Effect of Thermally Induced Stress on Fracture Toughness of SiC Fiber-Glass Matrix Composites

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Effect of thermally induced stress on fracture toughness was examined using a model composite composed of unidirectionally-aligned continuous monolayer SiC fiber and glass matrix. To study the effect of relative coefficient of thermal expansion (CTE), two different kinds of composites with the condition of $\alpha_f^L > \alpha_m$ where α_f^L and α_m denote CTEs of the fiber and matrix and L denotes longitudinal direction of the fiber, were fabricated by changing CTE of the matrix. The fracture toughness of the composite was obtained from pop-in load, which corresponded to initial matrix cracking at the notch root. It was found that the fracture toughness of the composite increased with increase the longitudinal thermally induced compressive stress in the matrix and the result is explained by simple theoretical analysis. The result suggests that the thermally induced compressive stress in the matrix is a key mechanism increasing the fracture toughness of the fiber-reinforced ceramic matrix composites.

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I. Introduction

There is evidence that continuous ceramic fiber-reinforced glass or ceramic matrix composites yield superior mechanical properties compared with un-reinforced matrix materials. Ceramic fiber-reinforced glass, glass ceramics, and ceramics have been extensively studied as candidates of tough structural ceramic materials. Such composites normally exhibit initial cracking due to matrix fracture and further crack growth occurs at substantially high fracture resistance with R curve behavior. The fracture toughness obtained from the initial cracking load, determinates the condition of the first fracture event from the root of a notch and this value is viewed as the minimum toughness of the composite⁽¹⁾.

Theoretically, the thermally induced compressive stress in the matrix phase is an important factor in increasing fracture resistance of composites such as toughness, cracking stress, etc.⁽²⁾⁻⁽⁵⁾. Reports also suggest that thermally induced residual stress affect the initial matrix cracking of fiber-reinforced ceramic matrix composites; these stresses also showed significant effects of coefficient of thermal expansion (CTE) mismatch on the cracking stress level and progressive damage process after the onset of a cracking⁽²⁾⁻⁽⁵⁾.

To the author's knowledge, however, there are no experimental reports on the effect of thermally induced stress on the fracture toughness of fiber-reinforced glass matrix composites. This experimental study looked at the effects of such stress on the fracture toughness using well-identified model composite materials. This approach was expected to yield more fundamental information and descriptions on this subject.

II. Experimental Procedure

A composite with a single array of aligned continuous SiC fiber (Textron Inc., Lowell; MA, USA, SCS-2) reinforced glass system was fabricated as a model material. The single layer SiC fiber was placed at the center of the through thickness direction in the composite. The SiC fiber consists essentially of a SiC sheath with an outer diameter of 142 μm surrounding a pyrolytic graphite-coated carbon core with a diameter of 37 μm . The fiber has an approximately 1 μm thick single coating layer consisting of fine SiC particles embedded in pyrolytic carbon⁽⁶⁾. As the matrix, two types of glass, soda-borosilicate glass (Corning Glass Works, NY, USA, Code: #7740, Pyrex) and 96 mass% silica glass (Corning Glass Works, NY, USA, Code: #9814, Vycor) were used to vary CTE. Typical properties of the fiber and glass matrices are listed in Table 1⁽⁷⁾⁻⁽¹⁰⁾.

The SiC fibers were aligned with uniform spacing by hand lay-up process and then the fiber was sandwiched between the 1.5 mm thick glass plates. The stacked materials were put into a boron nitride-lined graphite die and the materials were hot-pressed in a nitrogen atmosphere of 0.1 MPa. The maximum hot-pressing temperature was 850 and 1400 K for the Pyrex and Vycor glass, respectively. A pressure of 5 MPa for 30 min was applied during the forming process after the temperatures reached the set point. Then, the composite specimen was furnace cooled in the hot press die to room temperature in nitrogen atmosphere. The dimensions of the as hot-pressed composite panels were 75 mm by 75 mm by 2.9 mm thick. Existence of the thermally induced stress in the matrix was observed using transmission

Fig. 1 Polished section of SiC-Pyrex matrix composite, $f=0.05$.

Fig. 2 Shape and dimensions of DCB specimen.

photoelasticity technique. The specimens for the photoelasticity analysis were ≈ 1 mm.

Figure 1 shows a typical polished section of the as pressed composite perpendicular to the fiber axis. The center to center spacing of the SiC fiber was approximately $200 \mu\text{m}$ which corresponded to the shortest mean surface to surface distance $\approx 80 \mu\text{m}$. The volume fraction and fiber spacing were the same for the two kinds of composites.

The hot-pressed composite plates were mechanically ground and cut into a double cantilever beam (DCB) specimen. A side groove was introduced for one side of

the DCB specimen. The shape and dimensions of the specimens are shown in Fig. 2. The final volume fraction, f , of the DCB specimen at the side grooved area was ≈ 0.05 . The notch was cut by a thin diamond cutting blade. Width of the notch of the DCB specimen was approximately $\approx 200 \mu\text{m}$ and typical notch depth (a) to width (w) ratio (a/w) for the DCB specimen was approximately ≈ 0.2 . Special care was taken to locate the notch tip between adjacent fibers i.e., in the matrix phase[†]. For the purpose of comparison, un-reinforced matrix glass materials, both Pyrex and Vycor glass plates, were cut into the DCB specimen and the monolithic specimens were heat-treated by the same processing schedule as the composites. Notching of the un-reinforced glass specimen was done after the heat-treatment at the same a/w ratio as the composite specimens.

The fracture toughness test was conducted with a stiff pin-loading system using an Instron testing machine (maximum machine capacity: 500 kN, maximum load cell capacity 1 kN) in air at 300 K. The crosshead movement of the machine for both specimens was 0.05 mm/min. At least five specimens were tested for each composite. The load-displacement curve was recorded and crack growth behavior from the root of the notch, during and after the test, was measured by a low magnification traveling optical microscope. The crack extension length of the composite was defined as the crack length of the matrix observed at the side-grooved surface. After the tests, detailed the fracture behavior was observed by a scanning electron microscope (SEM).

Table 1 Properties of SiC fiber and glass matrix.

Properties	SiC fiber*	Pyrex glass**	Vycor glass***
Young's Modulus (GPa)	407	62.7	66.1
Tensile Strength (MPa)	3450	—	—
Poisson's Ratio	0.15	0.2	0.19
Coefficient of Thermal Expansion ($10^{-6}/\text{K}$)	5.50(//)	3.25(273–573 K)	0.75(0–473 K)
Strain Point (K)	—	783	1093

* Textron Corp.: SCS-2

** Corning Glass Works: Code #7740 (Pyrex)

*** Corning Glass Works: Code #7913 (Vycor)

[†] Data obtained under this condition was used in this experiment. More than 15 specimens were tested, however, only 1/3 of them satisfied this condition. The data (fracture toughness) obtained under insufficient condition was within $\pm 16\%$ of the sufficient composite.

III. Experimental Results

1. Crack growth behavior

Typical load-displacement curves for the composites (solid line) and the corresponding un-reinforced glass (broken line) obtained from the DCB specimen are shown in Fig. 3.

In the SiC fiber-reinforced Pyrex matrix composite, the matrix cracking at the root of notch was initiated when the applied load reached the peak load, P_i^c , with a pop-in behavior. Then, the crack proceeded perpendicular to the fiber with accompanied by shear sliding along the fiber-matrix interface. The fiber-matrix interfacial shear sliding parts along the fiber axis were clearly identified by visual observation of the surface of the specimen and seen as a triangular shaped array of white lines along the fiber axis. No fracture of the fiber occurred during the fracture experiment of the specimen.

The crack in the SiC fiber-reinforced Vycor matrix composite propagated perpendicular to the fiber axis with fracture of the fibers and without interface splitting. The first pop-in of the matrix in the Vycor matrix composite occurred at the first peak load, P_i^c , and the behavior was similar to that of the Pyrex matrix composite.

Fig. 3 Typical load (P)-displacement (u) curve for SiC fiber-reinforced glass matrix composite, (a) SiC-Pyrex matrix composite and (b) SiC-Vycor matrix composite (The solid line indicates composite and broken line indicates glass matrix).

After the pop-in, a steady state crack propagation perpendicular to the fiber axis was observed. The saw-toothed load-displacement curve after the maximum load indicates fracture of the fiber with the advance of the matrix crack.

2. Fracture toughness

The fracture toughness of the composites, K_{Ic}^c , for the DCB specimen was calculated from⁽¹¹⁾

$$K_{Ic}^c = \left(\frac{Pa}{\sqrt{It}} \right) \left[\left(1 + \frac{\delta}{a} \right)^2 + \left(\frac{E_c^L}{12G_c} \right) \left(\frac{h}{a} \right)^2 \right]^{1/2} \quad (1)$$

$$I = \frac{bh^3}{12}, \quad \delta = \left(\frac{4Iw}{t} \right)^{1/4}$$

where P is the applied load, a is the notch length, Δa is the crack extension length from the notch root, I is the moment of inertia, h is the beam height, t is the thickness of the grooved section, E_c^L is the longitudinal Young's modulus of the composite ($E_c^L = E_f f + E_m(1-f)$), G_c is the shear modulus of the composite ($G_c = G_m G_f / [(1-f)G_f + fG_m]$), f is the fiber volume fraction and δ is a constant based on the width of the guide notch. The fracture toughness was obtained using the pop-in load of the composite, P_i^c . The R-curve of the composite is obtained knowing the relationship between P and Δa .

The obtained results are listed in Table 2, where it can be noted that the fracture toughness of the Pyrex matrix composites was nearly the same as that of un-reinforced glass matrices, while the fracture toughness of the Vycor matrix composite was approximately 90% higher than that of the un-reinforced glass matrices. Figure 4 shows R-curve behavior of the composites. In both composites, the measured toughness increased with increase of the crack extension length. The toughness increase of the Vycor matrix composite during the rising R-curve behavior was smaller than that of the Pyrex matrix composite. This is probably due to the difference of the crack shield-

Table 2 Measured fracture toughness (K_{Ic}^c) of the composite and toughness of the glass matrix (K_{Ic}^m).

Material	Fracture toughness (MPa $\sqrt{m}$)	Average value of fracture toughness (MPa $\sqrt{m}$)
SiC-Pyrex glass matrix composite	0.65	0.82
	0.81	
	0.82	
	0.90	
	0.92	
SiC-Vycor glass matrix composite	1.62	1.72
	1.65	
	1.72	
	1.81	
	1.82	
Pyrex glass*	—	0.83
Vycor glass*	—	0.85

* The values are obtained average of ten specimen. The initial toughness value of the glass matrix is the same as fracture toughness.

Fig. 4 Typical R-curve of the composites.

ing mechanism by fiber bridging process due to the differences of interfacial shear stress transfer potential. Detailed analysis of the R-curve behavior is outside the framework of the present study, which is focused on the fracture toughness of the composites.

Figure 5 shows a typical example of the fracture surface of the Pyrex and Vycor matrix composites. In the Pyrex matrix composite, the specimen did not completely separate into two pieces, therefore, one side of the matrix was carefully removed mechanically. The photographs in the figure suggest that rising R-curve behavior is due to fiber-bridge toughening⁽¹⁾.

3. Existence of thermally-induced stress

The evidence for the possibility of stress transfer at the interface is clearly shown in transmission photoelasticity micrographs (Fig. 6). Both Fig. 6(a) and (b) reveal completely the same optical condition. The white areas of the photographs indicate the existence of thermally induced stress in the matrix. The greater enhancement of the parts in the Vycor matrix composite than in the Pyrex matrix composite indicates a higher level of thermally induced

Fig. 5 Fracture surface of SiC-Pyrex matrix composite (a) and SiC-Vycor matrix composite (b).

stress in the formers. Although, major stress of the photoelastic pattern originates from the difference between the radial and tangential stresses, the fact that it exists clearly indicates the possibility of stress transfer between fiber and matrix. The stress transfer mechanism at the interface is shown in the Appendix.

IV. Discussion

The discussion will be focused on the possibility of increased fracture toughness of a fiber-reinforced ceramics by a thermally induced longitudinal stress. The possible factors affecting the fracture toughness of fiber-reinforced composites are: (i) Young's modulus (modulus toughening) and (ii) longitudinal thermally induced stresses in the fiber and matrix.

The stress intensity at the matrix phase is scaled to the ratio of Young's modulus ratio between the matrix and composite. Thus, the stress intensity factor of the composite for the onset of the matrix cracking under a thermally induced stress free condition, $K_{i_0}^c$, is given by⁽¹²⁾⁽¹³⁾:

$$K_{i_0}^c \cong \left(\frac{E_c^L}{E_m} \right) K_c^m \quad (2)$$

SiC-Pyrex Matrix Composite

SiC-Vycor Matrix Composite

Fig. 6 Typical transmission photoelasticity pattern of a composite. (a) Pyrex matrix and (b) Vycor matrix.

where K_c^m is the critical stress intensity factor of the monolithic matrix. In this experiment, the calculated K_i^c from eq. (2) ranged from 1.1 to 1.2 because of low volume fraction of the fiber ($f=0.05$). Practically, eq. (2) can be applied to for the case of a relatively high fiber volume fraction ($f>0.3$)⁽¹³⁾. In this experiment, $K_{i_0}^c \approx K_c^m$ can thus be assumed for the thermally induced stress free condition. Consequently, the change in fracture toughness of the composites cannot be explained by the mechanism of modulus toughening.

If the stress transfer between fiber and matrix were maintained during the cooling from the processing temperature, thermally induced stress at the longitudinal direction should be built up in the fiber and matrix. This thermally induced stress would affect the fracture toughness. The fracture toughness depends on the magnitude and sign of the CTE mismatch between the fiber and matrix. In this experiment, plots of the increase of fracture toughness of the composites, $K_i^c - K_c^m$, versus $(\alpha_f^L - \alpha_m)\Delta TE_m$ for the Pyrex and Vycor matrix composite, respectively, are shown in Fig. 7. ΔT is defined as the temperature difference between the strain point temperature, which corresponds to the lower annealing point of

Fig. 7 Plots of increase of the fracture toughness, $K_i^c - K_c^m$, versus $(\alpha_f^L - \alpha_m)\Delta TE_m$.

glass and viscosity of glass: $10^{14.5}$ dPa·s⁽¹⁴⁾, and room temperature. In this experiment, the fracture toughness of the monolithic glass materials was found to be the same within the experimental errors and the Young's moduli of the glass were also the same (Table 1). Figure 7 shows that the fracture toughness of the composite depends strongly on the thermally induced stress in the matrix by mismatch of CTEs.

Consider that the crack has to propagate through alternate zones of uniform thermally induced stress, D_0 , as shown in Fig. 8, for its propagation through a uniform compressive zone. This compressive stress applies closure traction over a length at the crack tip equivalent to the zone length, and the increase in fracture toughness is determined by the compressive zone; the measured fracture toughness of the composite, $K_{i_0}^c$, is then given by⁽¹⁵⁾⁻⁽¹⁷⁾

$$K_i^c = K_{i_0}^c + 2\Delta\sigma_m^T \sqrt{\frac{2D_0}{\pi}} \quad (3)$$

where $\Delta\sigma_m^T$ is the longitudinal thermally induced compressive stress in the matrix[†].

In the axial direction, the average mismatch stress built up in the matrix, $\Delta\sigma_m^T$, in eq. (3) has been calculated approximately from the simple formula⁽³⁾:

$$\Delta\sigma_m^T = \frac{\lambda_2}{\lambda_1} \left[\frac{E_f}{E_c} \right] \frac{f(\alpha_f^L - \alpha_m)\Delta TE_m}{(1 - \nu_m)} \quad (4)$$

where E_c^L is the longitudinal Young's modulus of composite and λ_1 and λ_2 under the conditions of $\nu_f = \nu_m = \nu$ are given by:

$$\lambda_1 = \left\{ 1 - \left(1 - \frac{E_c^L}{E_f} \right) \frac{(1 - \nu)}{2} - \left(\frac{E_c^L}{E_f} \right) \nu^2 \right\} / (1 - \nu^2) \quad (5)$$

$$\lambda_2 = 1 - \left(1 - \frac{E_c^L}{E_f} \right) / 2 \quad (6)$$

where ν_f is Poisson's ratio of the fiber, ν_m is Poisson's

[†] The stress intensity factor at the crack tip oscillates with the stress field during crack growth. In this calculation, the crack growth resistance of the composite is assumed to be determined by the maximum compressive stress.

Fig. 8 Schematic drawing of effect of CTE mismatch on the thermally induced longitudinal stress in the matrix and fiber for continuous fiber-reinforced ceramics (a) and effect of thermally induced compressive stress on the toughness of the composite (b).

ratio of the matrix, ΔT is the temperature difference between the strain point temperature of the glass matrix (Table 1) and room temperature. The calculated compressive stress $\Delta\sigma_m^T$, for the Pyrex matrix composite and the Vycor matrix composite ($f=0.05$) is ≈ 19 MPa and ≈ 73 MPa, respectively[†].

Using above eqs. (3)–(6) and substituting $D_0 \approx 80 \mu\text{m}$, the calculated results of increase of the fracture toughness of the composites are $\approx 0.27 \text{ MPa}\sqrt{\text{m}}$ for the Pyrex matrix and $\approx 1.05 \text{ MPa}\sqrt{\text{m}}$ for the Vycor matrix composite. The comparison between the experimentally obtained value and theoretically calculated value is shown in Fig. 8 by solid line. Although the experimental result shows about $0.2\text{--}0.3 \text{ MPa}\sqrt{\text{m}}$ higher than that of the theoretical predictions, the experimental results are explained by the thermally induced stress in the matrix.

V. Concluding Remarks

Possibility of increasing the fracture toughness of a continuous fiber-reinforced ceramic matrix composite by a thermally induced compressive stress has been shown experimentally. The uses of (i) longitudinal thermally induced compressive stress in the matrix and (ii) increase of longitudinal Young's modulus was the key to improving the fracture toughness of the composite. The selection of raw materials with $E_f > E_m$ and $\alpha_f^T > \alpha_m$ will increase the fracture toughness when an adequate stress transfer mechanism operates between fiber and matrix.

Further research is needed to determine thermally induced stress with optimum interfacial condition for complete understanding of the effects of thermally induced stress on the total toughening process of fiber-reinforced

composite.

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Appendix

The introduction of thermally induced longitudinal stress requires stress transfer at the interface during cooling from the processing temperature. In fiber-reinforced ceramics, when α_f^T (T : transverse) is smaller than that of matrix, α_m ($\alpha_f^T < \alpha_m$), a compressive force perpendicular to the fiber axis is generated at the interface. On the other hand, when α_f^T is larger than that of matrix ($\alpha_f^T > \alpha_m$), the

Fig. A-1 Schematic drawing of stress transfer mechanism between fiber and matrix for SiC fiber-reinforced Vycor matrix composite. δ^* is the gap width between fiber and matrix due to CTE mismatch.

[†] The thermal expansion coefficient of the Pyrex and Vycor glass is $3.70 \times 10^{-6} \text{ K}^{-1}$ and $0.51 \times 10^{-6} \text{ K}^{-1}$, respectively.

interface between fiber and matrix tends to separate during cooling from the processing temperature. In this experiment, the thermal expansion coefficient of the fiber perpendicular to the axis was $2.53 \times 10^{-6} \text{ K}^{-1}$ ⁽⁷⁾ and the average thermal expansion coefficient of the Pyrex and Vycor glass, respectively, were $3.70 \times 10^{-6} \text{ K}^{-1}$ and $0.51 \times 10^{-6} \text{ K}^{-1}$ (Table 1).

According to the theoretical consideration, the stress transfer of Vycor matrix composite is impossible due to the separation of the interface during cooling from the processing temperature. In this system, the maximum separation distance between fiber and matrix at transverse direction, δ^* , as defined in Fig. A-1, is roughly

given by

$$\delta^* \approx R_f(\alpha_f^T - \alpha_m)\Delta T \quad (\text{A-1})$$

where R_f is the radius of the fiber. The estimated δ^* for Vycor matrix composite is $\approx 0.2 \mu\text{m}$ and the value is smaller than the peak roughness value of the surface of SCS coating (0.09 to $1.8 \mu\text{m}$ for SCS-6⁽¹⁸⁾; the same surface morphology was observed in SCS-2 fiber). Therefore the separation distance is small enough to achieve a frictional stress transfer between fiber and matrix, and this transfer seems possible after the onset of interface debonding.