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INTERFACES AND TOUGHENING IN CERAMICS

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RESUME

La modelisation par la mecanique de la rupture a recemment fourni un eclairage approfondi du role des interfaces dans l'augmentation de la resistance a la fracture des ceramiques et des composites ceramiques par le renforcement apporte par des ponts etablissant des liens dans le defaut. Quelques resultats de la modelisation sont resumes et une methode d'indentation est decrite pour mesurer les proprietes mecaniques des interfaces (energie de decollement, resistance de frottement au glissement et contraintes residuelles) des fibres individuelles des composites en presence de liaisons faibles aux interfaces entre les fibres et la matrice.

ABSTRACT

Fracture mechanics modelling has recently provided insight into the role of interfaces in toughening of ceramics and ceramic composites by reinforcements that form crack bridging ligaments. Some results of the modelling are summarized and an indentation method is described for measuring interfacial mechanical properties (debond energy, frictional sliding resistance, and residual stresses) at individual fibers in composites with weak interfacial bonding between the fibers and matrix.

1. INTRODUCTION

Brittle materials can be substantially toughened by reinforcements that form ligaments between crack surfaces. This toughening mechanism has been demonstrated in large-grained single phase ceramics such as Al_2O_3 ^{1,2} as well as in fiber,³⁻⁵ whisker⁶ and metal⁷ reinforced ceramic composites. In all of these examples the properties of the interface between the reinforcement and the matrix play a critical role in determining the extent of toughening.

The purpose of this paper is to examine some of the properties of the interface that need to be characterized in order to design optimum mechanical properties of the composite. First some recent developments in fracture mechanics analysis will be summarized. These demonstrate a means of obtaining the relation between macroscopic properties (e.g., strength, fracture toughness) and the micromechanical behavior of the crack-bridging ligaments, as influenced by interfacial properties and residual stresses. Some results are presented for a specific composite that has bridging ligaments dominated by frictional sliding. Then an indentation method which allows some of the important properties such as interfacial debonding, frictional forces and residual stresses to be measured directly at individual fibers is described.

2. TOUGHENING BY BRIDGING

2.1 Fracture Mechanics Analysis

Two approaches have been developed recently to analyze the influence of bridging ligaments on the toughening of ceramics. Both model the bridging ligaments as closure tractions acting on the crack surfaces within the bridging zone (Fig. 1). In one approach^{8,9} the modified crack tip stresses are calculated directly using a standard Green's function, analogous to the Dugdale/Barenblatt models of fracture.^{10,11} However, these models assume uniform closure tractions within the bridging zone and a failure criterion defined by allowing the stress singularities due to the applied loading and the bridging forces to cancel. More generally, the closure tractions must exhibit a dependence upon the crack opening displacements, characteristic of the particular reinforcing mechanism. Moreover, the usual stress singularity exists in the matrix near the tip of the crack, with stress intensity factor equal to the toughness of the unreinforced matrix. Evaluation of the influence of the bridging forces in this case requires solution for the crack opening displacement as a function of position within the bridging zone. With the exception of a few special cases, this requires numerical solution of an integral equation.

An alternative, equivalent approach involves use of the J-integral.¹²⁻¹⁴ Based on this formulation, the increase in fracture energy is given by

$$\Delta J = 2 \int_0^{u^*} \sigma(u) du \quad (1)$$

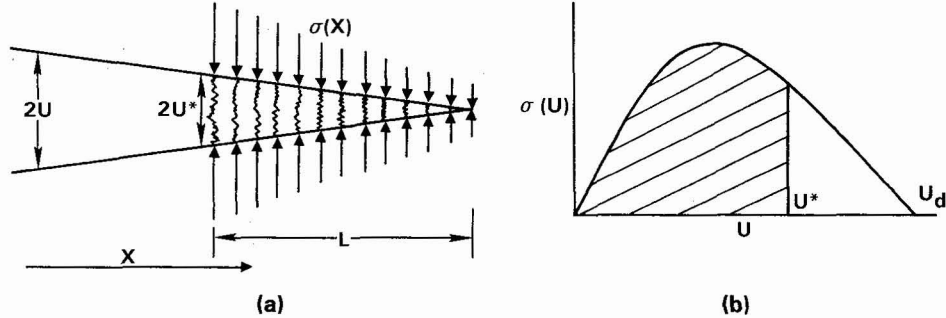


Fig. 1 (a) Closure tractions exerted by crack bridging ligaments, (b) stress-displacement relation for stretching of bridging ligaments.

where $\sigma(u)$ is the stress-displacement relation for stretching the bridging ligaments, u is the crack opening displacement and u^* the crack opening at the end of the bridging zone. For general initial crack configurations it is necessary to solve for u as a function of position in the crack, as in the stress intensity approach, in order to evaluate u^* . However, for the special case of steady-state toughening, defined by $u^* = u_d$, where u_d is the crack opening above which the ligaments cease to restrain the crack surfaces, the toughening is given simply by the area beneath the $\sigma(u)$ curve and solution for the crack opening displacements is not necessary.

In both of these analyses the properties of the reinforcement and the interface between the reinforcement and the matrix influence the macroscopic fracture properties through the stress-displacement function for ligament stretching. Some stress-displacement functions for brittle and ductile reinforcements are illustrated in Fig. 2. For brittle reinforcements with interfaces bonded sufficiently strongly to prevent debonding when a crack passes, the area beneath the $\sigma(u)$ curve is relatively small. Weak interfaces that allow some debonding result in more compliant ligaments with the possibility of ligament failure within the region of ligament embedded in the matrix, frictional pullout, and large steady-state toughening. Reinforcements held in place by mechanical forces without chemical bonding allow the largest toughening. On the other hand, ductile reinforcements appear to exhibit maximum toughening when the interface between the reinforcement and matrix is strongly bonded.⁷ If debonding occurs the bridging stress is limited to the uniaxial flow stress of the ligament material, whereas in a fully-bonded ductile particle, elastic constraint due to the matrix can lead to bridging stresses exceeding the uniaxial flow stress by nearly an order of magnitude.⁷

Residual microstructural stresses, which are often unavoidable in composites, influence the macroscopic toughening by modifying the $\sigma(u)$ function for the bridging ligaments.¹³ For several important bridging mechanisms this involves simply translation of the $\sigma(u)$ function along the stress axis. However, the resultant effect on the steady-state toughening is very sensitive (in both sign and magnitude) to both the functional form of the $\sigma(u)$ relation and the rupture criterion for the ligaments. Calculated trends for several types of bridging mechanisms are summarized in Table 1.¹³

2.2 Unbonded Reinforcements

The use of fracture mechanics to relate macroscopic composite properties to reinforcement interface properties can be illustrated by examining the behavior of composites containing unbonded reinforcements that are held in place by mechanical forces. This system has been analyzed in detail using both of the above approaches.^{8,9} Examples of such composites include glass and glass-ceramic matrices reinforced by graphite and SiC fibers.^{3,4} The macroscopic fracture behavior of these composites can vary dramatically, depending on the magnitude of the sliding resistance at the interface. For sufficiently high frictional stress failure occurs by growth of a single crack. Then a steady-state fracture toughness, K_C , can be defined:⁹

$$K_C/K_0 = (E/E_m)^{1/2} (1 + \alpha) \quad (2)$$

with

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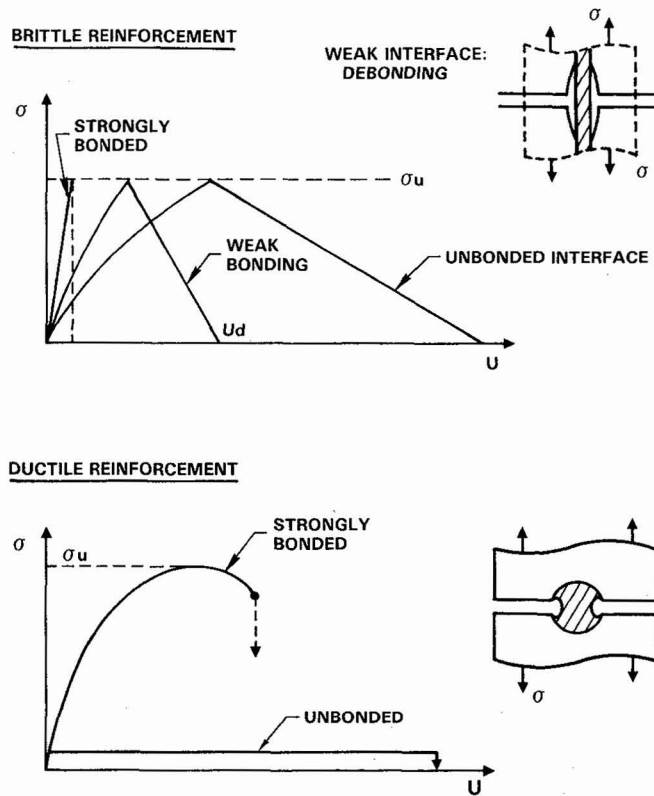


Fig. 2 Schematic stress-displacement relations for stretching of brittle and ductile reinforcements.

$$\alpha = \frac{S^3 R f (1 - f) E_m^3}{3(1 - \nu^2) \tau E_f E^2 K_0^2} \quad (3)$$

where τ is the frictional stress that resists sliding at the interface, R is the fiber radius, S is the strength of the fiber, K_0 is the toughness of the unreinforced matrix, f is the volume fraction of fibers, E_f , E_m , and E are the Young's moduli of the fibers, matrix and composite, and ν is Poisson's ratio of the composite. Therefore, it is evident that decreasing the frictional stress increases the steady-state toughness. However, if τ decreases below a critical value defined by $\alpha \sim 1$, a change in fracture mechanism can occur. A crack that is initially bridged by fibers can extend entirely through the matrix without any of the bridging fibers fracturing. The composite does not fail catastrophically, but instead can support further load increase (nonlinear) before failure. The stress for matrix cracking is then an intrinsic property of the composite (i.e., independent of flaw size) given by^{5,8,9,15}

$$\sigma_0 = \left[\frac{6(1 - \nu^2) \tau f^2 E_f E K_0^2}{R E_m^3 (1 - f)} \right]^{1/3} \quad (4)$$

Table 1
Effect of Residual Stress on Fracture Energy

Stress-Displacement Law	Rupture Condition	Residual Stress in Matrix	
		Tension	Compression
Linear	{Stress {Displacement	Decrease Decrease	Decrease <u>Increase</u>
Frictional (Fracture at Crack Plane)	{Surface Roughness {Displacement	<u>Increase</u> Decrease	Decrease <u>Increase</u>
	{Coulomb Friction {Displacement	Decrease Decrease	
Frictional (Pullout of Entire Reinforcement)	{Surface Roughness	Negligible	Negligible
	{Coulomb Friction	<u>Increase</u>	
Ductile	Bonded Displacement	Decrease	<u>Increase</u>

In this region of behavior, decreasing τ causes σ_0 to decrease. Therefore, optimum values of σ_0 or ΔK are achieved at an intermediate value of τ corresponding to the transition in failure mechanism ($\alpha \sim 1$).

3. MEASUREMENT OF INTERFACIAL MECHANICAL PROPERTIES

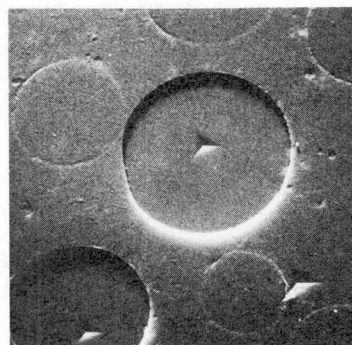
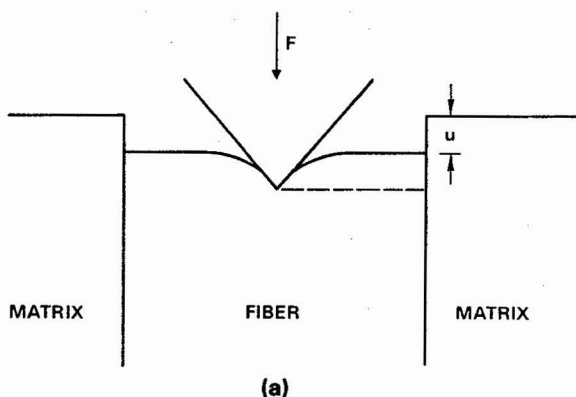
An indentation technique has been developed recently to allow measurement of frictional sliding resistance and debond fracture energy at individual fibers in weakly bonded composites.^{16,17} Analysis is also presented below to enable the method to be used to measure the magnitude of residual stress in the fibers.

The method entails loading a sharp indenter (e.g., Vickers hardness pyramid) onto the end of a fiber in a polished cross section of the composite and measuring the applied force and displacement continuously (Fig. 3). If this loading causes debonding at the fiber-matrix interface with frictional sliding over the debonded area, the displacement of the fiber relative to the matrix surface is

$$u = F^2/4\pi^2R^3\tau E_f - 2r/\tau \quad (5)$$

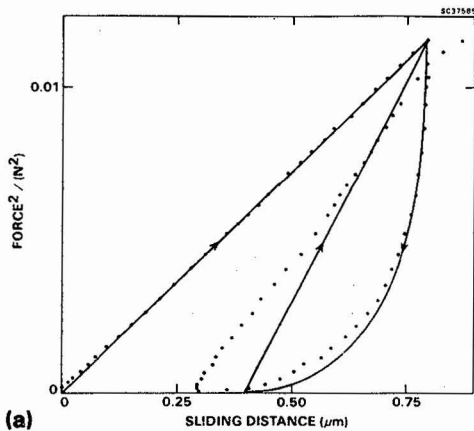
where F is the force applied to the fiber and r is the debond fracture energy (Mode II). The displacement measured in these experiments is the sum of the sliding distance given by Eq. (5) and the penetration of the indenter (elastic and plastic) into the fiber (Fig. 3a). The penetration must be either calculated or (preferably) calibrated in separate experiments where fiber sliding does not occur. The calibration has been obtained in a SiC/glass-ceramic composite by heat treating the composite in air at 1000°C to form a strongly bonded oxide layer at the fiber-matrix interface which does not debond during indentation.¹⁷ Measurements in the as-fabricated composite indicated that sliding occurred between the fiber and matrix (Fig. 3b). The measured sliding distances for this composite are compared with the predictions of Eq. (5) in Fig. 4a. The data follow a linear relation when plotted as F^2 versus u as predicted, with intercept $2r/\tau = 0 \pm 0.01 \mu\text{m}$. Measured values of peak force and displacements, $F_m = 0.11 \text{ N}$ and $u_m = 0.80 \mu\text{m}$ with $R = 8.0 \mu\text{m}$ and $E_f = 200 \text{ GPa}$, gave $\tau = 3.5 \text{ MPa}$ and an upper bound for the debond fracture energy (defined by the experimental errors) of $0.04 \cdot \text{J m}^{-2}$. Therefore, the debond fracture energy in this composite is very small (smaller than that expected for Van der Waals force) and the mechanical response of the crack bridging ligaments is dominated by mechanical sliding resistance.

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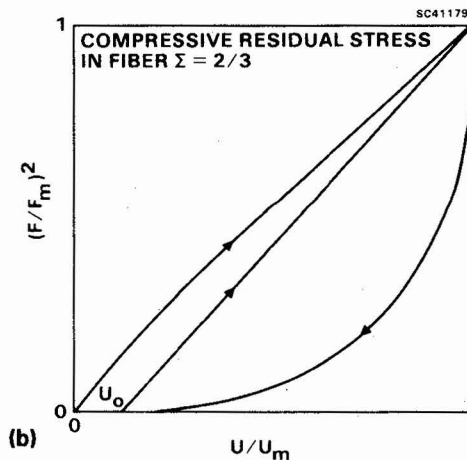


(b)

Fig. 3 (a) Indentation method for measuring frictional sliding resistance, and (b) scanning electron micrograph showing SiC fibers in glass-ceramic matrix after indentation with a diamond pyramid (triangular based). From Ref. 17.

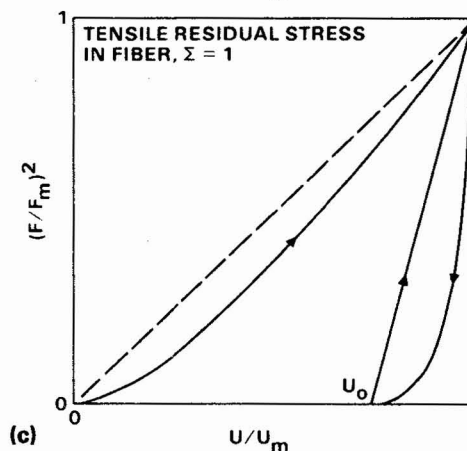


(a)



(b)

Fig. 4 (a) Force-displacement measurements from indentation of fiber in Fig. 3b. Curves represent theoretical predictions (from Ref. 17.) (b)-(c) Predicted force-displacement relations for compressive (b) and tensile (c) residual stresses in fiber.



(c)

Force-displacement measurements for unloading and reloading are also shown in Fig. 4a. Analysis of fiber sliding, assuming constant frictional stress, τ , and no bonding, predicts the displacements¹⁶

$$u/u_m = 1 - (1 - F/F_m)^2/2 \quad (6)$$

and

$$u/u_m = 1 + (F/F_m)^2/2 \quad (7)$$

for unloading and reloading. The measured fiber displacement after unloading is smaller than predicted, suggesting that the sliding resistance during reverse slip was lower than during the initial slip by about 20%.¹⁷

The sliding distances in these experiments are modified if residual stresses exist in the composite. Compressive residual stress in the fiber causes the plot of F^2 versus u to be changed as shown in Fig. 4b: the initial loading curve is nonlinear, with decreasing slope with increasing F , the residual displacement, u_0 , after unloading is smaller than the value $u_0 = u_m/2$ obtained in the absence of residual stress, and the reloading curve is linear. The residual displacement u_0 for an unloaded system with constant τ during forward and reverse sliding is

$$u_0/u_m = 1 - 1/2 [1 - \varepsilon(1 - \varepsilon/2)] \quad (8)$$

where

$$\varepsilon = 2\pi R^2 \sigma_R / F_m \quad (9)$$

The magnitude of the residual stress may be calculated from measurement of u_0 . Caution is required, however, for values of u_0 less than 0.5 can also be caused by the need to debond the interface before sliding can occur, or by a decrease in τ during reverse sliding as observed in Fig. 4a. The three potential causes for residual displacements to be less than $u_m/2$ can be distinguished by their different influences on the initial loading curve.

For tensile residual stress in the fiber, the force-displacement curve is modified as in Fig. 4c: the initial loading curve has curvature opposite to that caused by compressive stress, the residual displacement after unloading is larger than $u_m/2$, and the reloading curve is again linear. The residual displacement, u_0 , is given by

$$u_0/u_m = 1 - 1/2(1 + \varepsilon) \quad (10)$$

and the magnitude of the residual stress may be calculated from measurement of u_0 .

4. CONCLUSIONS

The amount of toughening achieved in ceramics with reinforcements that form crack-bridging ligaments is very strongly dependent on the nature of the interface between the reinforcement and matrix and its influence on the stress-displacement relation for ligament stretching. Important properties are debonding resistance, frictional sliding resistance and residual stresses. For brittle reinforcements enhanced toughening is obtained with weak, or no interfacial bonding, low frictional stresses, and tensile or compressive residual stresses in the matrix depending on the specific stress-displacement law for the bridging ligaments. For ductile ligaments large toughening is achieved with strong interfacial bonding and compressive residual stress in the matrix. Because of the sensitivity of both the magnitude and sign of toughening to the specific stress-displacement law, it is critical to develop methods for measuring interfacial mechanical properties. For weakly bonded brittle reinforcements, the indentation method provides quantitative measurements of debond energy, frictional sliding stress and residual stress at individual fibers.

5. ACKNOWLEDGEMENT

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N.W. Ashcroft Do you have to include Poisson ratio effects; in compression analysis of the fibers; that is, do you presume that the length over which longitudinal strain is accommodated is sufficiently long that transverse strain effects are negligible? This length should be related to the frictional component.

D. Marshall Poisson ratio effects are neglected in the analysis presented here. We have shown experimentally in the SiC/glass-ceramic composites that this is valid ($\tau=2\text{MPa}$; contribution from transverse expansion $<0.1\text{MPa}$) Ref. 17.

C. Hartley What was the phenomenological criterion used for debonding at the interface?

D. Marshall A critical fracture energy. Note that the loading is mode II, so the criterion is G_{IIC} (Ref. 17).

P.S. Ho Ceramics are strong and brittle. What can you say in general about the fracture behavior in a matrix which is considerably weaker and can sustain a large amount of deformation? Such an example is a polymer which can deform in a viscoelastic manner.

D. Marshall If deformation occurs in the matrix a completely new (and more complex) analysis of fiber displacements would be needed. Such a study would be worthwhile. A viscoelastic matrix would also require analysis of rate effects.

M.H. Yoo It is known that dislocation mobility in a soft matrix in a region of localized plasticity depends on the rate of deformation. Have you done the frictional stress measurement experiment with the rate of deformation as a variable?

D. Marshall The matrix deformations are purely elastic in these brittle materials. However we have investigated the effect of loading rate on the friction measurements: over the range of displacement rates 10^{-2} to $10^{+2} \mu\text{msec}^{-1}$ there was no change in the measured frictional stress (Ref. 17).