JOURNAL OF MATERIALS SCIENCE 10 (1975) 608–612

Surface damage and environmental effects on the strain-rate sensitivity of the strength of sapphire and silicon carbide filaments

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c-axis sapphire and silicon carbide-on-carbon filaments with pristine and damaged surfaces were tensile tested in water and dry toluene at various strain rates. There was no evidence of slow crack growth during the testing of the silicon carbide filaments. However, the surface damaged sapphire filaments exhibited slow crack growth in the water environment. The slope of the stress intensity factor versus crack velocity curve in region I of the characteristic curve was found to be 28 in excellent agreement with previous results from slow crack growth studies in which the stress corrosion phenomenon is adequately explained by the Hillig-Charles mechanism. The corresponding tests in dry toluene produced no evidence of slow crack growth. The sapphire filaments with virgin surfaces exhibited slow crack growth in both water and toluene which is apparently not related to the moisture assisted growth observed in the abraded filaments.

1. Introduction

Published schemes for predicting strengths of unidirectionally reinforced, continuous filament composites use a statistical description of the strengths of the filaments of interest (see for example, [1-4]). The most often employed strength distribution function is the Weibull. Phoenix has recently pointed out that the treatment of the position and failure strength of various types of flaws occurring along a fibre as a compound Poisson process is a more general approach to the statistical representation of strengths in brittle fibres, of which the Weibull distribution is a special case [5]. However, to use these approaches to predict composite strengths one must have data for filaments *characteristic* of the *in situ* filaments. In addition, the effects of dynamic fatigue must be included in these determinations since the testing strain-rate can markedly affect the experimental strength distribution. Yamada has recently used slow crack growth data (stress intensity factor versus crack velocity curves) to demonstrate the effect of testing strain-rate on the experimental strength distributions of glass rods [6].

Sapphire [7] and silicon carbide [8] filaments are both candidates for high temperature, high performance composite reinforcements. Relatively little data concerning gauge length, surface damage, and environmental effects on the strength distributions of these filaments have been reported. In this paper we report on a study of environmental effects on the strain-rate sensitivity of the strengths of virgin and surface damaged sapphire and silicon carbide-on-carbon filaments. The strength distributions of the filaments as a function of gauge length and surface damage are treated in a separate paper.

Previous studies of the strength of sapphire have documented the susceptibility of Al_2O_3 to stress corrosion in environments containing moisture [9, 10]. In addition, Pollock and Hurley have recently found that "virgin" sapphire, apparently strength limited by internal flaws, exhibit strengths which are strain-rate sensitive, indicating slow crack growth in the absence of a corrosive environment [11]. There is no published information concerning the stress corrosion susceptibility of or slow crack growth in silicon carbide in the absence of plastic flow at the crack tip.

A recent technique described by Evans and

Weiderhorn [12] and Davidge *et al.* [13] is very conveniently applied to constant strain-rate tensile strength results on filaments to determine the slope of the stress intensity factor versus crack velocity (K_I versus V) curves when slow crack growth occurs during testing. They have shown that the ratio of fracture strengths of specimens tested at strain rate $\dot{\epsilon}_1$, σ_1, to the strengths of specimens at strain rate $\dot{\epsilon}_2$, σ_2, is given by

$$\frac{\sigma_1}{\sigma_2} = \frac{\dot{\epsilon}_1}{\dot{\epsilon}_2}^{1/(n+1)}$$

where n is the slope of the (K, V) curve in region I. By testing an equal number of specimens at two quite different strain rates ranking the strengths in increasing order, and plotting the ordered strengths on logarithmic axes, one should find a straight line of slope equal to 1 and an intercept that includes n.

2. Experimental method

The $\simeq$ 0.025 cm diameter sapphire filaments used in this study were single crystal c-axis (c-axis parallel to the filament axis) material prepared by Tyco Laboratories* using a technique described elsewhere [14]. Details of the microstructure and fracture modes have also been reported previously [7]. The filaments were received with a coating of paraffin or epoxy resin containing silane which was applied during the filament growth process. This coating was removed by heating the filaments, supported by a platinum wire rack, at 800°C for 4 h in an oxidizing environment. Great care was taken to avoid touching the filaments in the eventual gauge length. The surface damaged filaments were prepared by tumbling the filaments in a paper tube mill for 4 h which caused self abrasion to strengths characteristic of filaments in metal matrix composites [15].

The $\simeq$ 0.01 cm diameter silicon carbide filaments were received from Avco† and were the recently developed chemical vapour deposited silicon carbide on a carbon substrate [8]. These filaments were very resistant to self-abrasion due to the carbon rich final deposition on the filament surface. The SiC filaments were abraded by applying a constant pressure normal to the filament surface while the samples were clamped between two sheets of 600 grit SiC grinding paper. The filaments were rotated with the clamping pressure held constant to create surface abrasion with the flaws introduced perpendicular to the fibre axis.

The 7.5 cm gauge length samples were mounted in hypodermic needles with structural epoxy. The gripping arrangement was the pivot bearing method described elsewhere [16]. Each hypodermic needle base rests in a pivot bearing which allows the needle to rotate and eliminate off-axis loading. The tensile testing was performed in a constant cross-head speed, table model Instron testing machine. Cross-head speeds giving strain rates a factor of 200 apart were used. An environmental chamber was built around the bottom grip to allow the sample to be submerged in water or toluene during testing.

Filament fragments were recovered for SEM evaluation of fracture surfaces to attempt to identify the fracture origins.

3. Results and discussions

If dynamic fatigue due to slow crack growth is operative, one should observe higher average fracture strengths at higher strain rates since the time available for crack extension is less. In Table I the average strengths of the virgin and abraded sapphire and silicon carbide tested in toluene and water at the slow and fast strain rates are tabulated. In the case of the silicon carbide filaments, applying statistical tests for significant differences of group means, there was no apparent effect of environment or strain-rate on the average fracture stress of either the virgin or abraded filaments, indicating the absence of slow crack growth.

However, significant effects were observed in the case of the sapphire filaments. Considering first the abraded filaments, the filaments tested in water showed an increase in average fracture stress with increasing strain rate. The filaments tested in toluene did not show a significantly different average fracture stress with a strain-rate increase of 200 times. Applying the Evans and Weiderhorn [12] treatment to the data from the water tests, in Fig. 1 the plot of the logarithms of the ordered fracture stresses yields a slope of approximately one. The value of the slope of the (K, V) curve in region I, calculated from the intercept in Fig. 1, is 28 which is in excellent agreement with the value of 26 determined from

*The filaments were supplied by the Manufacturing Technology Division of the Air Force Materials Laboratory, WPAFB, Ohio, USA.
†Avco, Space Systems Division, Lowell, Mass. USA.

TABLE I Average tensile fracture strengths and standard deviations of sapphire and silicon carbide filaments in the virgin and surface damaged conditions tested in toluene and water at room temperature

Filament and surface condition	Testing environment	Strain-rate (min^{-1})	Number of specimens	Average tensile fracture stress ($kg\ mm^{-2}$)	Standard deviation of fracture stress ($kg\ mm^{-2}$)
Silicon carbide					
virgin	Water	0.132	24	386	69
		0.026 4	21	435	39
		0.000 66	19	437	42
	Toluene	0.132	24	348	74
		0.026 4	19	391	60
		0.000 66	17	420	45
abraded	Water	0.132	24	197	43
		0.000 66	21	193	34
	Toluene	0.132	24	213	52
		0.000 66	20	203	47
Sapphire,					
virgin	Water	0.132	22	262	26
		0.000 66	20	231	28
	Toluene	0.132	20	267	26
		0.000 66	20	247	29
abraded	Water	0.132	26	174	14
		0.000 66	24	146	6.8
	Toluene	0.132	26	185	9.4
		0.000 66	23	178	17

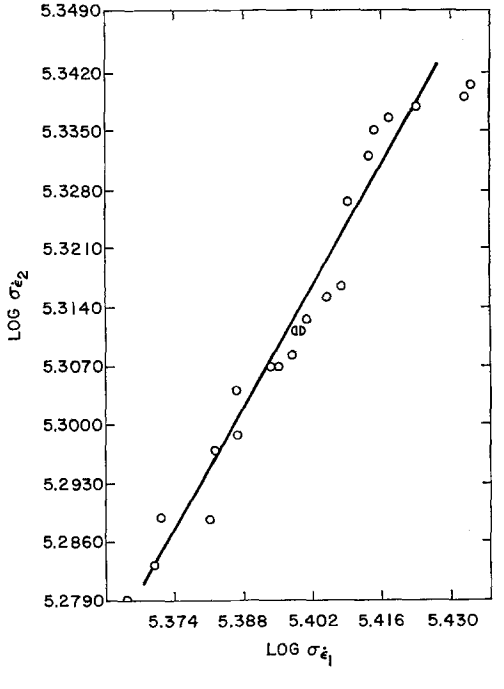

Figure 1 A logarithmic plot of the strengths σ_1 and σ_2 at equivalent probability for surface damaged *c*-axis sapphire filaments at two separate strain rates, $\dot{\epsilon}_1$ and ${}_2\dot{\epsilon}$ (where $\dot{\epsilon}_1/\dot{\epsilon}_2 = 200$).

slow crack growth studies [10]. Therefore, the abraded Al_2O_3 filaments show a strain-rate sensitive fracture stress when tested in a moist atmosphere due to moisture assisted surface flaw extension which is apparently the same phenomenon treated by Weiderhorn [10].

The virgin Al_2O_3 filaments also show strain-rate sensitive fracture stresses which are considerably higher than the abraded filament fracture stresses. The curves for the logarithms of the ordered stresses have slopes near one but the n values (slopes of the K, V curves) are 46 and 77 for filaments tested in water and toluene, respectively. These values do not agree with the value for moisture assisted surface flaw extension. Also, the fracture stresses for the tests in water and toluene are nearly the same indicating little or no effect of moisture on the observed slow crack growth.

The SEM examination (Figs.2 and 3) verified the previously reported result that the abraded filaments (fracture strengths less than $\simeq$ 200 kg mm^{-2}) fail from surface flaws while the virgin filaments fail from internal flaws [17].

For the virgin filaments one is then left with slow crack growth of internal flaws, similar to the effect observed by Pollock and Hurley [11]

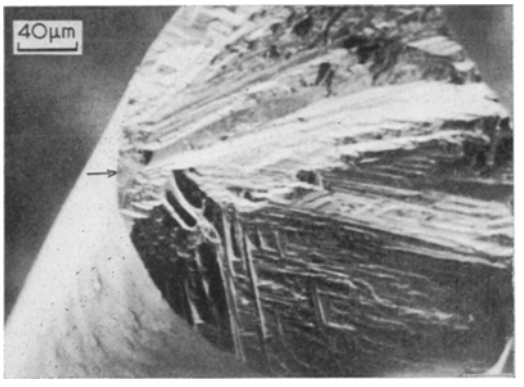

Figure 2 SEM micrograph of the fractured surface of a surface damaged *c*-axis sapphire filament showing fracture initiation at the surface (arrow indicates initiation site).

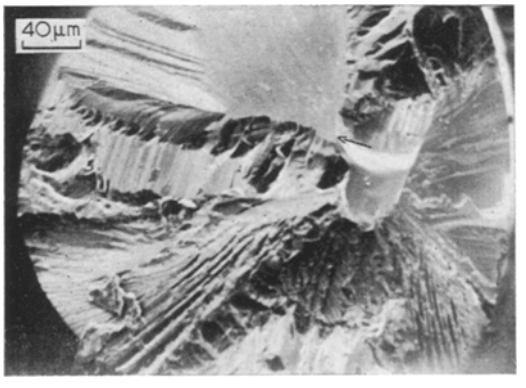

Figure 3 SEM micrograph of the fracture surface of a virgin *c*-axis sapphire filament showing internal initiation of fracture (arrow indicates origin of fracture).

who suggested that the slow crack growth is consistent with the Orowan model of subcritical crack extension by dislocation interactions with the crack tip.

The apparent lack of slow crack growth in the case of the abraded filaments tested in the dry toluene environment is probably related to the fact that these materials failed at much lower stresses, that is to say from larger flaws which do no extend as much as the smaller flaws [6].

4. Conclusions

By studying the strain-rate sensitivity of the tensile fracture stresses of pristine and surface damaged sapphire and silicon carbide-on-carbon filaments in water and toluene, the stress corrosion susceptibility of these materials was established. There was no evidence of slow crack growth in the silicon carbide-on-carbon under the conditions investigated in this study. The surface damaged sapphire exhibited slow crack growth in the water environment. From the slope of the stress intensity factor versus crack velocity curve this crack growth was shown to be due to stress-corrosion as characterized by Weiderhorn [10]. The corresponding experiments in toluene produced no evidence of slow crack growth. The pristine filaments exhibited slow crack growth in both water and toluene which could not be related to the moisture induced stress corrosion.

Acknowledgements

The authors gratefully acknowledge the financial support of the National Science Foundation (Grant number GK-37370) for this work.

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Received 23 September and accepted 7 October 1974.