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# Tensile fracture behaviour of long SiC whiskers

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The tensile mechanical properties of long (up to 77 mm length) SiC whiskers have been investigated using multiple-fracture testing. SiC whiskers fractured from two types of defects. Below approximately 10 GPa stress, fracture occurred from extraneous surface defects, such as foreign matter particles and growth appendages. Above 10 GPa, fracture initiated from intrinsic defects. The Weibull modulus for intrinsic whisker fracture was determined to be in the range 3 to 4. Tests on very long whiskers indicated a strength plateau at 16 GPa for intrinsic whisker fracture. A basic statistical fracture description for the intrinsic fracture of whiskers was derived, which predicts a plateau in the intrinsic whisker strength.

## 1. Introduction

Recent studies have shown that SiC whiskers possess excellent potential as reinforcement for the synthesis of high toughness ceramic composites [1–6]. SiC whiskers have been synthesized at Los Alamos by a vapour–liquid–solid (VLS) process and initial measurements of their mechanical properties made [7, 8]. The VLS process has yielded experimental quantities of SiC whiskers with lengths as long as 100 mm.

In the previous whisker mechanical property investigation [8], a relatively wide whisker strength distribution was observed. The present investigation was initiated in order to examine the reasons for this wide strength distribution by performing multiple-fracture tests on long SiC whiskers. Goals of the investigation were to characterize SiC whisker strength with respect to length, to determine the types of defects initiating whisker fracture, to establish the inherent statistical fracture properties of SiC whiskers, and to determine the mechanical properties of long SiC whiskers.

## 2. Experimental details

Tests were performed using the micro-tensile-tester and testing techniques described by Petrovic *et al.* [8]. Multiple-fracture tests involved initially testing a long SiC whisker to fracture, then progressively testing shorter fragments until finally the fragments became too short for test. In addition, the surface morphology of the entire length of each long whisker was characterized using the SEM *prior* to the commencement of multiple-fracture testing in order to identify and locate the positions of prominent surface features, so as to correlate the fracture location/behaviour with defects present on the whisker surface. Finally, multiple-fracture tests were performed with the whisker immersed in distilled water in order to minimize whisker shattering due to stress wave propagation and thus obtain more tests for each individual whisker.

## 3. Results

Table I shows the cross-sectional uniformity of the long SiC whiskers. These dimensional measurements were made in the SEM. The average variation in cross-sectional dimensions over separation distances of 7 to 17 mm was 10%. Measurements made on two very long SiC whiskers indicated cross-sectional variations of 8% and 22% over a 75 mm separation distance. These cross-sectional variations were non-systematic and did not constitute a gradual taper in the whisker cross-section from the whisker base to the catalyst ball in the VLS growth process. The SiC whisker cross-sectional variations observed were considerably less than those reported for Nicalon fibres, where variations as high as 300% have been observed [9–11].

Results of multiple fracture tests for SiC whiskers with an initial tested length as long as 25 mm are shown in Fig. 1. A significant increase in strength with decreasing whisker length was observed, with strength being a non-linear function of length for the individual whiskers. Fig. 2 plots all multiple-fracture data without regard to association with individual whiskers. In this form, average strength levels are shown in 4 mm length intervals, and an approximately linear decrease in strength with increasing whisker length is observed. In the range of 0 to 4 mm length a strength value of 15.9 GPa (2 310 000 psi) was obtained, while at 24 to 28 mm length the average strength was 7.5 GPa (1 090 000 psi).

From Fig. 2, one will note that the average strength value at 5 mm whisker length is 14.6 GPa. This may be compared to the value of 8.4 GPa obtained from 40 tensile tests on whiskers with a constant 5 mm tested length [8]. The reason for the higher value obtained here is that for multiple-fracture tests the more severe defects are being successively eliminated by previous fractures, while for tests at constant length such defects are included in the test population, thus reducing the overall strength level. From the viewpoint of composite material synthesis, the strength values shown in Fig. 2 are probably the most applicable, since whisker comminution processes applied to adjust the $L/D$ ratio prior to incorporation into the composite would have the same function as multiple-fracture

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TABLE I Cross-sectional uniformity of long SiC whiskers. Measurements on two very long SiC whiskers indicated cross-sectional variations of 8% and 22% over a 75 mm length

| Distance between measurement positions (mm) | | % difference in whisker width |
|---|---|---|
| 9.25 | | 5.64 |
| 12.85 | | 2.77 |
| 12.17 | | 8.35 |
| 7.52 | | 16.43 |
| 17.35 | | 1.99 |
| 7.37 | | 18.52 |
| 9.05 | | 18.19 |
| 16.64 | | 12.09 |
| 14.15 | | 9.46 |
| | Average | 10.38% |

tests in eliminating more severe defects, assuming, of course, that additional severe defects are not introduced by the comminution process itself.

In the test series shown in Fig. 1, we were able to associate the lower strength fractures with the locations of prior surface defects on the whiskers, strongly indicating that these defects initiated the fractures. Such surface defects were whisker growth appendages and foreign matter on the whisker. Typical examples are shown in Fig. 3. Foreign matter on the whiskers was observed to contain the elements iron, calcium, copper, zinc, sodium, nickel, chromium, cobalt and magnesium. The lineal density of such defects was estimated at 0.184 defects/mm, which translates into a 5.43 mm whisker length between each large defect. This result is consistent with the large scatter in strength values observed for the 40 whiskers tested at 5 mm length [8], since a number of those whiskers would have possessed a severe defect within that length.

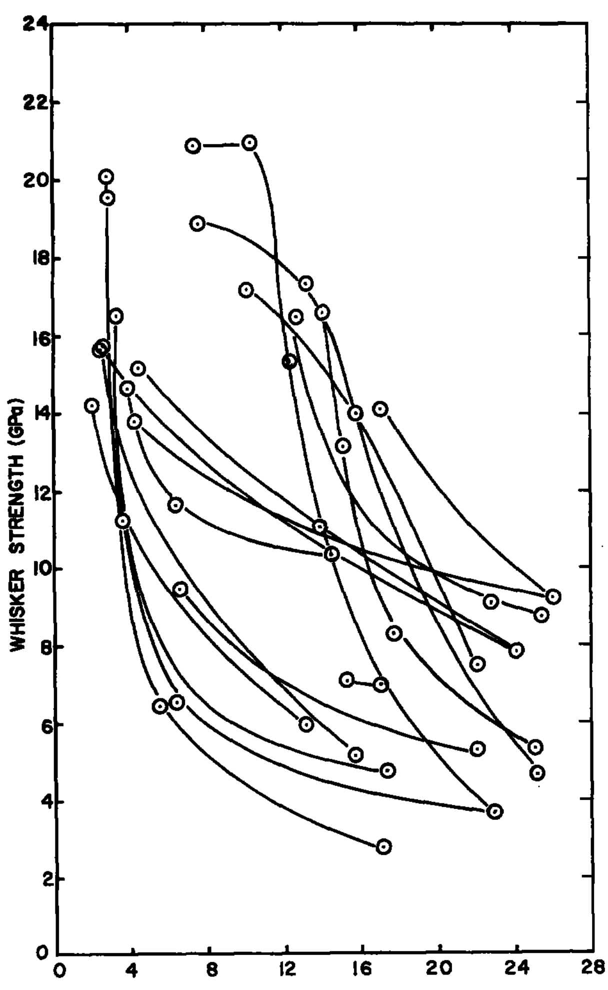

*Figure 1* Multiple-fracture tests on SiC whiskers initially 25 mm in length.

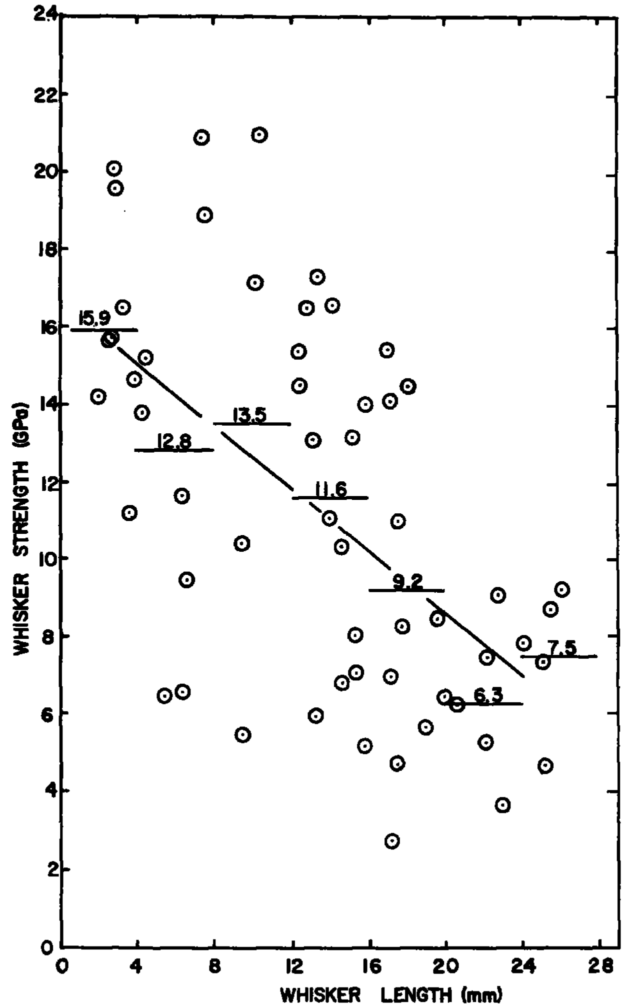

*Figure 2* Multiple-fracture data for 25 mm long SiC whiskers, plotted without association to individual whiskers.

Once these extraneous large surface defects have been removed by multiple-fracture, the fracture then becomes controlled by intrinsic defects in the whisker, which are less severe, leading to substantially higher strength levels. In Fig. 1, the high stress level fractures occurred at whisker locations which were free of any surface defects. From Fig. 1, the strength level transition between extraneous surface defects and intrinsic defects appears to be in the vicinity of 10 GPa.

The data presented in Fig. 1 may be analysed using the Weibull statistical fracture theory [12–14]. For uniaxial tensile loading, the Weibull theory predicts a volume effect on strength as:

$$\sigma_2/\sigma_1 = (V_1/V_2)^{1/m} \quad (1)$$

where $\sigma_2$ is the strength of the specimen with volume $V_2$ and $\sigma_1$ is the strength of specimen with volume $V_1$. For tests on whiskers of different lengths but constant cross-sectional area, this relation becomes:

$$\sigma_2/\sigma_1 = (L_1/L_2)^{1/m} \quad (2)$$

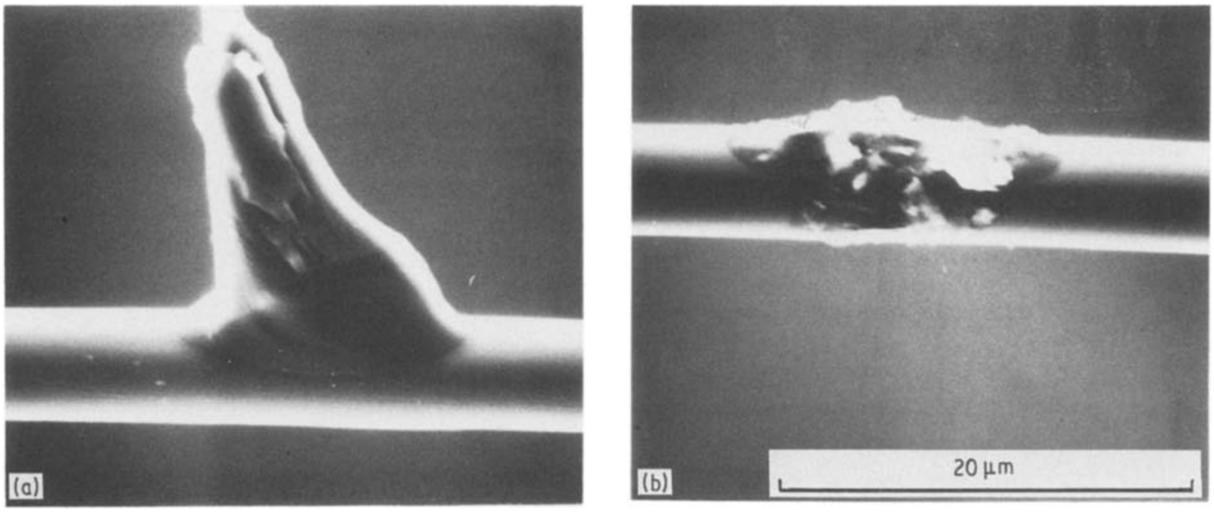


*Figure 3* Typical extraneous surface defects on SiC whiskers. (a) Appendage on whisker, (b) foreign matter on whisker.

where $L_1$ and $L_2$ are the whisker lengths. Thus, by comparing two whisker tests at different lengths, the Weibull modulus, $m$, may be calculated from

$$m = \ln (L_1/L_2)/\ln (\sigma_2/\sigma_1) \quad (3)$$

This calculation assumes that the same types of defects cause fracture at $L_1$ and $L_2$, with only a difference in defect size.

Using all the data in Fig. 1, the average Weibull modulus calculated from Equation 3 is $m = 1.96$. This value is in excellent agreement with the value of $m = 1.8$ obtained from 40 tensile tests at a constant 5 mm length [8], as would be expected.

Weibull modulus calculations using Equation 3 assume that the same types of defects initiate fracture. However, our results indicate that extraneous surface defects initiate low strength fractures while intrinsic defects initiate high strength fractures, with the transition at approximately 10 GPa strength level. It is thus of interest to calculate the Weibull modulus using only multiple-fracture data above 10 GPa. For this situation, a Weibull modulus of $m = 3.21$ is obtained and this value should be more representative of the inherent whisker fracture process. Thus, this value is the more accurate one to employ in estimating the strength of SiC whiskers at low values of $L/D$ ratio as would be employed in ceramic composite materials.

Multiple-fracture tests were performed on two very long SiC whiskers. The first whisker was 77 mm in initial tested length, while the second was 76 mm in initial length. Eight multiple-fracture tensile tests were obtained on the first whisker, while five were obtained from the second whisker. All the multiple-fracture tests reported occurred sequentially along the same direction with respect to the whisker length, so that the flaw population was sequentially sampled by each successive fracture test. These test results are shown in Fig. 4.

As may be seen in Fig. 4, the first very long SiC whisker exhibited a gradual increase with decreasing tested length until at approximately 30 mm length the strength increased abruptly to a plateau region at approximately 16 GPa. The lower strength fractures (i.e. those occurring at lengths of 30 mm and above) were clearly associated with surface defects on the whisker of the type shown in Fig. 3. However, the high strengths in the plateau region could not be associated with any observable whisker surface defects, suggesting that the fractures at these high strength levels were intrinsically controlled. Thus, this long whisker test series clearly indicates a transition in fracture initiating flaw type, since once the surface defect flaws had been all eliminated by previous fractures, the intrinsic defect flaws became responsible for fracture initiation, causing an abrupt increase in the fracture strength. For this whisker, strengths in the plateau region were observed to actually decrease slightly with decreasing length for the shortest lengths tested, which suggests the possibility that damage was being introduced into the whisker by the succession of very high stress fracture processes.

The second very long whisker tested showed an initial strength level for the 76 mm length which was close to that observed in the first whisker test. However, in this case the strength increased more gradually with decreasing length, and the abrupt increase in strength as was noted in the first test series was not as distinctly observed. There does seem to be an indication of the attainment of a strength plateau region at strength levels close to that observed for the first very long whisker, but shattering of the whisker on the fifth multiple-fracture test precluded the acquisition of further data in the plateau region.

In Fig. 4, it is interesting to note that the first whisker possessed a strength of approximately 7 GPa (1 000 000 psi) at a 30 mm length, while the second exhibited a strength of approximately 11 GPa (1 600 000 psi) at a 40 mm length. This indicates the potential which exists for the use of long SiC whiskers as reinforcement in continuous fibre ceramic composites.

## 4. Discussion

It is of interest to compare the Weibull parameters obtained from the SiC whisker multiple-fracture data above 10 GPa, where the whisker fracture appears to be intrinsically controlled, to those which have been observed from the mechanical testing of larger SiC

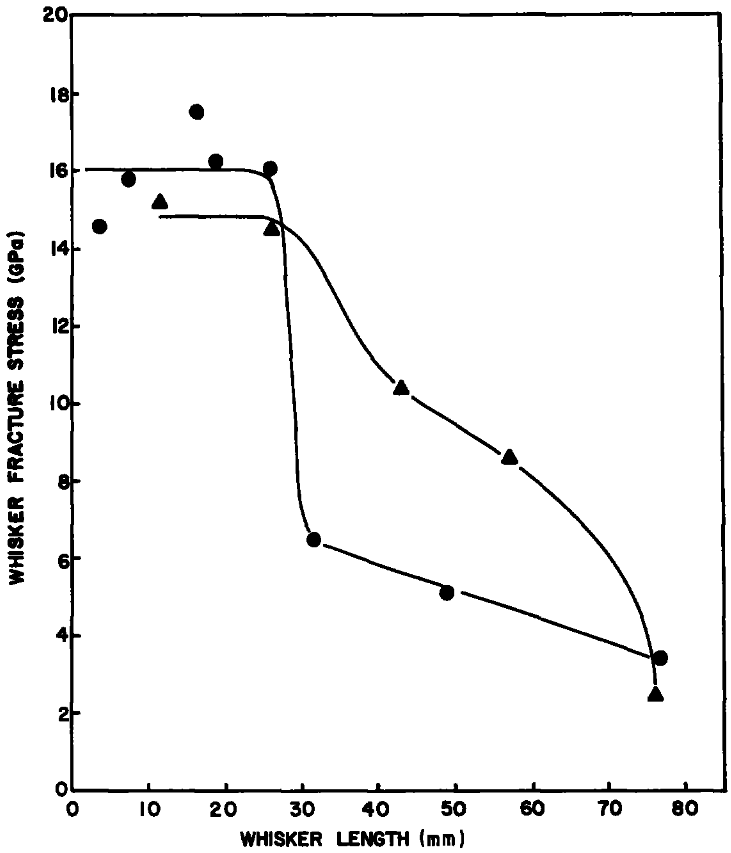


*Figure 4* Multiple-fracture tests on very long SiC whiskers.

single crystals. The only such data available in the literature appears to be the investigation of Batha and Hasselman [15, 16], where a limited number of SiC single crystals were mechanically tested. Relatively large platelet morphologies (up to 0.6 mm thick and 12.7 mm diameter) were grown from the vapour phase. Crystal structures were typically either 6H or 4H alpha-SiC. The SiC crystals were green in colour. Ten crystals were mechanically tested in three-point bending, using specimens of 9.53 mm length and 3.18 mm width. The specimen length was either parallel or perpendicular to the $(1\bar{1}00)$ growth edge. Fracture of specimens occurred primarily along the $(1\bar{1}00)$ planes.

The high purity, green SiC crystals showed an average strength of 375 MPa, with a scatter of 201 MPa to 586 MPa. There was no distinct effect of crystal orientation on the mechanical properties. Using these ten data values of Batha and Hasselman, the Weibull modulus was calculated in the usual way [17] assigning failure probabilities as $P_f = n/N + 1$, and also by assigning failure probabilities as $P_f = (n - 1/2)/N$, which has been reported [18] to give a better estimate of the true Weibull modulus for small groups of specimens (typically ten or less). The Weibull modulus using the method in [17] was calculated to be $m = 3.38$, while that using the method in [18] was $m = 4.22$. These Weibull modulus values obtained for high-purity, green, large SiC single crystals are close to the value of $m = 3.21$ obtained for SiC whiskers from multiple-fracture data above 10 GPa. This comparison suggests that Weibull moduli in the range 3 to 4 are to be expected for SiC single crystals.

The tensile fracture of single crystal SiC whiskers constitutes an elegantly simple fracture system. This being the case, we have attempted to model whisker fracture from intrinsic defects (perhaps substructural in nature) using basic probability of fracture concepts. This analysis is outlined below.

Divide the whisker length, $L$, into equal smaller lengths $\Delta L$, with each of these smaller lengths having the same probability of failure function $P_0$. The probability of survival of whisker of length, $L$, under stress is:

$$(1 - P_L) = (1 - P_0)_1(1 - P_0)_2(1 - P_0)_3 \ldots (1 - P_0)_n = (1 - P_0)^n \quad (4)$$

where $n = L/\Delta L$. Equation 4 constitutes the basic starting point for the Weibull statistical fracture theory [12–14]. The probability of whisker failure then becomes:

$$P_L = 1 - (1 - P_0)^{L/\Delta L} \quad (5)$$

To proceed further requires a form for the probability of failure function $P_0$ for each $\Delta L$ segment. We assume a very simple function as:

$$P_0 = \frac{(\sigma/\sigma_c) - \alpha}{1 - \alpha} \quad (6)$$

where $\sigma$ is the stress, $\sigma_c$ is the theoretical cohesive strength of the whisker, and $\alpha$ is a fraction less than one. Equation 6 states that if we tested a large number of individual $\Delta L$ whisker elements, none would fail below $\sigma < \alpha\sigma_c$, all would fail for $\sigma > \sigma_c$, with $P_0$ a linear function of $\sigma/\sigma_c$ in the range $\alpha \leqslant \sigma/\sigma_c \leqslant 1$.

Using this form for $P_0$, the probability of whisker failure becomes:

$$P_L = 1 - \left\{1 - \left[\frac{(\sigma/\sigma_c) - \alpha}{1 - \alpha}\right]\right\}^{L/\Delta L} \quad (7)$$

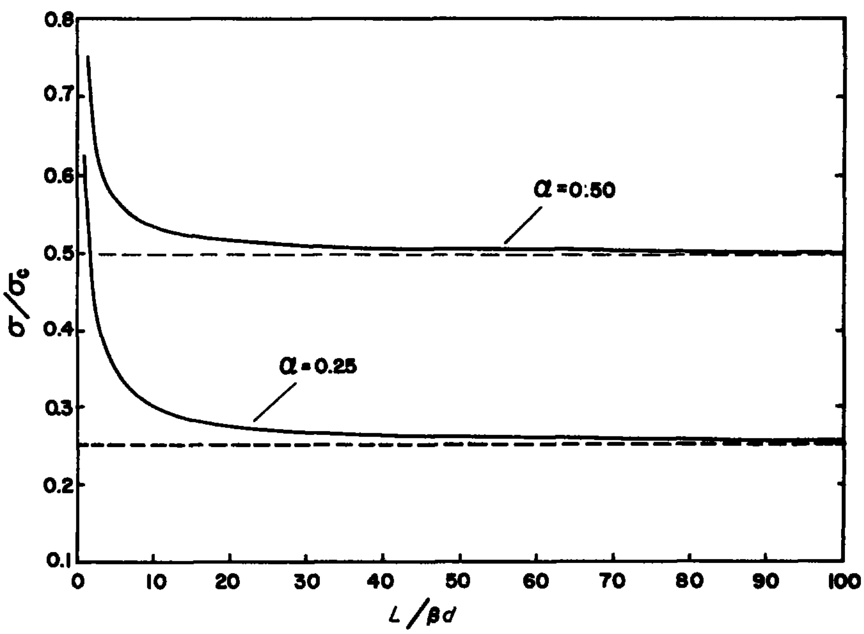


*Figure 5* Predicted relationship between intrinsic whisker strength and whisker length, from Equation 9.

If one further assumes that $\Delta L$ will be given by some multiple of the equivalent circular diameter $d$ of the whisker, $\Delta L = \beta d$, then the final form of the whisker failure probability is:

$$P_L = 1 - \left\{1 - \left[\frac{(\sigma/\sigma_c) - \alpha}{1 - \alpha}\right]\right\}^{L/\beta d} \qquad (8)$$

It seems likely that $\beta > 1$.

The above formulation, while rooted in the same basis as the Weibull theory, does not contain assumptions of the Weibull theory which may not be applicable to the intrinsic fracture behaviour of a SiC whisker. Rather, the assumptions made in developing Equation 8 may be a more realistic description of the actual whisker fracture statistical nature. We may employ Equation 8 to determine how the whisker *intrinsic* strength might be expected to vary with whisker length. For $P_L = 0.5$ (50% probability of whisker fracture), the form of the predicted fracture stress is:

$$\frac{\sigma}{\sigma_c} = \alpha + (1 - \alpha)\,[1 - (\tfrac{1}{2})^{\beta d/L}] \qquad (9)$$

This expression depends on the term $\alpha$, the ratio of the applied stress to the theoretical cohesive strength. Based on our long whisker tests, a value of $\alpha$ in the range of 0.25 to 0.5 might appear reasonable, since intrinsic fracture occurs in the range 10 to 16 GPa, while the cohesive strength of SiC is approximately 40 GPa [8].

Using $\alpha = 0.25$ and $\alpha = 0.5$, predicted whisker strength plotted against whisker length is shown in Fig. 5. One may see that the strength increases with decreasing length only at very short lengths ($L/\beta d < 30$). At longer lengths, the strength approaches the constant value of $\alpha$. Our long SiC whisker tensile test results (Fig. 4) suggested the possibility of a constant strength, plateau region for high strength, intrinsic fracture. Since these tests were in the regime $L/\beta d \gg 30$, the experimental data seem to be in agreement with the present theoretical predictions.

With regard to the intrinsic fracture of SiC whiskers (that is, fracture not initiated by extraneous surface defects), the question to be addressed is What is the nature of the fracture initiating defect? On rare occasion, a SiC whisker has been observed to fail from a distinct internal defect [19]. The diameter of this defect was 329 nm. This allowed the determination of a SiC whisker fracture toughness value of 3.23 MPa m$^{1/2}$. However, this whisker fractured at a strength level of only 7 GPa, and this fracture did not constitute the intrinsic fracture occurring at strength levels of 16 GPa, where no distinct internal defects were observed. We can attempt to estimate the intrinsic flaw size using the expression for fracture of an internal penny-shaped flaw in an infinite medium in unixial tension [20]:

$$K_c = \frac{2}{\pi}\,\sigma_F(\pi a)^{1/2} \qquad (10)$$

where $\sigma_F$ is the fracture stress and $a$ is the flaw radius. Using the measured value of $K_c = 3.23$ MPa m$^{1/2}$ for a SiC whisker and a fracture stress of 16 GPa from Fig. 4, the calculated flaw radius from Equation 10 is $a = 32$ nm. Thus, the diameter of the intrinsic defect would be 64 nm.

It is interesting to speculate as to what type of defects initiate intrinsic fracture of the SiC whiskers. Certainly the fracture surfaces of the whiskers do not present significant insights in this regard, being rather featureless in SEM observation. Some possibilities would seem to be surface defects such as local variations in the whisker diameter or steps on the whisker surface, and also substructural defects such as stacking faults, twins, and dislocations [21, 22]. Clearly, this is an area which requires further investigation.

## 5. Conclusions

The following conclusions may be drawn from the present investigation:

1. The fracture of long SiC whiskers is controlled by two different types of defects. Below a stress level of approximately 10 GPa, fracture occurs from extraneous surface defects such as foreign matter

particles and growth appendages. Above 10 GPa, fracture is initiated from intrinsic whisker defects.

2. The strength of long SiC whiskers increases with decreasing whisker length, until a strength plateau region in the vicinity of the 16 GPa occurs at shorter whisker lengths. This plateau region represents fracture from intrinsic defects.

3. From multiple-fracture tests, the Weibull modulus for the intrinsic fracture of SiC whiskers is in the range of 3 to 4. This value is in good agreement with the Weibull modulus obtained from the mechanical testing of large SiC single crystals.

4. A statistical fracture model was derived for the intrinsic fracture of SiC whiskers, based on a first principles probability approach. This model predicts a plateau region for whisker strength, in agreement with experimental results.

5. The nature of the defects initiating intrinsic fracture of SiC whiskers was not determined in the present investigation. Some possibilities include surface roughness, stacking faults, twins, and dislocations.

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