TENSILE AND CYCLIC FATIGUE BEHAVIOR OF SiC WHISKER-REINFORCED Al_2O_3 AT ROOM AND ELEVATED TEMPERATURES

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ABSTRACT

Uniaxial tensile and cyclic fatigue data are reported for a commercial grade of silicon carbide whisker-reinforced alumina matrix composite (SiC_w/Al_2O_3) tested at room and elevated temperatures. The data show that addition of short SiC_w (30 vol%) in Al_2O_3 can significantly increase room temperature tensile strength and resistance to cyclic fatigue of monolithic Al_2O_3 by ~40% and ~100%, respectively, bringing the high-cycle fatigue strength ($>10^5$ cycle range) to ~95% of its tensile strength. These dramatic improvements in tensile and fatigue behavior were attributed to the presence of SiC_w which effectively inhibited cyclic fatigue crack growth. The composite further exhibited excellent retention of tensile and cyclic fatigue strengths at elevated temperatures as high as 1000°C for practical engineering applications.

INTRODUCTION

Studies (Becher et al., 1984; Wei et al., 1985; Tiegs et al., 1985) show that addition of short SiC whiskers in Al_2O_3 can significantly improve the strength and fracture toughness of composite materials for structural applications at high temperatures. This paper is intended to generate baseline data of a commercial composite material for component design and analysis use and to aid in understanding beneficial features, inherent weaknesses, and limitations that are associated with this composite.

Tensile strength of SiC_w/Al_2O_3 composite has been investigated to aid in material development using four-point bending (Tiegs et al., 1986). However, uniaxial tensile and cyclic fatigue properties of the composite are virtually unknown. Therefore, elevated temperature mechanical properties were investigated for the commercial SiC_w/Al_2O_3 ceramic composite tested under monotonic and tension-tension cyclic fatigue loading in uniaxial mode using solid rod specimens. Results are compared with those obtained previously for monolithic Al_2O_3 to examine the extent of the beneficial effects due to SiC_w reinforcement as well as limitations at elevated temperatures.

EXPERIMENTAL DETAILS

Material and Specimen

The composite material used in this experiment was a commercial grade of hot-pressed SiC_w -reinforced Al_2O_3 matrix composite containing nominally 30 Vol% of short SiC whiskers (~1- μ m diam $\times$ ~25- μ m length) dispersed throughout the matrix. The average diameter of Al_2O_3 matrix grains was ~3 μ m. Several square tiles (153 mm $\times$ 153 mm $\times$ 19 mm), commercially designated as PAD-AS34W, were procured from CERCOM of Vista, California.

A detailed NDE inspection by ultrasonic scanning was performed on each tile before specimen fabrication. Figure 1a shows an image of an ultrasonic scan on a focal plane at a depth of about 2.5 mm from the surface of tile "A" (Lot No. 4-318-6, 1989 vintage). Large dark spots discernable in Fig. 1a indicate voids or imperfections which were suspected to be either SiC_w clumps or agglomerates of matrix grains. Note that images of the large dark spots were exaggerated by the technique with the actual sizes being much smaller. Eight buttonhead specimens (Fig. 2) were machined from each tile, and each specimen was identified with a tile designation letter preceding a serial

Fig. 1 - (a) Result of ultrasonic scanning on a focal plane 2.5 mm below the surface of tile A and (b) that of tile B.

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Fig. 2 - (a) Buttonhead tensile specimen and (b) specimen geometry.

number. Some of specimens made from tile A revealed small, shallow, craters on the specimen surface under a light microscope. The specimens were tested in the as-machined condition with no surface polishing. Interestingly, none of the specimens initiated fracture from the surface defects.

Additional tiles were procured subsequently for fatigue testing, being identified as tiles B, C, D, and E. These tiles were obtained from another lot (Lot No. 4-442-X, 1990 vintage), showing more uniformly dispersed whisker mixing compared to tile A. Results of ultrasonic scanning inspection showed these tiles were virtually devoid of large defects, as shown in Fig. 1b for tile B. Examination showed all specimens fabricated from the second lot were virtually unblemished except machining marks.

Testing Equipment and Method

All testing was performed using a closed-loop controlled electrohydraulic testing machine equipped with a set of hydraulically operated self-aligning couplers integrated in the load-train assembly shown in Fig. 3. The self-aligning couplers were essential components in the load train assembly to minimize specimen bending in the reduced gage section. Because of space limitation, details of the mechanisms and operation of the couplers are referred to elsewhere (Liu and Brinkman, 1986).

Testing was conducted at ambient and elevated temperatures to 1200°C. Specimens were heated by a compact induction heater. Tensile strain was directly measured from the uniform gage section using strain gages for room temperature testing only or an in-house developed mechanical strain extensometer (Liu and Ding, 1993). A view from the transducer end of the extensometer installed at the test

Fig. 3 - Load-train assembly for tensile fast fracture and cyclic fatigue testing. Elevated temperature testing is accomplished using induction heating.

position is shown in Fig. 3. The heating element in the near side of the heating coil is removed so that the other SiC susceptor placed immediately behind the specimen is visible. The output signals from the load cell and strain transducers were plotted on an XY-plotter to establish the relationship between stress and strain. The values of elastic moduli were then determined directly from the stress-strain curves.

All testing was performed either in monotonic tension for fast fracture or in cyclic tension-tension for fatigue. Monotonic tensile testing was run at a stressing rate of 2100 MPa/min and cyclic fatigue testing was run at a much higher rate of 21,000 MPa/min so that a fatigue test could be completed in a reasonable period of time. A triangular wave form as shown in Fig. 4a was used in cyclic fatigue tests. If the test specimen showed no imminent failure after completing a block of 30,000 to 100,000 cycles at a given constant peak stress, the peak stress was increased intermittently in steps of about 30 MPa until failure occurred, as shown in Fig. 4b.

TEST RESULTS AND DISCUSSION

Six specimens in "A"-series were tested in pure tension at room temperature. Three specimens (A1 to A3) were instrumented with strain gages. Remaining three (A4 to A6) were tested with a mechanical strain extensometer for the purpose of cross calibration. The average value of the elastic modulus at room temperature determined from the three strain gage data was 398.7 GPa and that

- (a) Wave Form I:
Constant Amplitude Loading
 $R = \text{min./max. stress} = 0.1$
- (b) Wave Form II:
Increasing Multiple-Step Loading

Fig. 4 - Waveforms used in tension-tension cyclic fatigue testing.

from extensometer data was 413 GPa, showing a respectable agreement with a small discrepancy of 3.5%. A value of 393 GPa has been reported for the composite of similar composition (Han and Suresh, 1989). The average tensile strength was 432 MPa, which was higher than that of monolithic alumina by about 40% (Liu and Brinkman, 1987). The tensile strength was reproducible within a scatter of $\pm 5.5\%$. Although a reliable value of the Weibull modulus can not be determined based on six tests, the test data implied that the Weibull modulus would be high for this material.

An exploratory cyclic fatigue test was then performed using specimen A7. Since little was known about the fatigue behavior of this composite material, a cyclic stress amplitude (CSA) of 330 MPa, equivalent to about 75% of the tensile fracture strength, was used as a starting point. Each time after a block of 30,000 to 100,000 cycles was completed, the CSA was increased intermittently in steps until fatigue failure occurred. Specimen A7 failed after completing 617 cycles at the ultimate CSA of 414 MPa, which was about 96% of the tensile fracture strength. To validate the high fatigue performance exhibited by the pilot specimen, additional tests were conducted on two specimens of a different origin, tile D. The number of cycles to failure at each ultimate CSA are 9,964 cycles at 446 MPa and 13,456 cycles at 432 for specimens D1 and D2, respectively (Table 1). Because the ultimate CSA attained in the fatigue tests are within the scatter of the tensile strength, fatigue damage due to intermediate cycling below the ultimate CSA is assumed to be of insignificance. Therefore, the data (filled squares) are plotted in Fig. 5 as if no precycling were done to the specimens. The three unfilled square

Fig. 5 - Comparison of fatigue behavior of monolithic Al_2O_3 and SiC_w reinforced Al_2O_3 matrix composite at room temperature and 1000°C .

symbols indicate the test conditions before the three specimens were cycled to failure at the highest CSA level attained. The arrow attached to the unfilled symbols indicates that the fatigue life would be longer if the test was continued. To facilitate comparison, the fatigue data of monolithic alumina reported previously by our laboratory (Liu and Brinkman, 1987) are included in Fig. 5 also.

The linear lines representing the average fatigue behavior were determined by the least squares method. Due to the limited information, the fatigue behavior of the composite beyond 10^5 cycles must be considered to be tentative. Figure 5 shows that the fatigue curve of the composite was practically flat compared to that of monolithic alumina. The difference in fatigue behavior was obviously attributed to the strengthening by SiC whiskers that have effectively inhibited the slow crack growth being promoted by cyclic fatigue. The flatness of the fatigue curve implied that little or no fatigue induced crack growth was occurring in the specimen until the applied cyclic stress exceeded a critical value which must be high enough to fracture the SiC whiskers. In the case of this composite material, the critical level of cyclic stress to initiate a crack growth may exceed as high as 90% of its tensile fracture strength.

Careful examination of Fig. 5 indicated that data points that represented the fatigue resistance of tile D generally fell on the upper side of the fatigue curve, whereas those made from tile A fell on the lower side, probably due to material variation. A plausible explanation can be made when comparing the two ultrasonic scanning images shown in Fig. 1. Large process defects detected in Fig. 1a were clearly detrimental to the fatigue resistance of specimen A7.

Results of fatigue tests at 1000, 1100, and 1200°C are summarized in Table 1 and data are plotted in Fig. 6. Again, an exploratory test at 1000°C was performed initially for specimen B1 which was cycled to a CSA of 250 MPa as a starting point. The CSA was raised to 275 MPa after completing $\sim 10^6$ cycles at 250 MPa. The specimen failed after completing an additional 194,000 cycles, bringing to a total of 1.256×10^6 cycles to failure. To determine the fatigue behavior at 1000°C , several specimens were tested at various levels of peak stresses above 250 MPa. Because of the data scatter,

Fig. 6 - Cyclic fatigue behavior of SiC_w -reinforced Al_2O_3 matrix composite materials tested at elevated temperatures. The strengthening effects due to SiC_w reinforcement degenerated entirely at 1200°C when compared with the fatigue data of monolithic Al_2O_3 (Lin, 1991).

the fatigue behavior is represented by a narrow scatter band to embrace all data points. The composite material at 1000°C retained about 2/3 of the room temperature fatigue strength. Unfortunately, no fatigue data for monolithic alumina at 1000°C are available for comparison at this time. However, the beneficial effect of whisker reinforcement is retained and discernible when the 1000°C fatigue curve are compared with the room temperature fatigue curve of the monolithic alumina shown in Fig. 5.

Cyclic fatigue tests at 1100 and 1200°C were then performed, and results are also given in Table 1 and Fig. 6. The 1100°C fatigue data are not as consistent as the 1000°C fatigue data. The reason for the low fatigue performance exhibited by specimen C6 is unknown. Therefore, this data point was tentatively excluded from the current data analysis. Because of data scatter, the 1100°C fatigue behavior is also bracketed by a narrow scatter band of one standard deviation, as illustrated in Fig. 6.

Fig. 7 - Average values of elastic moduli determined from the tensile and cyclic stress-strain curves.

Fatigue data for both monolithic (Lin, 1991) and composite materials tested at 1200°C are compared in Fig. 6. The four data points for the composite material correlate well linearly along the line determined by the least squares fit. Using the linear relationship as a guide, all data points can be bracketed in a scatter band as shown in Fig. 6, suggesting that 1200°C fatigue behavior of monolithic Al_2O_3 and that of $\text{SiC}_w/\text{Al}_2\text{O}_3$ are about the same. The beneficial effects of SiC whiskers reinforcement apparently degenerated entirely at 1200°C .

The fatigue behavior of the composite may be characterized by the following simple power-law relationship:

$$\sigma = \alpha N_f^\beta \quad (1)$$

where σ is the cyclic stress amplitude in MPa, N_f the number of cycles to failure, α , and β are functions of temperature as

$$\alpha = 429.83, \quad \beta = -0.0017 \quad \text{for } T = 20^\circ\text{C}; \quad (2)$$

$$\log \alpha = 0.55325(\pm 0.18312) + 4.62(\pm 0.2345) \frac{T}{10000} - 2.5784 \left(\frac{T}{1000} \right)^2, \quad (3)$$

$$\beta = 0.88757 - 2.0164 \left(\frac{T}{1000} \right) + 1.1498 \left(\frac{T}{1000} \right)^2 \quad (4)$$

for $900^\circ\text{C} < T \leq 1200^\circ\text{C}$.

Fatigue behavior beyond 10^6 cycles must be regarded as tentative due to the lack of data $> 10^6$ cycles.

Although not shown here, discernible plastic strain ratchetting, i.e., accumulation of small plastic strain (small permanent elongation) occurring in each loading cycle, was observed in cyclic stress-strain curves for all tests of the composite material at temperatures above 1100°C . It is speculated that the enhanced fatigue resistance by SiC whiskers may have been degenerated or negated by the strain ratchetting at high temperatures. Information concerning the strain ratchetting behavior for monolithic Al_2O_3 at 1200°C is currently not available for comparison and further discussion.

The average values of elastic modulus at elevated temperatures were determined from the stress-strain curves and summarized in Table 1 and Fig. 7. The error bar indicates one standard deviation. A dramatic decrease in the value of elastic modulus occurred as temperature increased from 1100 to 1200°C , and this behavior might be correlated to the ratchetting behavior discussed in the preceding section.

FAILURE ANALYSIS

Results of optical (OM) and scanning electron microscopy (SEM) examinations are illustrated in Figs. 8 and 9, respectively, for specimen A1 tested in tensile fast fracture and specimen A7 in cyclic fatigue at room temperature. Both specimens fractured from internal defects indicated by a black arrow as shown in Figs. 8a and 9a. High magnification photos revealed that specimen A1 fractured from a huge single grain of $\sim 70\text{ }\mu\text{m}$ (Fig. 8b) and specimen A7 from an agglomerate of large grains (Fig. 9b). X-ray analyses indicated these grains were all pure Al_2O_3 . Absence of SiC whiskers inside the Al_2O_3 agglomerates suggested that poor SiC whisker dispersion in the region apparently failed to inhibit the overgrowth of Al_2O_3 grains. Inherent low fracture toughness of monolithic Al_2O_3 apparently facilitated fracture initiation. The macroscopic features of fracture surfaces for specimen A1 tested in tensile fast fracture and specimen A7 tested in cyclic fatigue are virtually the same. This observation strongly supports the earlier theory that SiC whiskers can effectively inhibit slow crack growth. Few whisker pullouts were observed, indicating excellent bonding between the whisker and matrix.

Typical fracture surfaces of three specimens tested at elevated temperatures are shown in Fig. 10. Excluding a specimen that initiated fracture from a large internal cavity, all other specimens tested at high temperatures initiated fracture from the specimen surface or subsurface. The reason might be that all of these specimens were made from the second batch of tiles which showed uniform dispersion of SiC_w , according to the earlier ultrasonic examination. In the absence of large defects in the bulk, any defects located near the specimen surface are conducive to fracture initiation according to fracture mechanics. While a large agglomerate devoid of SiC_w (circled areas shown in Figs. 10a and 10c) was an easy fracture initiation site, segregated SiC_w clumps (indicated by arrows shown in Fig. 10b) devoid of Al_2O_3 were equally deleterious. Crack initiation was particularly sensitive to the situation where many SiC_w clumps lying in the plane transverse to the tensile direction, as shown in Fig. 10b. While little or no flat mirror regions were discerned on the fracture surfaces of specimens tested at room temperature, well defined flat mirror regions resulting from slow crack growth were visible on the fracture surfaces of the specimens tested at high

Fig. 8 - (a) Fracture surface of specimen A-1 tested in uniaxial tension. The arrow indicates the fracture initiation site. (b) An over-sized Al_2O_3 grain at the fracture origin.

Fig. 9 - (a) Fracture surface of specimen A-7 tested in uniaxial cyclic fatigue, and (b) an agglomerate of Al_2O_3 grains at the fracture origin.

temperatures. The area of the mirror region grew larger as temperature increased, indicating progressive weakening of SiC_w reinforcement and the ultimate loss of the beneficial effect at 1200°C . Fracture was dominantly transgranular with little or no whisker pullouts for specimens tested at or below 1100°C . However, a mix mode of transgranular fracture in Al_2O_3 rich area and interfacial fractures in SiC_w was more visible at 1200°C , indicating the degradation of whisker reinforcement. To take the full advantage of the SiC reinforcement with proven consistency in fatigue behavior, the composite can be used in structural applications at temperatures as high as 1000°C .

CONCLUSION

On the basis of the limited test data, the following conclusions are made.

1. Significant increases in both tensile and fatigue strengths by about 50% and 100%, respectively, were observed in SiC whisker-reinforced Al_2O_3 matrix composite compared to monolithic Al_2O_3 .
2. The enhancement of resistance to cyclic fatigue in the composite was attributed to the presence of SiC whiskers which had effectively inhibited crack growth promoted by cyclic fatigue.
3. The composite material at 1000°C still retained good tensile as well as fatigue strengths higher than the room-temperature strengths of monolithic alumina. However, the strengthening due to the SiC whiskers reinforcement degenerated as temperature increased to 1200°C .
4. The fatigue behavior of the composite material at elevated temperatures can be correlated by a simple power-law relationship between the cyclic stress amplitude and number of cycles to failure.
5. Absence of whiskers inside the Al_2O_3 agglomerates suggested that poor whisker dispersion in the region was responsible for the failure to inhibit the overgrowth of large single crystals and the formation of agglomerates.

Fig. 10 - Typical fracture surfaces of cyclic fatigue specimens: (a) B1 at 1000°C showing the site of fracture origin devoid of SiC_w ; (b) C1 at 1100°C showing many segregated SiC_w nearly devoid of Al_2O_3 ; (c) D7 at 1200°C showing dominantly transgranular fracture in Al_2O_3 rich area and interfacial fracture in SiC_w that lie in the plane transverse to the loading direction.

6. Large internal defects such as an isolated overgrown Al_2O_3 single grain, an agglomerate of Al_2O_3 grains, and SiC_w clumps were easy fracture initiation sites.
7. To take the full advantage of the SiC reinforcement with proven consistency in fatigue behavior, this composite can be used in structural applications at elevated temperatures but limited to 1000°C.

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Table 1 - Results of tensile and cyclic fatigue tests on SiC_w/Al₂O₃ at room and elevated temperatures.

Specimen No.	20°C [E=406±18 GPa]	
	σ(MPa)	N _f
A1-6	432*	1
A7	414	617
D1	446	9,964
D2	432	13,456

Specimen No.	1000°C [E=292±15 GPa]	
	σ(MPa)	N _f
B1	275	193,906
B2	275	529,161
B3	285	191,265
B4	300	106,983
B5	310	58,159
B6	325	29
B7	315	266,883
B8	320	220,993
E1	325	645
E2	323	28,383
C8	421	1

Specimen No.	1100°C [E=282±25 GPa]	
	σ(MPa)	N _f
A8	255	20,815
C1	250	1,360
C2	225	223
C3	200	2,156
C5	180	5,050
C6	160	966
C7	379	1

Specimen No.	1200°C [E=203±16 GPa]	
	σ(MPa)	N _f
D4	160	27
D5	138	107
D6	104	638
D7	70	22,248

Note a: The value represent the average of six tensile tests on specimens A1 to A6.