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Interfacial fracture toughness measurement using indentation

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Recent models have been developed for relating fracture toughness to indentation strengths for brittle monolithic materials. Thus, indentation may provide a simple and non-destructive means to measure fracture toughness. The indentation technique is further explored to evaluate the interfacial toughness in this work. When microindentations at loads ranging from 50 g to 4 kg were placed close to the interface of an aluminium/aluminum composite laminate, the interface stayed intact. In contrast, the interface of a niobium silicide/niobium laminate indented under similar conditions was found to debond, indicating a weaker interface. However, a Rockwell indenter at loads ranging from 60 to 150 kg were sufficient to debond the interfaces of two of the aluminum composite/aluminum laminates. Finally, the interfacial toughness is correlated to the indenter load and the delamination length along the interface. A power-law relationship was found between the load and the delamination length.

1. Introduction

Fracture toughness is an important parameter for the design of brittle materials. Traditional measurements such as compact tension or bending tests require complicated procedures and a minimum specimen size. The microindentation technique has been shown to be a simple and economic technique in its use of materials for providing a good estimate of fracture toughness of brittle materials. In addition, microindentation is almost non-destructive and causes little damage to the bulk material. Recent models [1–3] have been developed for relating fracture toughness via indentation for brittle materials. The approach involves direct measurement of Vickers-produced radial cracks under a given indentation load.

Fig. 1 shows a schematic of the Vickers indentation, after Anstis *et al.* [1]: P is the peak load and a and c are characteristic dimensions of the "plastic" impression and the radial/median crack, respectively. From dimensional analysis [1–3] the fracture toughness can be calculated as:

$$K_c = \beta P/c^{3/2} \tag{1}$$

where β corresponds to a complex geometrical factor for penny-like configurations, incorporating interaction effects of free surface, multiple-plane crack configuration and the relative fracture toughness of the material against its Young's modulus. It is usually determined by experimental calibration. From Evans and Charles [2], $\beta \approx 0.08$ gives a good fit to polycrystals where the c/a ratio is larger than 2.5. When applied to polycrystals with properties of hardness, toughness and Poisson's ratio that range between 1 to 70 $\mathrm{GN\,m^{-2}}$, 0.9 to 16 $\mathrm{MN\,m^{-3/2}}$, and 0.2 to 0.3, respectively, this characterization, according to Evans and Charles [2], should enable fracture toughness data to be obtained by indentation to within an accuracy of either $\approx 10\%$ if the Young's modulus is known or $\approx 30\%$ if the Young's modulus is unknown.

The interfacial properties have been shown to greatly affect the mechanical behaviour of laminated structures [4, 5]. In order to optimize the mechanical behaviour of materials systems with built-in interfaces, a basic understanding of the interfacial strength is of great importance. For example, the issue of adhesion strength in a metal film with a ceramic substrate has been investigated by researchers from areas as diverse as electronic packaging to composites engineering. Microscratch and microindentation have been used to characterize the adhesion of thin films [6].

In this paper, we explore the microindentation technique for measurement of the interfacial fracture toughness. Macroindentations using a Rockwell indenter are also performed to evaluate qualitatively the interfacial strength of an aluminium/aluminium composite laminate.

2. Materials and methods

Vickers microindentation experiments have been performed on two laminate systems, Nb_5Si_3/Nb and Al/Al + 15% SiC. In the first laminate system, the niobium silicide was made via a powder metallurgy process. Mechanical alloying was used for the production of Nb_5Si_3 powders. Laminated Nb_5Si_3/Nb was fabricated using vacuum hot-pressing. Additional processing information can be found elsewhere [7].

In the second laminate system, monolithic 7093 aluminum alloy and 7093 with 15% SiC reinforcement

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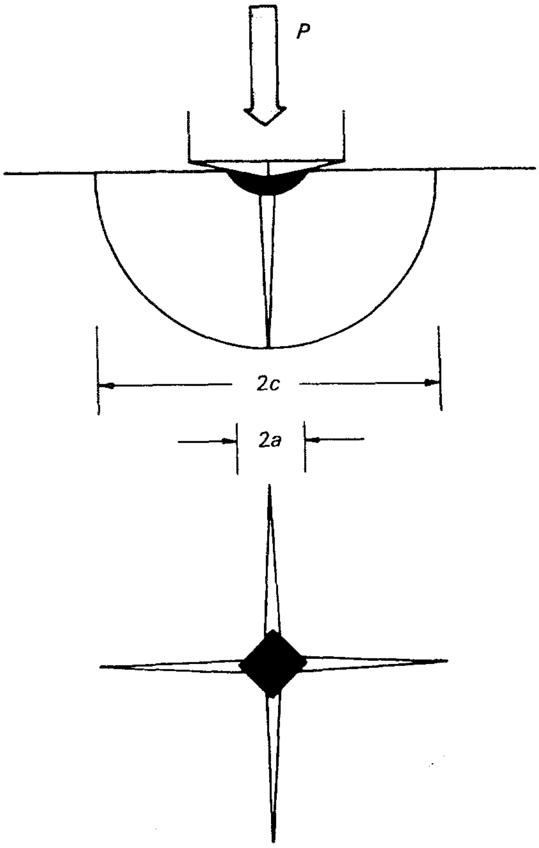

Figure 1 Schematic of Vickers-produced indentation-fracture system, after Anstis *et al.* [1], showing peak load, P, the characteristic dimensions of c and a of radial crack c and the hardness impression, respectively.

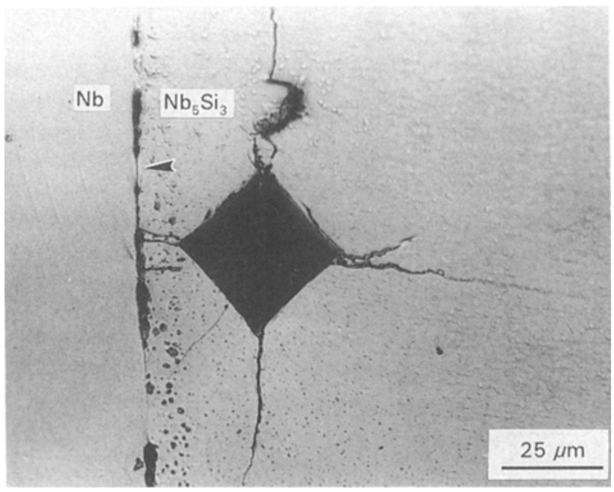

Figure 2 Optical micrograph of Nb_5Si_3/Nb laminate showing interfacial debonding, "plastic" impression and radial cracks produced by Vickers microindentation placed in niobium silicide. The indenter load is 2 kg.

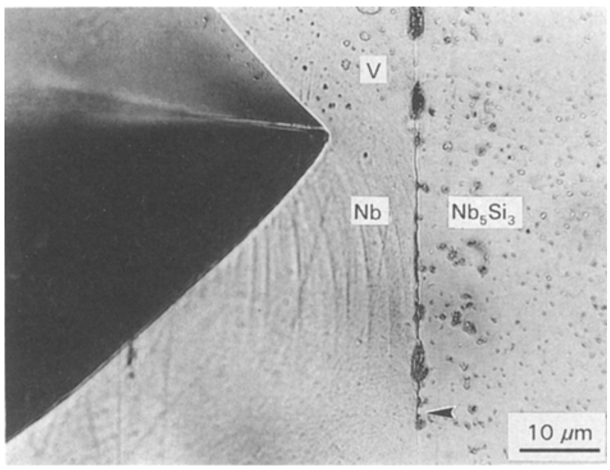

Figure 3 Optical micrograph of Nb_5Si_3/Nb laminate showing interfacial debonding and "plastic" impression produced by Vickers microindentation placed in niobium. The indenter load is 2 kg. Note that Vickers-produced radial cracks are absent since the niobium is fairly ductile.

were produced in billet form through a powder metallurgy route. The billets were extruded and hot-rolled to form the starting plates for the metal/composite laminates. Three types of laminates were made in collaboration with Alcoa. Laminate A was roll-bonded. Laminate B was roll-bonded with a 25 μm commercial purity aluminum interlayer between the aluminum layer and the composite layer. Laminate C was epoxy bonded. Laminates A–C were all heat-treated to the specification of Alcoa as detailed elsewhere [8].

Specimens of laminates A–C and Nb_5Si_3/Nb were polished using fine grade SiC papers. Microhardness indentations at room temperature were performed on both laminate systems using a Zwick 3212 microhardness tester. A Vickers indenter, with loads ranging from 50 g to 4 kg, was used. In addition to the above tests, a Rockwell C ball indenter (1/16" diameter), with a load range of 60–150 kg, was used to probe the interfacial behaviour of the aluminium/aluminium composites system because the interface of this laminate system was too strong to enable debonding under the load range of a microindenter. Encouraged by the fact that delamination length was strongly dependent on indenter load, a macro-type of indenter to probe the relative strengths of laminates A–C was selected.

3. Results

Micro-Vickers indentations were performed on both the niobium silicide/niobium and the aluminium/aluminum composite laminates. While the interface of the second laminate system stayed intact when indented close to the interface from either side with loads in the range of 50 g to 4 kg, the interface of the first laminate system indented under similar conditions was found to debond, indicating a much weaker interface (shown in Figs 2 and 3). The schematic shown in Fig. 4 is for indentation of the niobium silicide in the Nb_5Si_3/Nb laminate.

Most indentations of the Nb_5Si_3/Nb laminate were placed on the side of niobium silicide. On the silicide side, the indentation edge-cracks either arrested at the interface or did not reach the interface when the indenter was placed far from the interface, $d > 2a$. If the indenter was placed directly on the interface, $d \approx 0$, little or no interfacial debonding was observed (Fig. 5). The maximum amount of debonding was observed when the indenter was in the range of $0 < d < 2a$. The interfacial debond lengths appeared

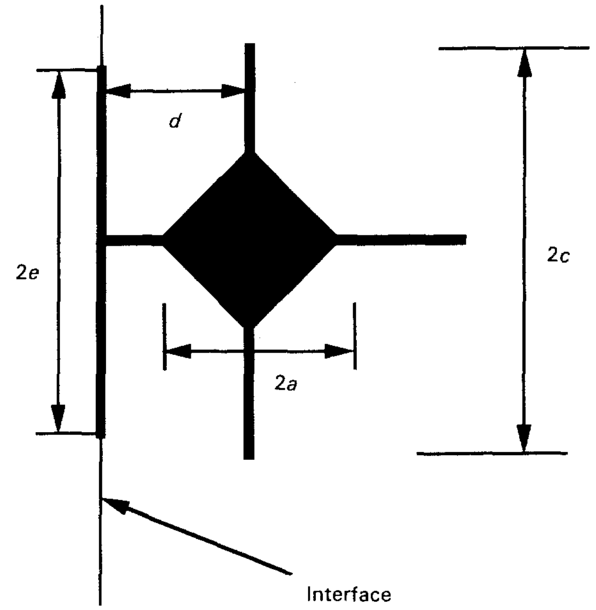

Figure 4 A schematic shows the characteristic dimensions of impression, a, distance to the interface, d, radial crack, c, and the interfacial debonding, e, when the indentation is placed in the brittle constituent. Thick lines indicate cracks.

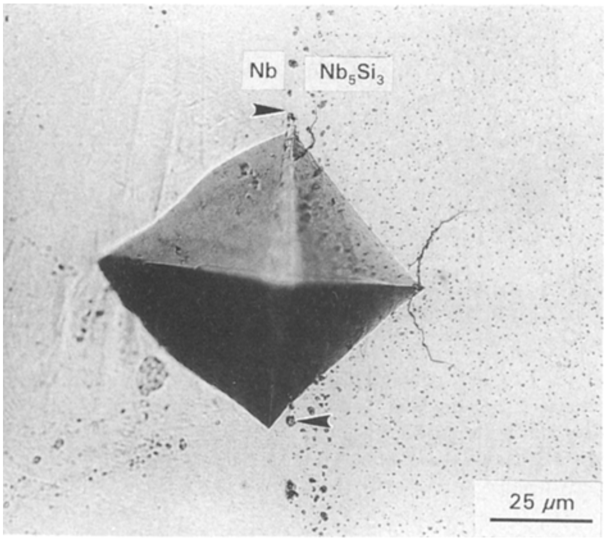

Figure 5 Optical micrographs showing no interfacial debonding when the indentation was placed directly on the interface of Nb_5Si_3/Nb laminate. The arrows indicate the interface. The indenter load is 2 kg.

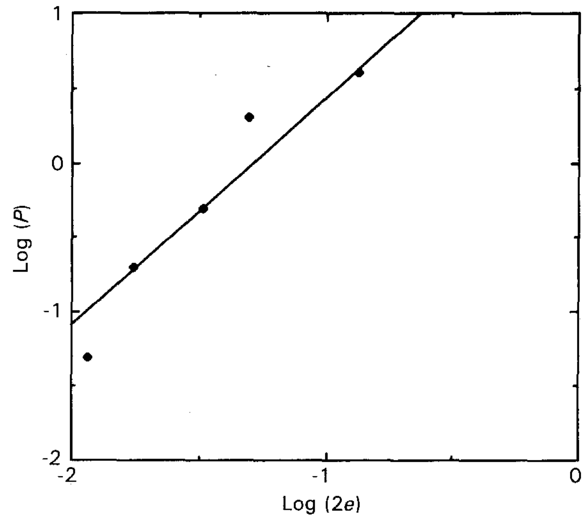

Figure 6 The indenter load in kg plotted against the interfacial crack length in mm for the Nb_5Si_3/Nb laminate. The solid line has a slope of 3/2.

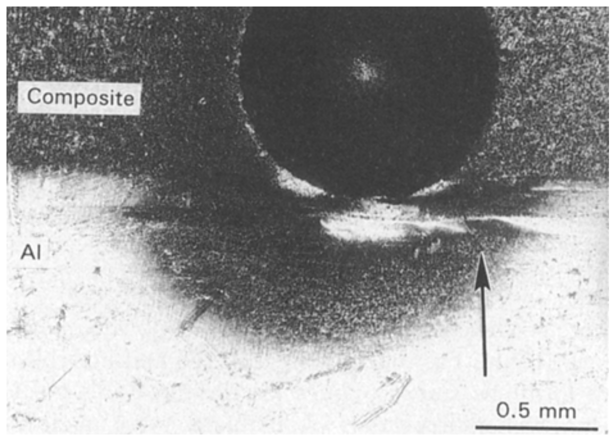

Figure 7 Rockwell indentation (150 kg load) on Al/Al + 15%SiC laminate A, with large plastic deformation in the interfacial region indicted by the arrow. No debonding was observed in the interface.

to be independent of the distance, d, within this range. On the other hand, the interfacial delamination length was a strong function of the indenter load, P (Fig. 6). For example, the average interfacial debond length increased from ≈ 12 to $\approx 130\ \mu m$ when the load increased from 0.05 to 4 kg. When plotted in a log–log plot, a power-law relationship was found to fit the data quite well. A few indentations at loads of 0.5 and 2 kg were placed close to the interface in the niobium. In these cases, the interface debonded to about the same length as that arising from indentations in the niobium silicide. Again, this supports the contention that the degree of delamination is a strong function of the indentation load and is relatively independent of the extra location in indentation provided that $0 < d < 2a$. The microindentation results are summarized in Table I.

Since the microindentation results indicated that interfacial delamination depended strongly on the indenter load, Rockwell indentations were selected to

TABLE I Vickers indentation of the niobium silicide in the NB_5Si_3/Nb laminate

Indenter load (kg)	0.05	0.2	0.5	2.0	4.0
Interfacial crack length, 2e (μm)	11.5 ± 1.5	17.5 ± 6.0	31.7 ± 6.3	49.0 ± 27.8	134 ± 31.0
Distance to interface ($d/2a$)	0.55	0.35	0.5	0.75	0.45
Number of indentations	6	4	3	5	9

study the interface of the Al/Al + 15% SiC laminate. The loads used were 60, 100 and 160 kg. While no debonding was found for laminate A (Fig. 7), debonding was indeed observed for laminates B and C (Figs 8 and 9). Also, the largest debond length was found for the highest load with laminate B. This indicates qualitatively that the order of the interfacial toughness of laminates A–C is C > B > A, in agreement with results obtained in a separate study by Zhang and Lewandowski [9] using more conventional measurements of interfacial toughness via four-point bending. The Rockwell indentation results are summarized in Table II.

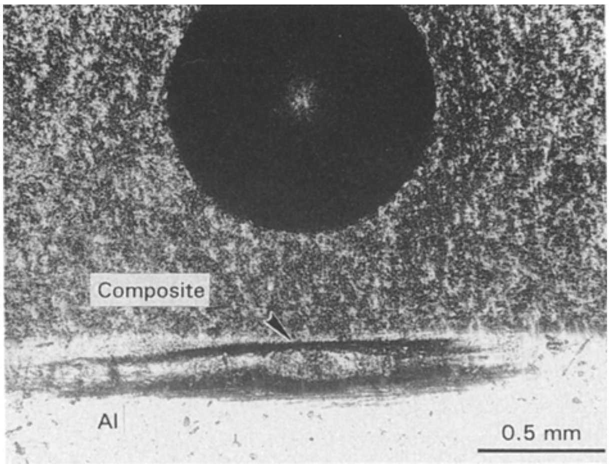

Figure 8 Rockwell indentation (150 kg load) on Al/Al + 15%SiC laminate B, with interfacial debonding indicated by the arrow.

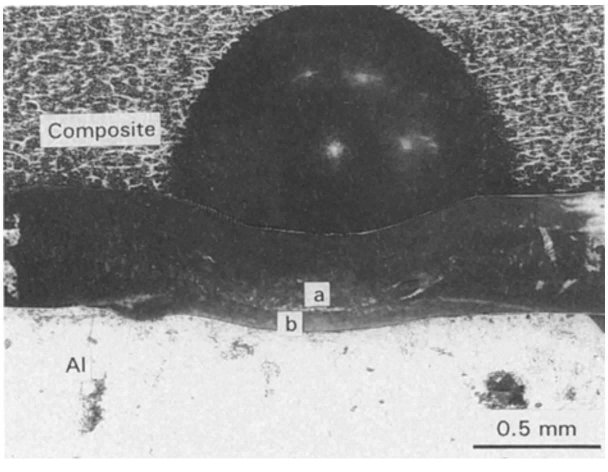

Figure 9 Rockwell indentation (150 Kg load) on Al/Al + 15%SiC laminate C. Zone b indicates debonding along the interface. Zone a shows the epoxy bond.

TABLE II Rockwell indentation on Al/Al + 15% SiC laminates

Type	Indenter load (kg)	60	100	150
A	Interfacial crack length (mm)	No crack	No crack	No crack
B	Interfacial crack length (mm)	–	1.4	1.7
C	Interfacial crack length (mm)	1.0	1.2	1.6

4. Discussion and conclusions

The power-law relationship (Fig. 6) exhibited between the measured interfacial crack length and the micro-indenter load can be rationalized as follows. The fracture toughness, or the critical stress intensity factor, by definition is proportional to the remote stress, σ, times the squared root of the crack length, d:

$$K_c = A\sigma d^{0.5} \tag{2}$$

where A is a constant depending on the loading conditions, specimen geometry, etc. In indentation the same type of argument is valid. For a brittle monolithic matrix, the value of stress can be estimated as:

$$\sigma \alpha \frac{P}{c^2} \tag{3}$$

where P is the indenter load and c is the indentation edge-crack length (Fig. 1).

Then, the fracture toughness can be calculated as:

$$K_c \,\alpha \frac{P}{c^2} c^{0.5} \alpha \frac{P}{c^{1.5}} \tag{4}$$

This reduces to the same form as Equation 1 derived by Evans and Charles [2].

A similar argument can, of course, be employed to calculate the fracture toughness of the interface. When the indentation is close to the interface, the average stress level can be estimated as (Fig. 7):

$$\sigma \alpha \frac{P}{e^2 + d^2} \approx \frac{P}{e^2} \tag{5}$$

This leads to a relationship between the indentation load and the crack length:

$$K_i \,\alpha \frac{P}{e^2 + d^2} e^{0.5} \approx \frac{P}{e^{1.5}} \tag{6}$$

or

$$K_i = \gamma \frac{P}{e^{1.5}} \tag{7}$$

where γ is a fitting constant.

Since the interfacial fracture toughness should be a constant, the interfacial crack length, according to the above equation, should also be a constant when d is small. This probably explains the observation of the degree of delamination being about the same irrespective of distance d, and its independence of whether the indentation is placed in the brittle or ductile constituent of the Nb_5Si_3/Nb laminate.

Using 1 MPa $m^{0.5}$ as an estimate for the interfacial strength of Nb_5Si_3/Nb laminate [10], the proportionality constant of 0.014 was obtained from Equation 7. If we recall the fitting constant of 0.08 for brittle ceramics in Equation 1 it is quite reasonable. A smaller constant suggests that crack deflection along the interface is more difficult than crack extension across the interface if the interface has the same strength as the matrix.

In conclusion, indentation is a promising technique to evaluate interfacial toughness. Additional experiments are in progress to finely calibrate the relationship between the interfacial toughness and the interfacial debond length.

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