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Tensile mechanical properties of SiC whiskers

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An initial evaluation has been made of the tensile mechanical properties of SiC whiskers synthesized by a vapour–liquid–solid process. A micro-tensile tester and associated testing techniques were developed for this purpose. The SiC whiskers exhibit an average tensile strength of 8.40 GPa (1 220 000 psi) and an average elastic modulus of 581 GPa (84 300 000 psi), and were considerably stronger and stiffer than continuous, polycrystalline SiC fibres. These results indicate the significant potential of SiC whiskers as short-fibre reinforcement elements for ceramic matrix composites.

1. Introduction

Recent studies have indicated that glass and glass-ceramic matrix–silicon carbide (SiC) fibre composites possess excellent potential for achieving significant increases in both the fracture toughness and forgiveness towards catastropic fracture of brittle load-bearing systems [1–4]. For most of these studies, a continuous SiC fibre (Nicalon) produced by the Nippon Carbon Company has been employed as the reinforcement element due to its ability to withstand oxidizing environments below 1200° C. This material is composed of very fine, polycrystalline SiC grains in a matrix of residual carbon. Due to modifications in this microstructure, the fibre has been shown [5] to exhibit a significant degradation of mechanical properties as a result of high temperature exposures above 1200° C. This response has retarded the development of high-temperature composite materials for potential use above 1200° C and there is presently great interest in advanced reinforcement materials with improved strength, oxidation resistance, and chemical stability at these higher temperatures.

SiC single crystal whiskers may constitute an improved reinforcement material for certain high-temperature ceramic composites. These whiskers are produced by a vapour–liquid–solid process [6, 7] and are essentially small (5 to 6 μm diameter) single crystals of β-SiC which can presently be produced in lengths as long as 100 mm. In the present investigation, techniques were developed initially to evaluate the room temperature mechanical properties of the SiC whiskers. Measurements have been made of strength and elastic modulus and these compared to the properties of Nicalon SiC fibres.

2. Experimental details

2.1. SiC whiskers

The SiC whiskers tested were produced at Los Alamos by a vapour–liquid–solid (VLS) process [6, 7]. These were received for testing in the form of mattes of intertwined individual whiskers. The whiskers were green in colour. Individual whiskers could be easily extracted from the matte by viewing under a low power stereo microscope and handling the whisker with fine-tipped tweezers. The whiskers were single crystals of β-SiC. This was confirmed by X-ray diffraction from a single, isolated whisker. The whiskers possessed a rounded triangular cross-sectional shape, with equivalent circular diameters of most whiskers falling in the range of 4 to 6 μm. As-received whisker lengths for the present study were typically approximately 10 mm, with some as long as 25 mm in length. The whiskers were tested in the as-synthesized condition, and no attempt was made to remove the metal catalyst balls at the whisker growth interface which are associated with the VLS process.

2.2. Micro-tensile tester

The SiC whiskers constitute an awkward geometry for tensile mechanical testing due to their very

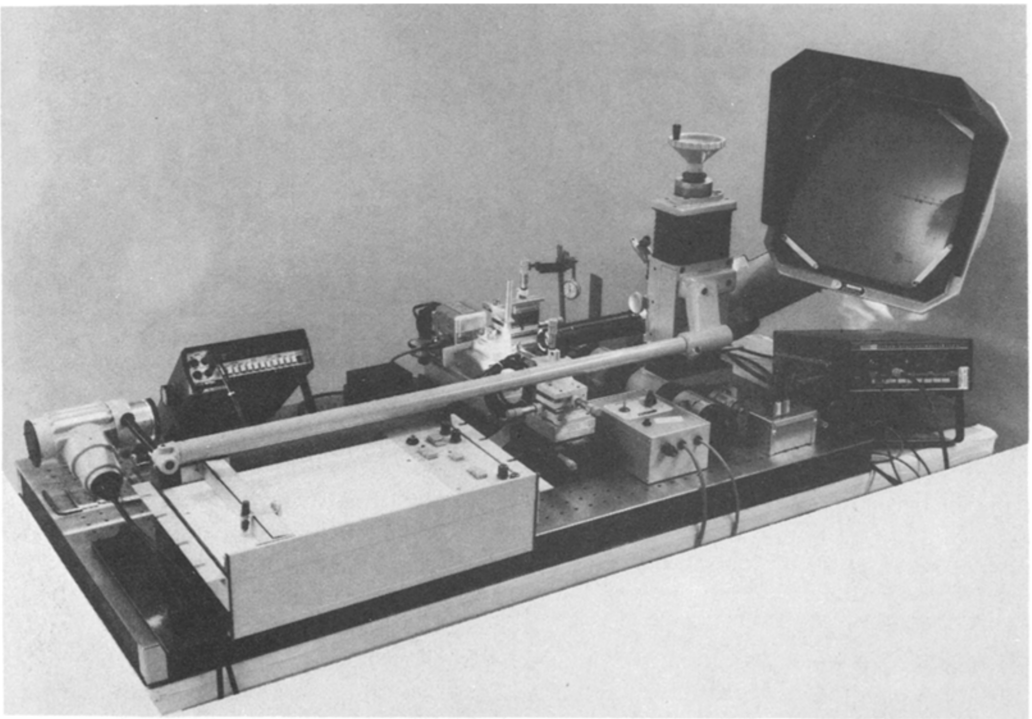

Figure 1 Overall view of micro-tensile tester.

small diameter and limited length dimension. In order to evaluate these materials, it was necessary to design and construct the micro-tensile test device shown in Fig. 1. Other devices for testing whiskers have been developed in the past [8–17], and this experience proved to be very helpful in designing the present unit.

The device in Fig. 1 consists of the following elements. Low load capacity (50 and 100 g) load cells are employed in conjunction with a precision, linear, variable-speed displacement device (0.8 to 9 μm sec^{-1} displacement rates) for loading of the whisker. Both of these elements are mounted on an optical track. X–Y–Z stage manipulators and a 40× vertical viewing tube are used for specimen alignment prior to testing. A 50× horizontal profile projector is employed for specimen alignment as well as measurement of the whisker elastic modulus. Finally, all the device elements are mounted on an optical table for convenience of mutual alignment as well as minimization of vibration. A closer proximity view of the load train is shown in Fig. 2.

2.3. Tensile test techniques

Whisker testing begins with the extraction of a single whisker from the whisker matte, which is done with very fine-tipped tweezers while observing through a stereo microscope. One end of the whisker is then epoxy glued to the bevelled edge of a glass microscope slide. The current epoxy employed is Bipax Tra-bond, a fast curing type, with SiC powder intermixed to increase the elastic modulus of the glue and allow for better load transfer to the whisker. This first joint is allowed to cure for at least 24 h under ambient conditions and once it is made the whisker can be easily handled for subsequent insertion into the testing unit.

The mounted whisker is then clamped in place in a stage mounted on the linear displacement device. A second glass slide is run up under the whisker and the whisker is accurately aligned both horizontally and parallel to the load–displacement axis using a 40× viewing tube from above and the 50× profile projector from the side. Once adequate alignment (0.025 mm or less deviation in the axiality of all load train components) has been established, a second glue joint is made on the load cell side as shown in Fig. 3. A resistively heated wire is employed to accelerate the hardening of this second joint.

Once the whisker is aligned and mounted in the loading train, it is loaded using the variable speed,

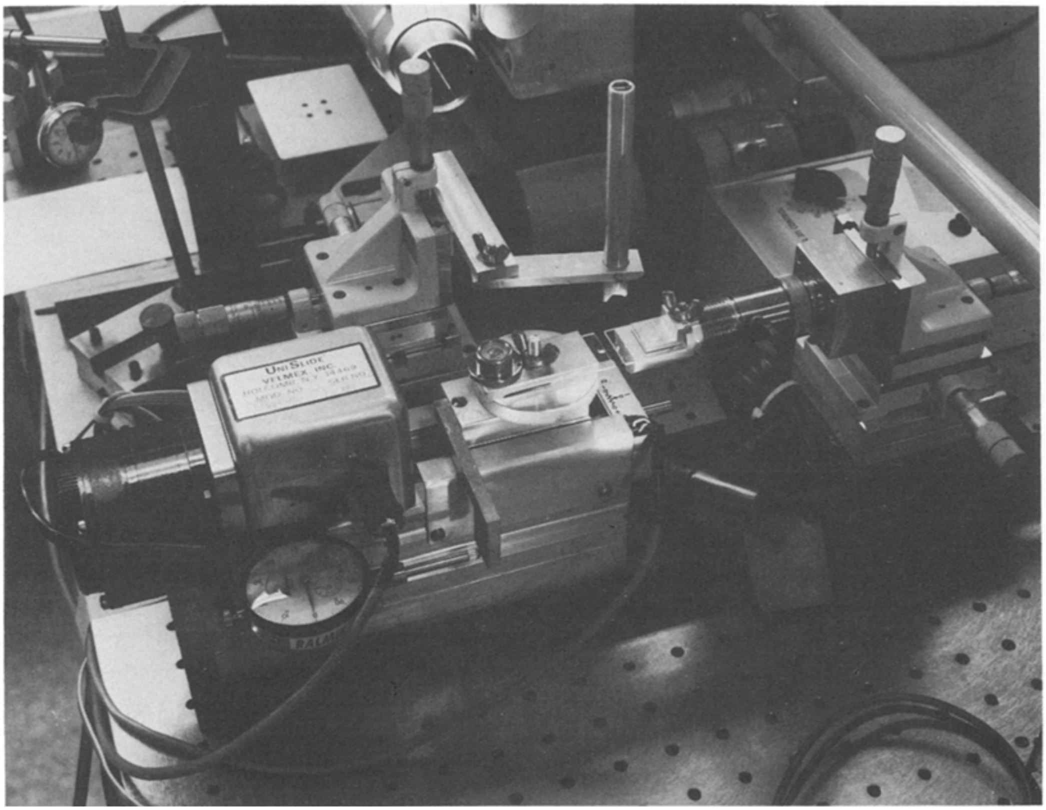

Figure 2 View of whisker tensile loading train.

linear displacement device. This unit allows precision uniaxial loading at continuously variable displacement rates of 0.8 to 9 μm sec^{-1}. Load is measured using either 50 or 100 g capacity load cells, and the load–displacement curve is continuously recorded with an X–Y recorder.

In addition to providing a means for whisker alignment, the 50 x profile projector is essential to the accurate measurement of whisker elastic moduli. For this measurement, an initial gauge section is established directly on the whisker by carefully dabbing on small balls of epoxy. Some of these may be seen on the whiskers in Fig. 3. The whisker is then loaded to a fixed load, the test stopped, and the change in gauge length measured using precision micrometers interfaced with the profile projector. This procedure leads to a straightforward measurement of elastic strain and eliminates any uncertainties associated with load train and glue joint displacements.

In order to calculate both the fracture stress and elastic modulus, it is essential that the whisker cross-sectional area be accurately known. The SiC whiskers were both small (4 to 7 μm equivalent circular diameter) and non-circular in cross-section (rounded triangular shape). Thus, light optical measurements of the whisker cross-sectional area can be in significant error (up to 45%), with this error observed to be non-systematic. For this reason, a calibrated scanning electron microscope (SEM) was employed to make accurate measurements of the cross-section of each tested whisker.

3. Results

3.1. SiC whisker tensile tests

Forty SiC tensile tests were performed in order to characterize the mechanical properties of the whiskers. For these tests, the whisker length under stress was held constant at a length of 5 mm. The distribution of whisker equivalent circular diameters observed is shown in Fig. 4. The average "diameter" (recall that the whiskers actually exhibit a rounded triangular cross-sectional shape) was 5.89 μm, with the smallest tested whisker being 3.34 μm and the largest 10.8 μm. Fracture strength values are shown in Fig. 5. An asymmetrical strength distribution was observed, with an average strength of 8.40 GPa (1 220 000 psi).

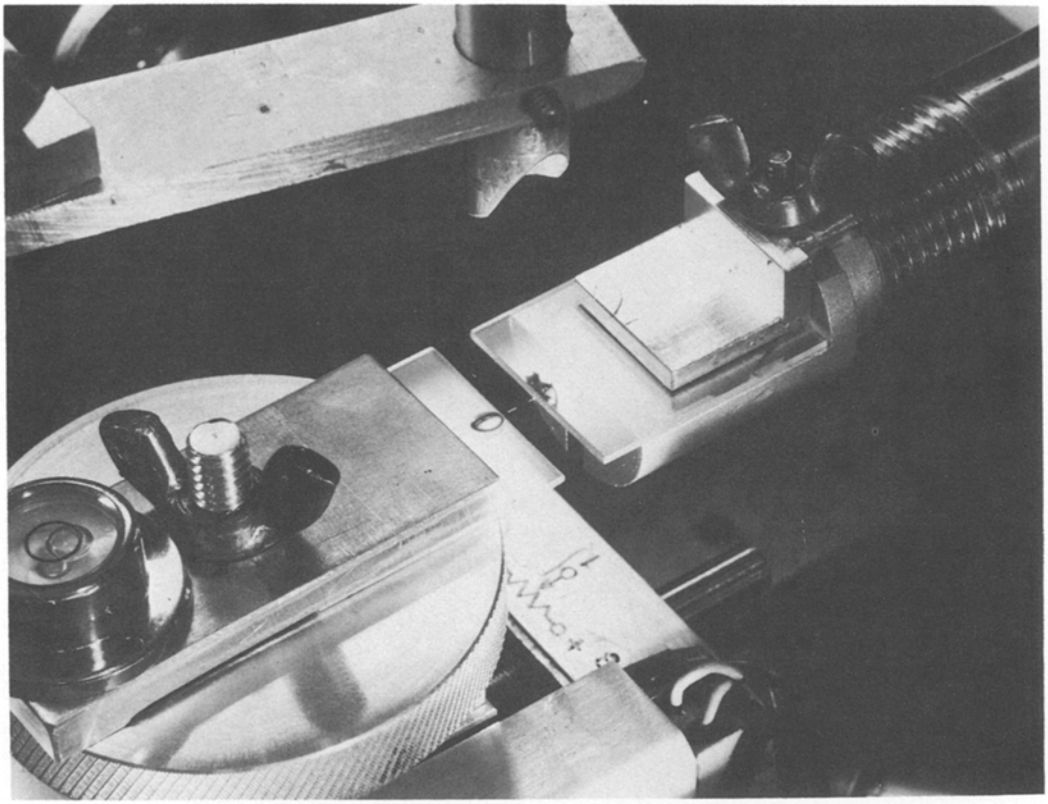

Figure 3 SiC whiskers in place for tensile testing.

The low and high strength values were 1.66 and 23.74 GPa, respectively. The average fracture strain observed was 1.74%, with a range of 0.73 to 2.95%. Fig. 6 shows the elastic modulus distribution obtained. The average elastic modulus was 578 GPa (83 800 000 psi), with a low of 361 GPa and a high of 890 GPa.

Measured strength values exhibited a significant range, with the ratio of high-to-low strength of 14.3. The strength data was analysed in terms of the Weibull statistical fracture theory [18–20]. A plot of probability of fracture versus fracture stress is shown in Fig. 7. A Weibull modulus of 1.77 was obtained from the SiC whisker strength data, with a coefficient of determination of 0.95.

The SiC whisker data were analysed to determine any potential correlations between strength, modulus, and equivalent circular diameter. There was no correlation between strength and diameter, within the data scatter. For the range of diameters tested, strength did not increase with decreasing diameter in any definitive way. The same was true for modulus with diameter. Finally, modulus did not increase with increasing whisker strength, within the data scatter.

3.2. Nicalon fibre tensile tests

In order to obtain the best possible comparison to the SiC whisker mechanical property results, ten Nicalon SiC fibres* were tested with the microtensile tester using the same test techniques. All Nicolon fibres were tested with a 5 mm stressed length, identical to that used for the SiC whiskers. Prior to testing, the protective coating placed on the fibres by the manufacturer was removed by soaking the fibres in acetone.

The average size of the fibres tested was observed to be 17.3 μm, with a range of 11.4 to 26.8 μm. All fibre cross-sections were round in shape. The average fracture stress observed was 2.40 GPa, with a range of 0.52 to 3.53 GPa. The average fracture strain was 1.62%, with a range of 0.80 to 4.31%. Thus, the ratio of high-to-low strength was 6.8. The average elastic modulus was

*Obtained from J. J. Brennan, United Technologies Research Center, East Hartford, Connecticut, USA.

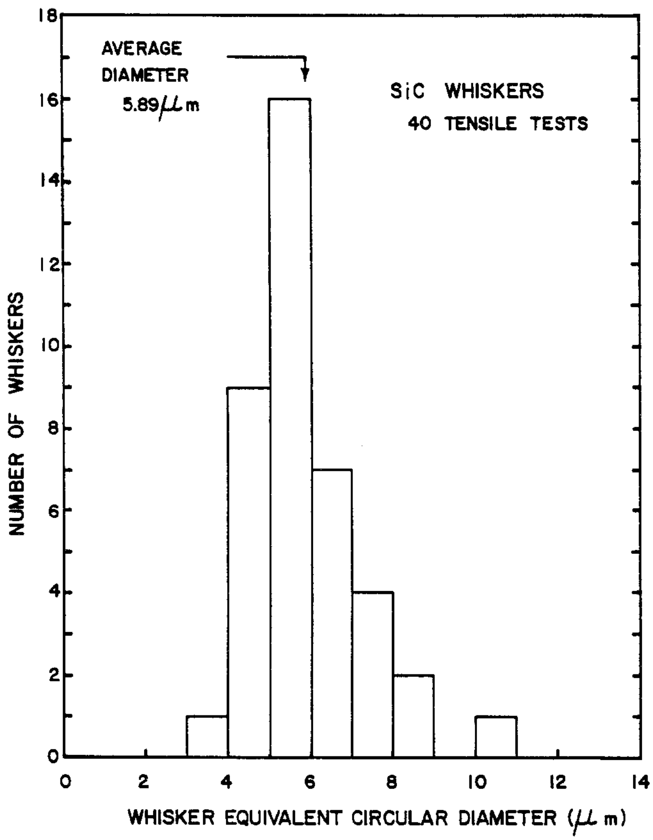

Figure 4 Distribution of SiC whisker equivalent circular diameters.

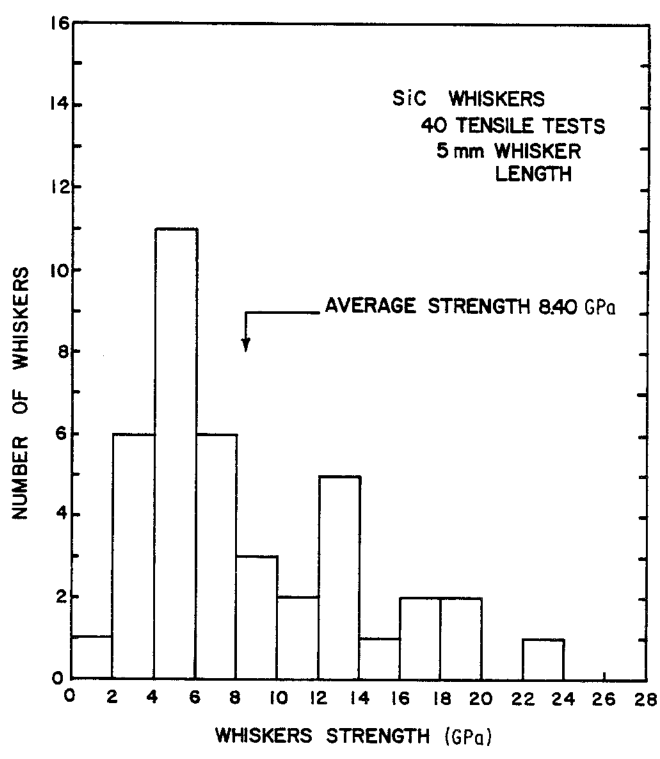

Figure 5 Tensile strength distribution for SiC whiskers.

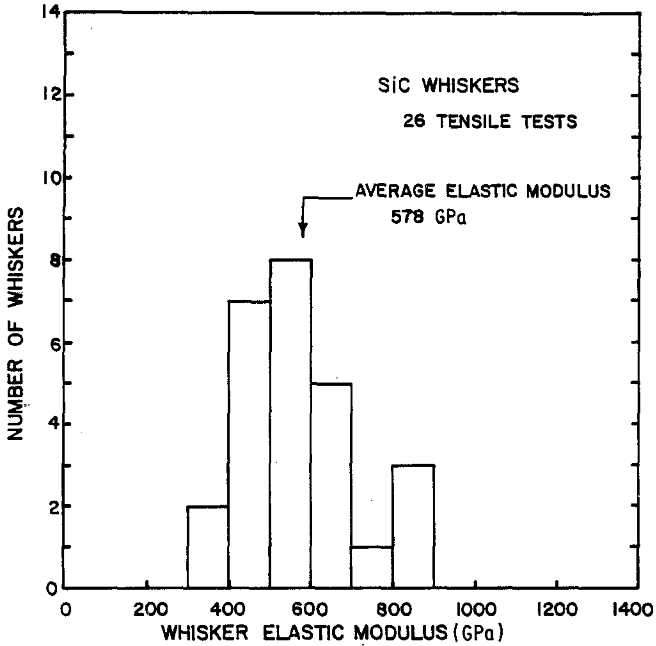

Figure 6 Elastic moduli for 5 mm length SiC whiskers.

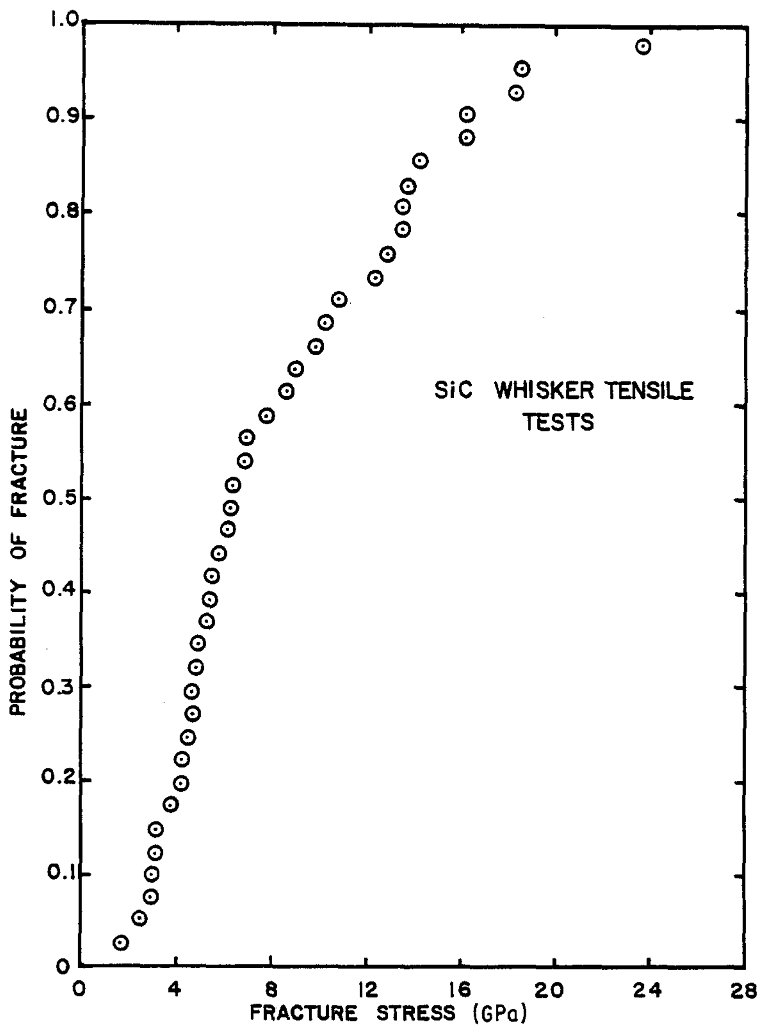

Figure 7 Weibull probability of fracture plotted against fracture stress for SiC whiskers.

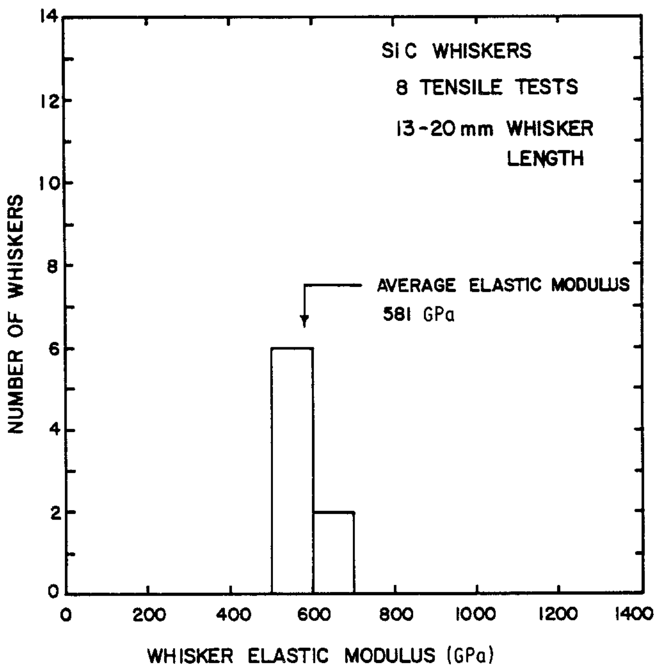

Figure 8 Elastic moduli for 13 to 20 mm length SiC whiskers.

180 GPa, with a range of 21 to 318 GPa. Two of the ten tests exhibited anomalously low mechanical properties, both fracture strength and elastic modulus.

These results on the Nicalon fibres may be compared to mechanical property data obtained by Mah [21, 22] for forty-one tests on as-received fibres. The average fibre diameter reported was 16.3 μm. The average tensile strength was 1.94 GPa, and the average elastic modulus was 112 GPa. Analysis of the strength data yielded a Weibull modulus of $m = 3.67$ for the Nicalon fibres. High-to-low strength scatter was comparable to the present results.

The results of Mah [21, 22] appear low in both strength and elastic modulus as compared to the present Nicalon fibre results. Other workers [23, 24] have reported strengths in the range of 2.1 to 3.2 GPa and elastic moduli in the range of 179 to 200 GPa, more in accordance with the present data. The lower strengths reported by Mah might be accounted for by the fact that their tested fibre length was significantly longer, approximately 23 mm, than in the present case. The low modulus value may be due to glue joint expansion effects, since a gauge section directly on the fibre itself was not employed.

3.3. Long SiC whisker tests

Nine tests were performed on long SiC whiskers (tested lengths in the range of 12.5 to 19.6 mm, effective circular diameters in the range of 3.9 to 9.5 μm) in order to obtain more accurate elastic modulus values (elastic displacements would be larger, thus minimizing experimental errors associated with the measurement of smaller displacements) and to obtain preliminary data on strength against whisker length.

Elastic modulus values are shown in Fig. 8. The average modulus was 581 GPa (84 300 000 psi), with a range of 516 to 650 GPa. The average value compares well to the average value of 578 GPa obtained from tests on 5 mm long whiskers (Fig. 6). Less scatter was observed in the modulus of the longer whiskers as compared to the 5 mm tested whiskers, suggesting that some of the modulus scatter was due to experimental uncertainties associated with the requirement of measuring smaller elastic displacements when the tested whisker length was shorter.

A plot of strength against whisker length is shown in Fig. 9. As may be seen, the longer whiskers do not exhibit any significant weakening. In fact, the average fracture stress of 9.45 GPa was somewhat higher than the average value for 5 mm long tested whiskers. This observation may be significant for the use of longer length whiskers in ceramic composite materials. Limited multiple test data for individual whiskers shown in Fig. 9 do suggest a strong dependence of strength on

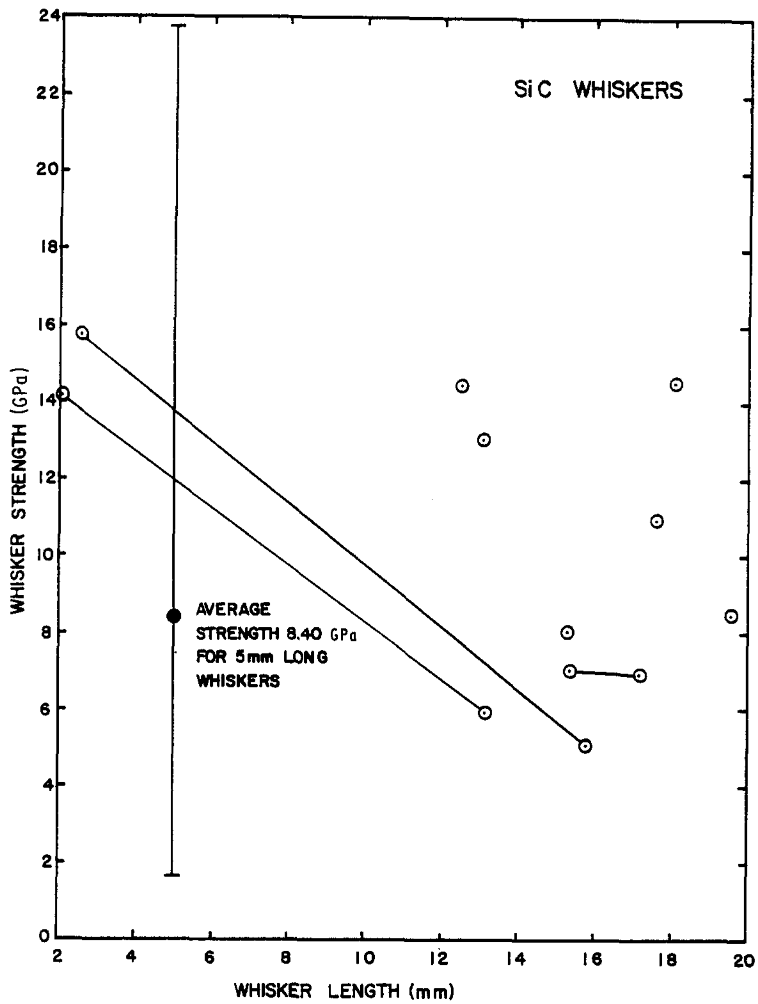

Figure 9 SiC whisker strength plotted against whisker length.

whisker length for an *individual* whisker. The data also indicate that the initial fracture process does not degrade the subsequent mechanical properties of the whisker due to the introduction of defects.

3.4. Scanning electron microscopy on tested whiskers

Due to the rather wide strength variation observed in Fig. 5, scanning electron microscopy (SEM) comparisons of the surface morphologies of selected high and low strength tested whiskers were performed, in order to determine if strength could be correlated to distinct, observable surface features.

The broken ends of seven tested whiskers were examined in the SEM. These whiskers possessed fracture strengths of 1.66, 2.49, 3.16, 6.92, 10.78, 12.37, and 16.20 GPa. They were selected on the basis of fracture strength level, as well as length of whisker remaining after the test. The examination consisted of viewing the entire tested whisker along its remaining length. Results are summarized in Fig. 10, for whiskers of strength 3.16 GPa and 12.37 GPa respectively. Basically, no distinct differences in surface morphology were observed that would correlate with the fracture strength. Both low and high strength whiskers exhibited smooth, relatively featureless surfaces. Thus, the SEM study provided no indication as to the source of the fracture strength scatter observed. In view of these results, one possible explanation for the strength scatter might be that it is associated with handling damage due to harvesting procedures used in the whisker fabrication process. Although no evidences of surface damage were observed, such an effect might be obscured by the fact that the whiskers were examined after testing, when any such damaged regions may have been removed by the fracture process itself.

Fractured cross-sections of the whiskers did not

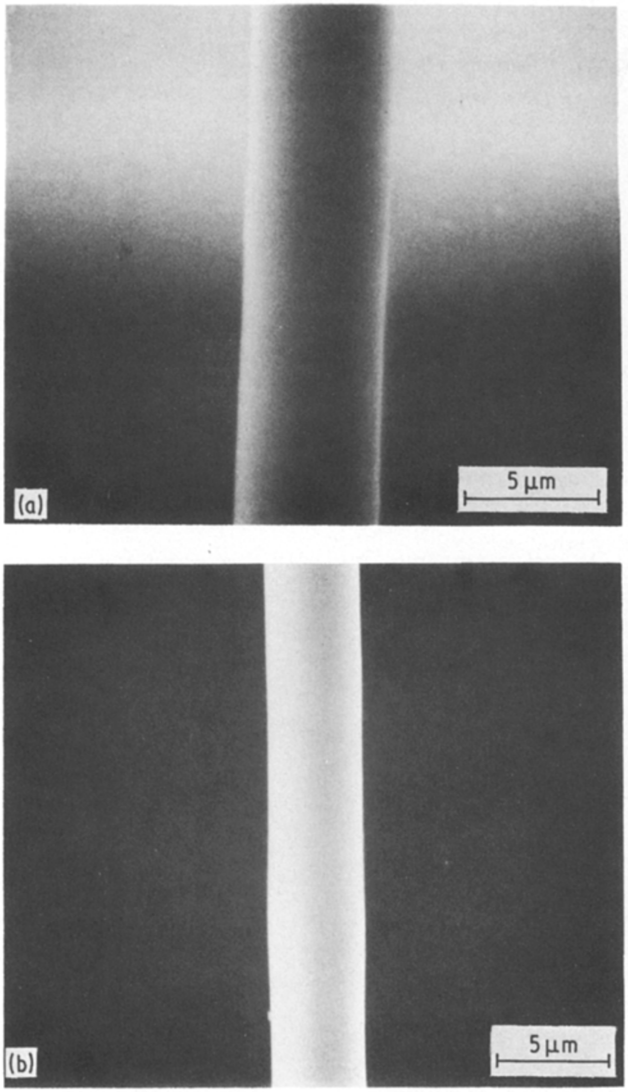

Figure 10 SEM of surfaces of low and high strength whiskers. (a) Fracture strength 3.16 GPa, (b) fracture strength 12.37 GPa.

reveal any distinct features which could be correlated with the strength scatter. A typical fracture cross-section is shown in Fig. 11. This particular whisker fractured at a level of 8.29 GPa. The enlarged area in Fig. 11 does not represent an inhomogeneity in the whisker cross-sectional area along its length, but is an epoxy glue ball on the whisker in the vicinity of the fracture. Measurements of whisker cross-sections at various positions along the lengths of long whiskers showed that there was very little variation in the whisker cross-sectional area with position.

Many fractured whiskers appeared as in Fig. 11, relatively featureless but with faint fracture markings suggestive of a surface fracture. However, no distinct surface defects were ever observed. Other whiskers exhibited cleavage-type steps at the fracture, possibly related to whisker bending once the fracture was initiated. Because the whiskers often tended to shatter into multiple pieces upon fracture due to stress-wave effects, in many cases it was impossible to ascertain whether the observed fracture cross-section was the primary fracture location or a secondary one.

4. Discussion

It is of interest to compare the present mechanical property results on SiC whiskers with what has been reported in the literature. Kelly [25] indicates maximum tensile strengths for SiC whiskers of 21 GPa and elastic modulus of 700 GPa. Koyama and Fukuta report SiC whisker strengths

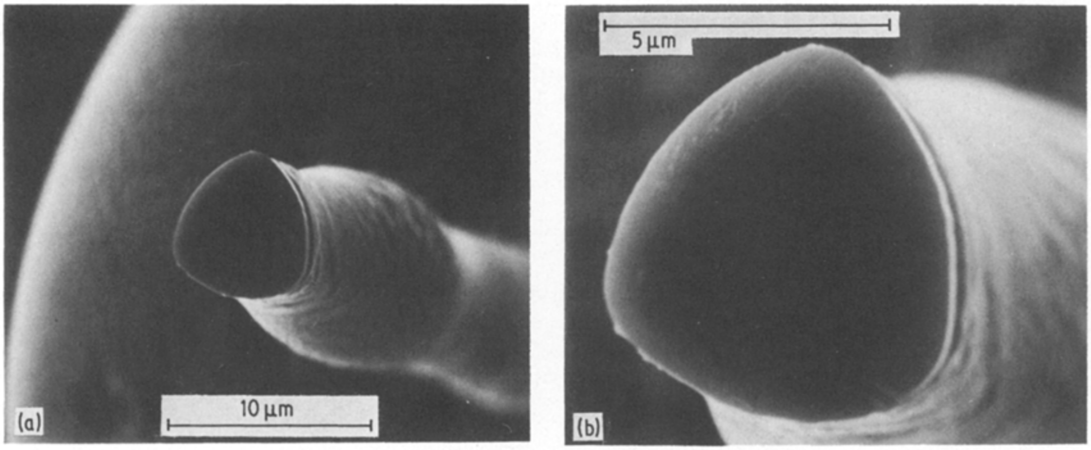

Figure 11 Fracture surface of a SiC whisker.

of 21.6 GPa and modulus of 500 GPa [26]. Op Het Veld and Veldkamp [12] have reported mechanical properties for cubic and hexagonal hair-like SiC whiskers. Their average tensile strength was 12 GPa, with a range of 8.7 to 32 GPa. Elastic modulus values for cubic SiC whiskers with a ⟨1 1 1⟩ growth direction were 530 to 630 GPa. Thus, the present results are certainly comparable with what has previously been observed for SiC whiskers.

The theoretical cleavage strength of a single crystal can be approximated by the Orowan expression [12, 25]:

$$\sigma_{\max} = \frac{1}{2}\left(\frac{E\gamma}{a}\right)^{1/2} \tag{1}$$

where $\sigma_{\max}$ = cleavage strength, E = Young's modulus, γ = surface tension, and a = equilibrium distance of the atomic planes parallel to the fracture plane. Using this expression, Op Het Veld and Veldkamp [12] calculated a theoretical cleavage strength for SiC of 46.3 GPa which was equivalent to a ratio of $\sigma_{\max}/E = 0.067$. Although calculations of this type are only approximate, it does suggest that the highest strength whisker tested in the present investigation (23.74 GPa) possessed a fracture stress close to one-half of the theoretical strength.

Since the β-SiC whiskers are single crystals, it is of interest to determine how the presently determined experimental elastic modulus compares with a calculated modulus for a cubic SiC single crystal in the ⟨1 1 1⟩ direction, which is the whisker growth direction. For a cubic single crystal, the elastic modulus in the ⟨1 1 1⟩ direction is a function of the three elastic compliances as [27]:

$$E_{\langle 111\rangle} = \frac{1}{S_{11} - \frac{2}{3}(S_{11} - S_{12} - \frac{1}{2}S_{44})}. \tag{2}$$

Thus, this calculation requires a knowledge of S_{11}, S_{12} and S_{44} for β-SiC.

Elastic constant data for β-SiC are not extensive. The values indicated by Pandey and Dayal [28] appear to be the best values currently available. Their reported stiffness values for β-SiC are: $C_{11} = 352.3$ GPa, $C_{12} = 140.4$ GPa, $C_{44} = 232.9$ GPa. Stiffnesses may be converted to compliances using the following relationships for cubic crystals:

$$S_{11} = \frac{C_{11} + C_{12}}{(C_{11} - C_{12})(C_{11} + 2C_{12})} \tag{3}$$

$$S_{12} = \frac{-C_{12}}{(C_{11} - C_{12})(C_{11} + 2C_{12})} \tag{4}$$

$$S_{44} = 1/C_{44} \tag{5}$$

Using these compliances, a value of $E_{\langle 111\rangle} = 511$ GPa is calculated. This single crystal value is within 15% of the experimental value of 581 GPa obtained from tests on long SiC whiskers. This agreement is considered quite good in view of the uncertainties associated with the elastic constant values [28], and indicates that whisker elastic modulus values obtained in the present investigation are both realistic and accurate.

The present investigation of SiC whisker mechanical properties indicates that these properties are significantly higher than those of Nicalon fibres. Compared to Nicalon, SiC whisker strengths are greater by a factor of 3.5 while the elastic modulus is higher by a factor of 3.2. However, due to the limited length of the SiC whiskers they cannot be employed to synthesize continuous fibre composites as is possible with the Nicalon fibres. Rather, the superb mechanical properties of the whiskers coupled with their inherent elevated temperature stability due to their phase-pure nature makes them excellent candidates for short-fibre reinforcement elements in high-temperature ceramic matrix composites.

5. Conclusions

1. Using a specially developed micro-tensile tester, SiC whiskers 5 mm in length have been shown to possess an average tensile strength of 8.40 GPa (1220 000 psi) and an average elastic modulus of 581 GPa (84 300 000 psi). However, a large amount of strength scatter was observed, the source of which is as yet undetermined.

2. In a direct comparison, limited-length SiC whiskers were observed to be 3.5 times stronger and 3.2 times stiffer than commercially available continuous Nicalon polycrystalline SiC fibres.

3. The high mechanical properties of SiC whiskers indicate their significant potential as short-fibre reinforcement elements in high-temperature ceramic matrix composites.

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