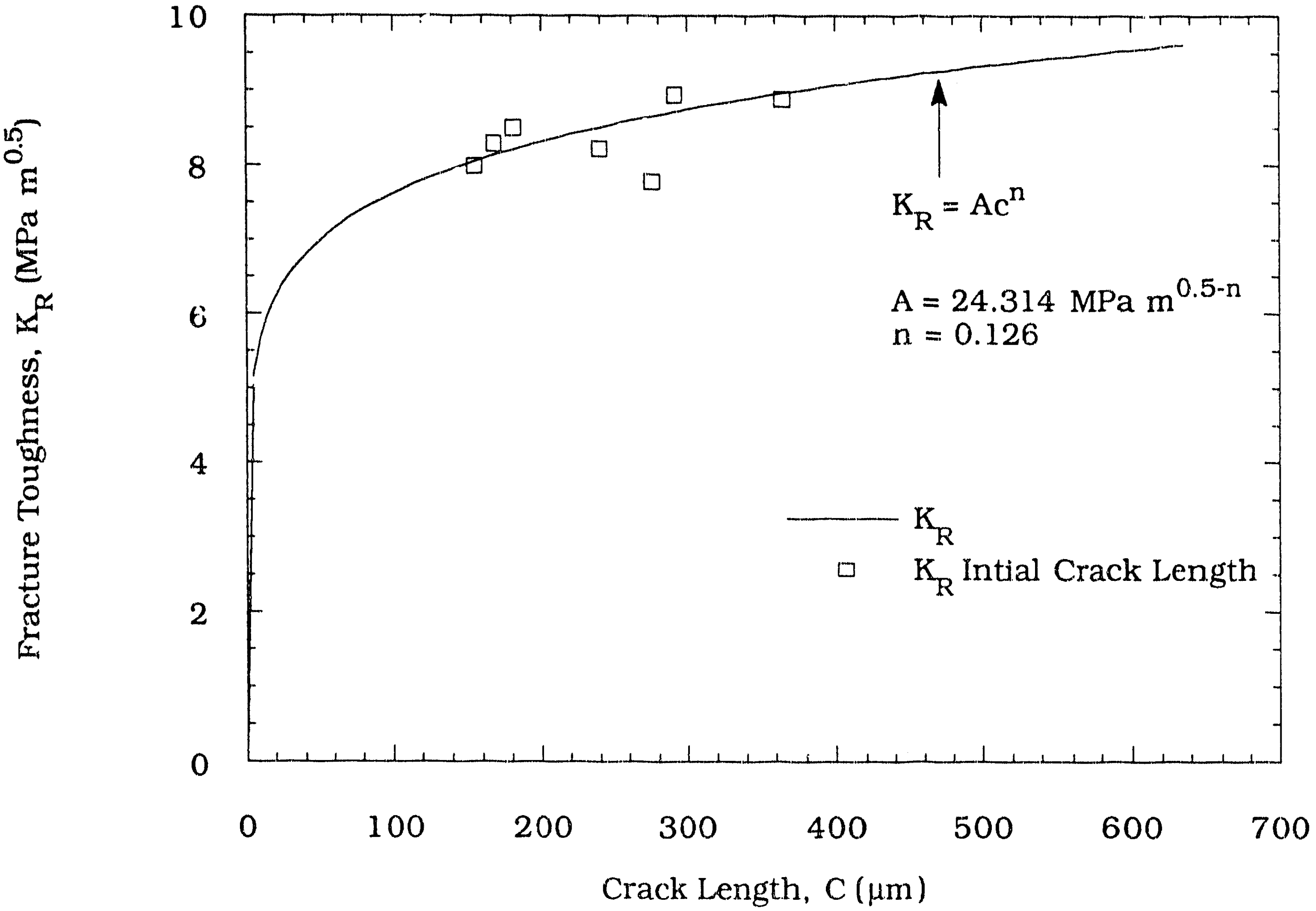

Figure 6. Rising Crack Growth Resistance (R–Curve) Behavior in Si3N4-5 vol.% Si3N4(w) Composites

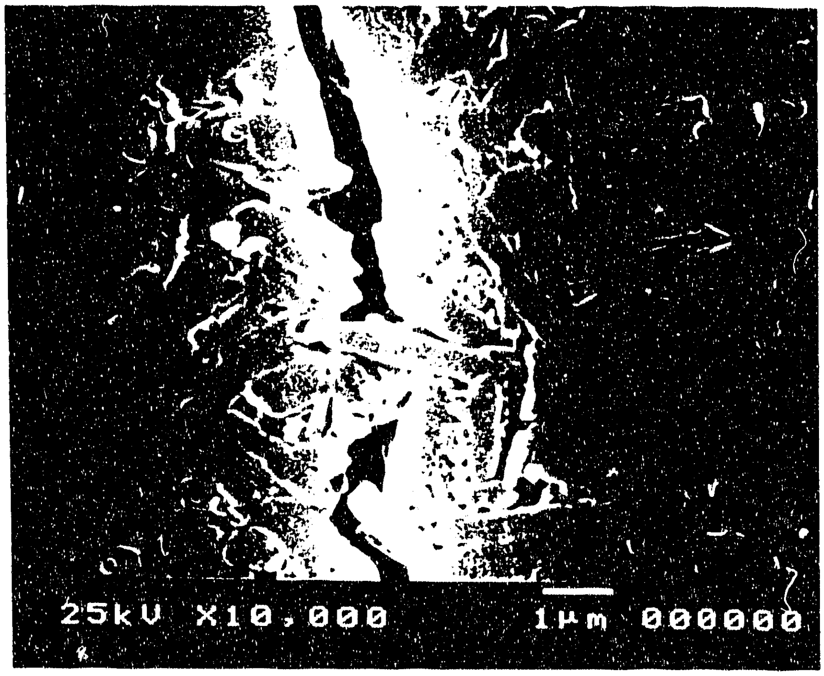

Figure 7. Evidence of Grain Bridging in the Wake of a Crack in Si3N4-5 vol.% Si3N4(w) Composites Composites.

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