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Fatigue and stress rupture of silicon carbide fibre-reinforced glass–ceramics

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Fatigue and stress-rupture testing of unidirectional Nicalon-type silicon carbide fibre-reinforced lithium aluminosilicate glass–ceramic matrix composites is described. Tensile fatigue testing was performed at 22° C on two different composite systems to contrast the behaviour under applied stresses above and below the levels necessary to cause matrix cracking. The higher strength of the two composites was then also tested in flexural fatigue and constant-load stress rupture at 22, 600 and 900° C in air. It is shown that the level of tensile stress at which composite inelastic stress–strain behaviour begins is an important factor in the control of overall composite performance, and that properties at elevated temperature are significantly different to those at room temperature.

1. Introduction

In previous papers it has been shown that Nicalon silicon carbide-type yarn can be used very effectively to reinforce glass and glass–ceramics to achieve tough, strong composites [1–3]. Recent publications on the nature of the chemistry of the fibre–matrix interface [4] and the stress–strain behaviour of these composites [5–7] have also shed more light on the nature of their strength-controlling mechanisms, and on the observation that degradation of composite performance can take place at elevated temperature through the presence of oxygen in the test environment [8]. The purpose of the present paper is to describe further composite behaviour resulting from fatigue and stress-rupture testing at room and elevated temperature. Two different SiC fibre-reinforced lithium aluminosilicate (LAS) glass–ceramic composites were chosen for this investigation because of their markedly contrasting stress–strain behaviour. One, SiC-reinforced LAS-I, exhibits a linear tensile stress–strain curve to failure, while the other, SiC-reinforced LAS-II, exhibits very non-linear behaviour as a result of extensive matrix cracking prior to ultimate fracture. The LAS-II matrix composite ultimate tensile strength of 520 MPa is also twice that of the LAS-I composite specimens evaluated in this study. Despite these differences, both of these composites are extremely tough and at room temperature are characterized by fibrous fracture modes.

2. Experimental procedure

2.1. Composite fabrication

Composite specimens were fabricated using the hot-press diffusion bonding techniques described previously [6]. Two LAS glass–ceramic matrices, designated LAS-I and LAS-II, were used and the reinforcing fibre was of the silicon carbide type referred to as Nicalon (Nippon Carbon Co., Yokohama, Japan). The resultant composites contained 45 to 50 vol % of these fibres.

2.2. Mechanical testing

Both uniaxial tension and four-point bend flexural tests were performed at room temperature. Only flexure tests were performed at the elevated temperature and these were on specimens located in a tube furnace with a static air environment. For the tensile tests, strains were measured using glued-on strain gauges and extensometers while for all bend tests, composite mid-span deflection measurements were made. The four-point flexture tests were performed within 0.2 cm thick specimens having major and minor spans of 6.25 and 1.9 cm, respectively.

3. Experimental results and discussion

In a previous paper [6] it was shown that the tensile stress–strain curve for silicon carbide uniaxially reinforced LAS composites depended on matrix composition and the *in situ* fibre strength. For LAS-I matrix composites the tensile stress–strain curve was found to be linear right up to the point of fracture. In contrast, for the stronger LAS-II matrix system, the fibres were significantly stronger, resulting in a composite which survived to strains of up to 1% and exhibited a non-linear stress–strain curve shape. The non-linear region can be attributed to the presence of matrix cracking [5–7] and thus is a region of material behaviour where the interior of the composite and the fibre–matrix interface are exposed to the surrounding environment while under stress.

The fatigue of both of these two distinctly different composites was performed at 22° C in tension, while only the LAS-II system was tested further at high temperatures.

3.1. Tension–tension fatigue of 0° SiC-reinforced LAS-I matrix composites

Unidirectionally reinforced LAS-I matrix composites were tested only in tension–tension fatigue. Stress was applied in the 0° direction in a sinusoidal manner at a rate of 10 Hz with a minimum tensile stress of

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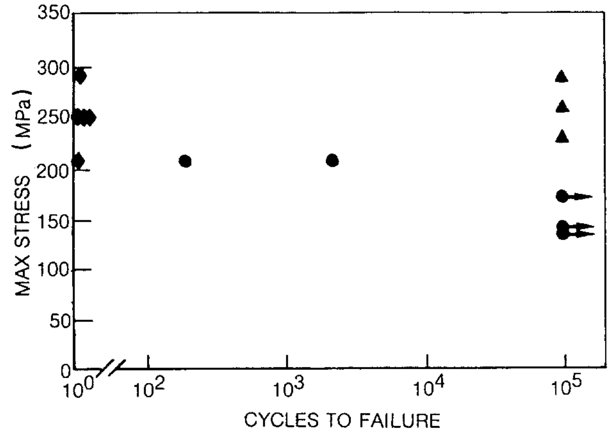

Figure 1 22° C tension–tension fatigue of 0° SiC-reinforced LAS-I: (◆) tensile test fracture, (●) fatigue fracture, (●→) fatigue run-out, (▲) tensile fracture after fatigue. Minimum stress = 20.7 MPa in all cases.

20.7 MPa maintained for all tests. Tests were interrupted at 10^3, 10^4 and 10^5 cycles to measure the axial composite elastic modulus. Composite failure was taken as the point at which specimens fractured into two pieces. Also, any specimens that survived 10^5 cycles without fracture were then tensile tested to failure. The fatigue data are summarized in Figs 1 and 2.

The average tensile strength for these composites was only 261 MPa, which is considerably lower than the 445 MPa reported previously [6]. It was not determined why these lower strengths were obtained; however, stress–strain behaviour was linear to failure and composite fracture appearance was quite fibrous, typical of LAS-I matrix composites.

The fatigue stress against cycles to failure plot (Fig. 1) shows that with a maximum applied stress of 207 MPa it was possible to cause composite failure at 190 and 2160 cycles. At lower maximum applied stresses of 172 and 138 MPa, however, the specimens survived for the 10^5 cycle life and their residual tensile strengths, also indicated in the figure, were approximately equivalent to those of unfatigued tensile specimens.

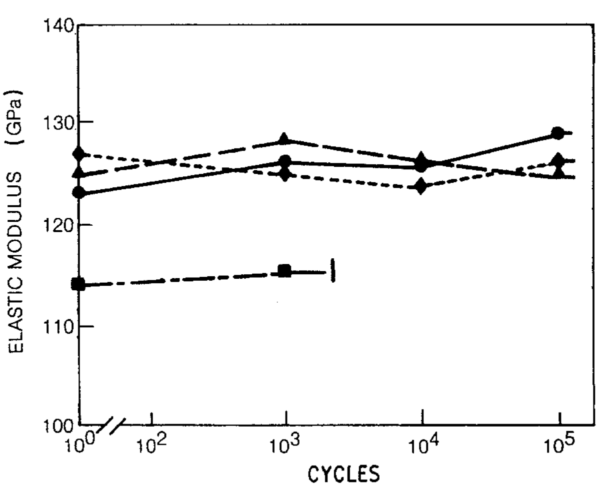

Figure 2 Retention of elastic modulus in tension–tension fatigue of 0° SiC-reinforced LAS-I. Maximum stress (MPa): (●, ▲) 138; (◆) 172; (■) 207.

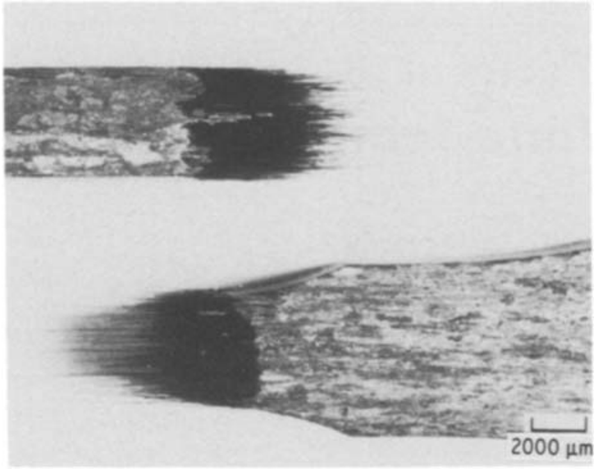

Figure 3 Optical photograph of 0° SiC-reinforced LAS-I specimen fractured at 190 cycles under 205 MPa maximum applied stress.

The measurement of composite elastic modulus at intermediate numbers of cycles was used to determine whether any composite structural deterioration had occurred with increasing numbers of cycles. The resultant data (Fig. 2) indicate that the measured elastic modulus was essentially unchanged for all of the specimens. Even the specimen that failed at 2160 cycles exhibited full retention of its modulus at the 1000 cycle test point. It should be noted that all of these specimens were of the type that exhibited perfectly linear tensile stress–strain curves. Thus, the strains achieved at the maximum applied fatigue stresses of 207 MPa were below those observed to cause a change in composite elastic modulus during monotonic tensile loading. Apparently fatigue loading did not alter this situation.

Examination of the fractured fatigue specimens revealed a fibrous mode of failure similar to that of the monotonic tensile specimens (Fig. 3). Fractures occurred in the gauge section with pulled-out fibres having an average length of approximately 0.38 cm but with individual fibres approaching 1.0 cm. This was also true of those specimens which survived the 10^5 cycle loading and then were tensile tested to failure.

3.2. Tension–tension fatigue of 0° SiC-reinforced LAS-II composites

These tensile fatigue tests were performed at a rate of 5 Hz and with a minimum applied stress for each cycle of less than 20 MPa. Fatigue minimum stresses were set at 10% of the maximum applied stress and, as above, tests were halted after 10^5 cycles or when composite fracture occurred.

The typical tensile stress–strain curve obtained for this composite is shown in Fig. 4. As described previously [6], composite strength is considerably greater than that for the LAS-I matrix composite system and the non-linear curve associated with matrix microcracking is obtained. The stress level at which the first deviation from linear elastic behaviour occurs is at approximately 270 MPa. To provide a consistent method to measure this proportional limit stress, a convention was chosen wherein a 0.02% offset in

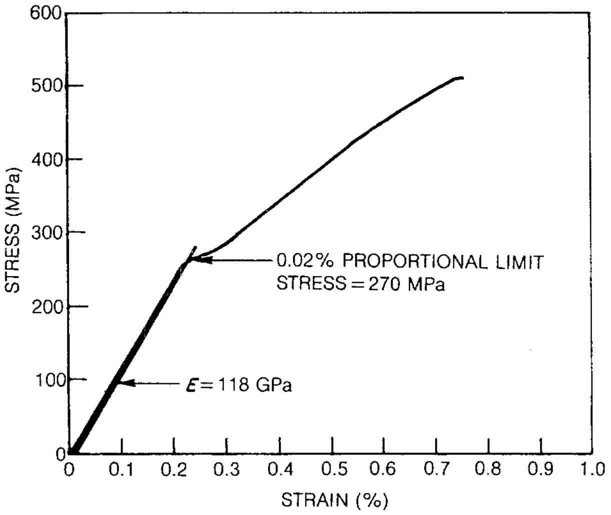

Figure 4 Tensile stress–strain curve for 0° SiC-reinforced LAS-II.

strain parallel to the elastic modulus was taken, and its intersection with the material stress–strain curve is chosen as a measure of the stress at which inelastic behaviour begins. In Fig. 4 this stress is taken to be at approximately 270 MPa. Fatigue tests at up to 10^5 cycles were performed to examine the effects on composite elastic modulus. None of the composites fractured within the range of cycles tested and composite residual strengths are nearly equivalent to the initial unfatigued composite strength (Table I). For those specimens tested at a maximum fatigue stress below the proportional limit, no change in composite elastic modulus was observed with fatigue cycles. The stress–strain cycles for various stages of test for one such specimen are shown in Fig. 5. The stress–strain behaviour was linear for each cycle and the final tensile test of the sample after 10^5 cycles also revealed no substantial change in proportional limit stress. Fatigue testing at a stress slightly above the proportional limit also did not cause failure prior to 10^5 cycles (Table I); however, it did cause a substantial change in stress–strain loop shape (Fig. 6). In this case, on each unloading and reloading cycle there are two distinctly different regions of behaviour. Above a stress of approximately 120 MPa the slope of the curves is well below that of the initial elastic modulus of the composite. This is in agreement with findings in the previous publication [6]. However, below this stress the curve appears to have the same elastic stiffness as the original composite. This behaviour was not noted in the previous paper because at that time the minimum cyclic stress was not taken low enough to see the effect, and it may relate to some closing back together of the matrix microcracks on unloading.

TABLE I Tension–tension fatigue of 0° SiC-reinforced LAS-II

Specimen	Fatigue stress applied (MPa)	Number of fatigue cycles	Residual tensile strength (MPa)
1	None	None	520
2	None	None	580
3	225	10^5	620
4	275	10^5	485
5	310	10^5	525
6	355	10^5	485

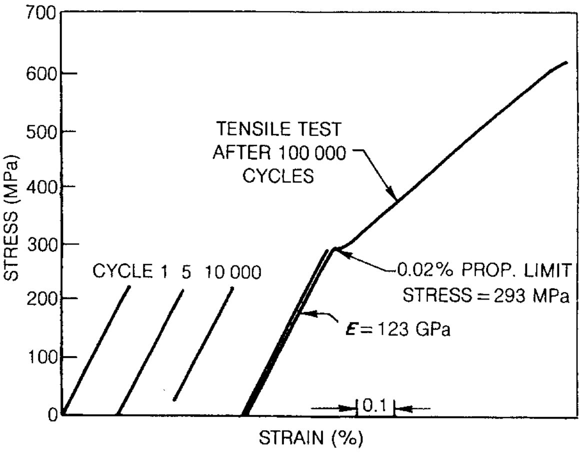

Figure 5 Tensile fatigue and residual tensile stress–strain curves for 0° SiC-reinforced LAS-II.

Thus, as a result of high stress tensile fatigue the proportional limit stress is found to decrease, in this case to approximately 40% of the initial value.

3.3. Flexural fatigue of 0° SiC-reinforced LAS-II matrix composite

The LAS-II matrix composite system was selected for further fatigue testing in air at the temperatures of 22, 600 and 900° C. These tests were performed in four-point flexure at a rate of 0.5 Hz and were terminated either after 10^5 cycles, which corresponded to approximately 55 h of testing, or after specimen failure. In all cases, however, specimen failure was not by fracture into two pieces. Instead, due to the high toughness of these composites, cracks propagated only partially through the specimens, causing a significant increase in mid-span deflection.

The data obtained from the 22° C tests are shown in Fig. 7. Three specimens were monotonically tested first to obtain the beginning level of strength and load–deflection behaviour. Their flexural strengths ranged from 710 to 813 MPa and all failed on their compression sides, consistent with previous results [6]. As in the tensile test regime described above, the applied load against mid-span deflection curves for these composites were very non-linear, and the stress at which these curves first deviated from linearity is

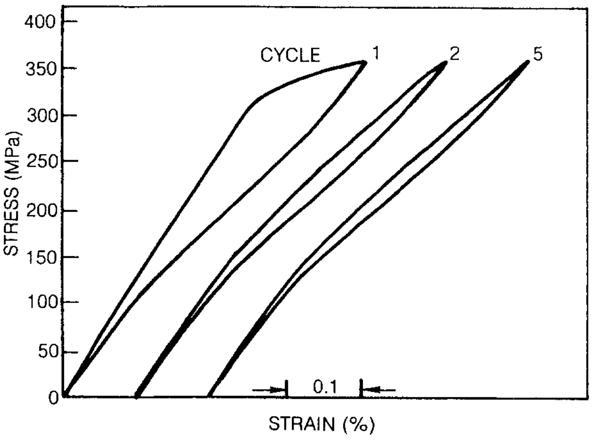

Figure 6 Tensile fatigue stress–strain curves for 0° SiC-reinforced LAS-II at room temperature.

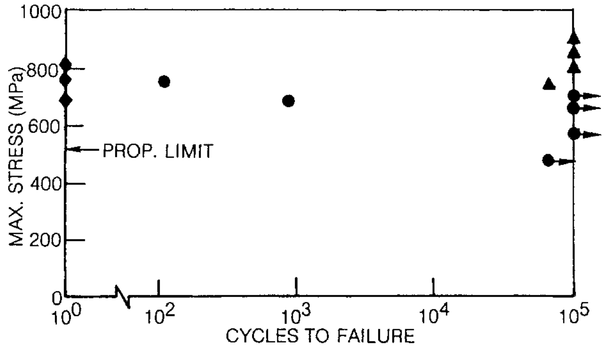

Figure 7 22° C flexural fatigue of 0° SiC-reinforced LAS-II: (◆) bend test fracture, (●) fatigue fracture, (●→) fatigue run-out, (▲) bend test after fatigue.

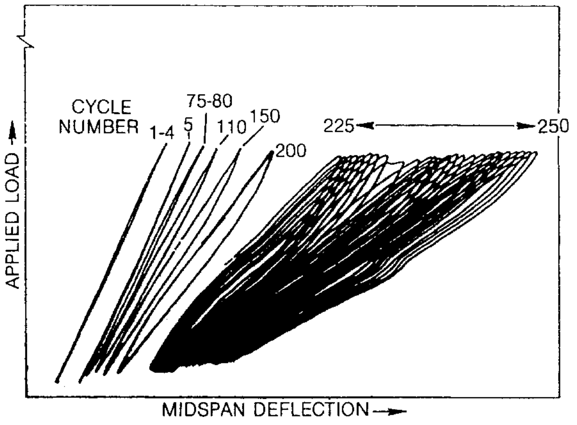

Figure 9 Load against mid-span deflection at 600° C in air during fatigue at applied maximum stress of 374 MPa.

analogously referred to as the proportional limit. This stress, as well as all other stresses referred to in this section, were calculated using the linear elastic beam formula. It is clear from the extensive non-linear behaviour of these composites that such calculations at loads above the proportional limit are not strictly valid. They are used, however, as a consistent method for describing the loading conditions and composite performance.

The average proportional limit stress for the three monotonically tested specimens is also indicated in Fig. 7, and it can be seen that all but one of the fatigue tests was run at stress levels above the proportional limit. For those composites that survived 10^5 fatigue cycles, residual strengths were found to be equal to or even greater than those of the unfatigued specimens. Also, in all cases, specimen failure initiated on the compression side.

During these fatigue tests composite effective elastic modulii were calculated for each specimen (using the initial linear portions of the curves) at 1, 5, 10^3, 10^4 and 10^5 cycles. In each of the cases of specimen run-out to 10^5 cycles no change in effective elastic modulus was noted, indicating no structural deterioration of the specimens during fatigue.

Fatigue test data obtained at 600° C are presented in Fig. 8. Three specimens tested in a single loading cycle to failure at 600° C exhibited flexural strengths between 1000 and 1100 MPa. The average proportional limit, measured at the 600° C test temperature, is also indicated in the figure. The fractures of these monotonically loaded specimens were extremely fibrous and indicated a high level of toughness. However, in contrast to room-temperature, the stress level that permitted specimen run-out to 10^5 cycles was only about one-third of the initial flexural specimen strength. Most of the specimens fractured at intermediate numbers of cycles, and in one fortuitous case it was possible to monitor the failure process by continuously recording the applied load against mid-span deflection traces (Fig. 9). The relative stiffness of the composite specimen remained unchanged for approximately the first 80 cycles. From the 110th cycle on, however, a gradual decrease in stiffness can be noted. Specimen failure was taken to occur at the 200th cycle in that the specimen indicated a marked decrease in stiffness and a visible interlaminar shear crack accompanied by two major tensile cracks on the tensile surface. It is interesting to examine the progression of the sample beyond this initial point of failure. The load–deflection traces between cycles 225 and 250 (Fig. 9) indicate that the specimen gradually continues to deteriorate; however, it is still able to support the maximum applied stress. This gradual failure process is an excellent illustration of why a fibre-reinforced ceramic would be preferred to its unreinforced counterpart. It also points to the difficulty of selecting a failure criterion. In all of the flexural fatigue tests performed in this investigation, the specimens did not fracture into two pieces but instead indicated a gradual process of deterioration. Thus, the selection of a failure criterion in this test is somewhat subjective. The criterion chosen was based on visible damage

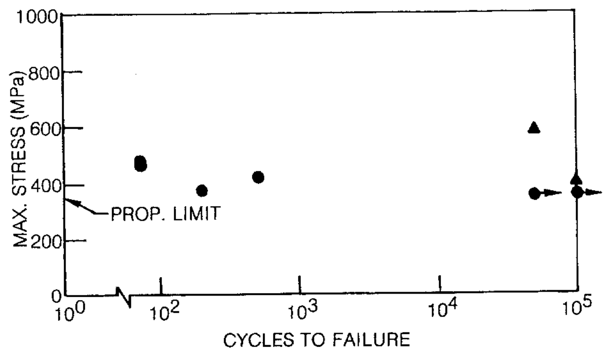

Figure 8 600° C flexural fatigue of 0° SiC-reinforced LAS-II: (◆) bend test fracture, (●) fatigue fracture, (●→) fatigue run-out, (▲) bend test after fatigue.

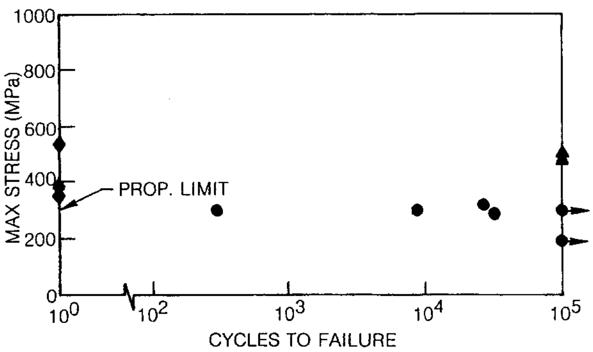

Figure 10 900° C flexural fatigue of 0° SiC-reinforced LAS-II: (◆) bend test fracture, (●) fatigue fracture, (●→) fatigue run-out, (▲) bend test after fatigue.

TABLE II Four-point flexural stress-rupture testing of 0° SiC-reinforced LAS-II

Temperature (°C)	Proportional limit (MPa)	Applied stress (MPa)	Time at stress (h)	Residual strength (MPa)
22	–	–	0	896
	565	–	0	834
	330	–	0	680
600	365	–	0	1006
	367	–	0	1008
	–	366	52	406
	–	368	42*	–
	–	391	50	448
	–	413	50	468
	365	443	43*	–
	–	603	0.4*	–
900	330	–	0	505
	–	362	65	517
	–	364	66	620
	328	408	0.4*	–
	–	413	0.1*	–
	344	450	0.4*	–

*Indicates specimen fractured at time indicated.

and a notable, but not specific, decrease in stiffness. In more design-oriented property determinations it would be expected that a specific criterion, such as a 10% decrease in stiffness, could be chosen. The tensile fatigue tests performed did not share this ambiguity in that ultimate specimen fracture was the selected criterion.

The fatigue data for the 900° C tests are shown in Fig. 10. As for 600° C, a majority of tests were discontinued prior to 10^5 cycle run-out. At this temperature, however, the fatigue stress levels were 50% or greater than the unfatigued specimen bend strength, and the residual strengths of the surviving specimens coincided with this original undamaged strength. The applied load against mid-span deflection curves for these residual strength measurements were very nonlinear, and specimen fracture occurred gradually and non-catastrophically. The resultant fracture surfaces from these tests were non-fibrous in nature and consistent with the previously described flexural test fractures at this temperature [6].

As in the 22° C test case, at both 600 and 900° C the specimens which ran out to 10^5 cycles exhibited no decrease in elastic stiffness.

3.4. Flexural stress-rupture testing of 0° SiC-reinforced LAS-II matrix composite

Four-point bend tests were performed in air at 600 and 900° C using a constant applied load to determine the time to cause specimen fracture as a function of applied stress and temperature. All specimens were unidirectionally reinforced and tests were terminated either by composite fracture or after approximately 50 to 65 h under stress. This limit of test time was chosen to correspond approximately to the total time required by specimens to run out to 10^5 cycles in the flexural fatigue tests described above.

The data obtained in these tests are summarized in Table II. The data include specimens at each temperature which were tested to obtain their ultimate strengths and also those subjected to a constant stress for long times. Also recorded are the proportional limit stress levels obtained from several of the initial loading curves and the residual flexural strengths (at temperature) of specimens which survived the 50 to 65 h test.

These data can be compared with the previously described fatigue results using the bar charts shown in Fig. 11. First, the comparison of composite room-temperature properties shows that 10^5 cycle run-out can occur at stresses above the proportional limit, and that the residual strength of such samples is equivalent to or greater than the original unfatigued composite strength. In contrast, at 600° C, while the composite monotonic loading strength is quite high, all other fatigue and stress-rupture properties alike are controlled by the level of the composite proportional limit, i.e. the stress level at which matrix microcracking occurs. At 900° C the case is again somewhat different in that the run-out levels of stress in fatigue and stress-rupture both appear controlled by the level of the proportional limit, while residual strengths are nearly equivalent to the composite monotonic strength. The 900° C residual strength values also exceed the residual strengths obtained at 600° C. This indicates that, under levels of applied stress below those necessary for microcracking, i.e. below the proportional limit, the composite is more stable at 900° C than at 600° C. At 600° C, prolonged exposure under even low levels of stress causes a major decrease in composite strength and also a change in fracture mode to a relatively non-fibrous appearance (Fig. 12). At 900° C, however, the fracture mode of specimens can still remain very fibrous (Fig. 13).

4. Summary and conclusions

The proportional limit, i.e. the tensile stress above which inelastic deformation of the ceramic matrix composites tested takes place, plays an extremely important role in their overall mechanical performance. At room-temperature the tensile fatigue behaviour of two very different composites was contrasted. The first, an LAS-I matrix composite, normally exhibits a perfectly linear tensile stress–strain curve

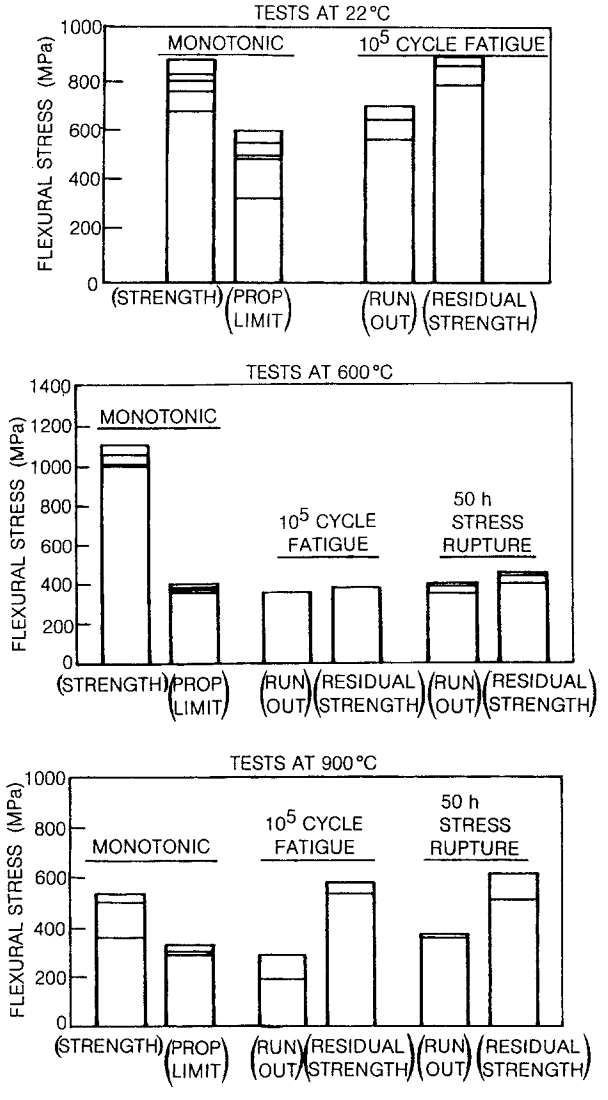

Figure 11 Comparison of four-point flexure test data obtained at temperature under monotonic, cyclic fatigue and stress-rupture test conditions.

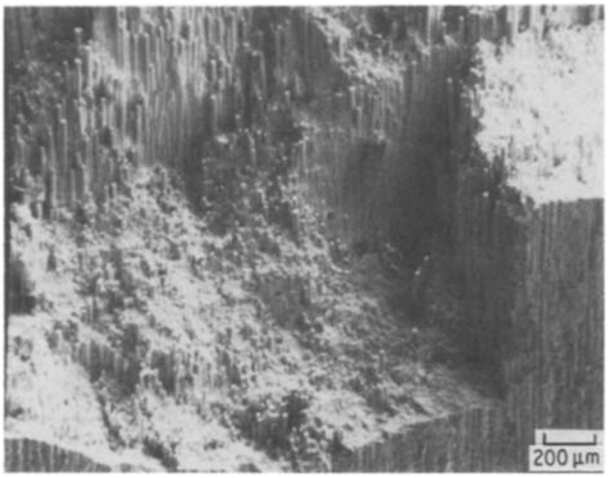

Figure 12 SEM photograph of 0° SiC-reinforced LAS-II tested in air at 600° C after 52 h at 366 MPa (residual strength = 406 MPa).

right up to fracture. Tensile fatigue of this composite did not cause any change in stress–strain behaviour, composite elastic modulus remained unchanged during fatigue and the stress to survive 10^5 cycles was approximately 65% of the ultimate composite tensile strength. The residual tensile strength of these pre-fatigued composites was also equivalent to that of the as-fabricated material. While the second system, an LAS-II matrix composite, was considerably stronger and exhibited a non-linear stress–strain curve, fatigue testing below the proportional limit resulted in the same sort of behaviour. It was only when fatigue stresses were raised above the proportional limit that a change in composite stress–strain behaviour was induced. These higher stress levels caused a significant reduction in the proportional limit stress and the appearance of a second linear stress–strain region having an elastic modulus less than the original. This change in behaviour, presumed to be due to the occurrence of matrix microcracking which occurs at stresses above the proportional limit, did not appear to cause a significant loss in composite residual strength. Too few tests were performed, however, to draw any definite conclusions and to fully address the important question as to whether repeated stressing in the matrix microcracking region causes composite degradation.

By flexural testing of the LAS-II matrix composite in air at higher temperatures the effects of exceeding the proportional limit were more clearly evident. At both 600 and 900° C, fatigue or stress-rupture testing above the proportional limit caused composite fracture with a noticeably embrittled composite fracture mode. As matrix microcracking is caused to occur, the oxidizing environment has an opportunity to penetrate the composite and cause a loss in strength similar to that noted in controlled-atmosphere tensile tests [8]. At applied stresses below the proportional limit composites survived the approximately 50 h of testing, regardless of whether fatigue or stress-rupture loading conditions were used. The importance of the proportional limit stress was particularly evident at 600° C, where composite initial flexural strength remained quite high yet even the residual strengths of those samples that survived the 50 h testing were limited to the proportional limit. Thus, undoubtedly some composite degradation took place during the test time of exposure to 600° C air. At 900° C this degradation at low stresses was not evident, i.e. residual composite strengths exceed the proportional limit stress. At the higher temperature it would appear

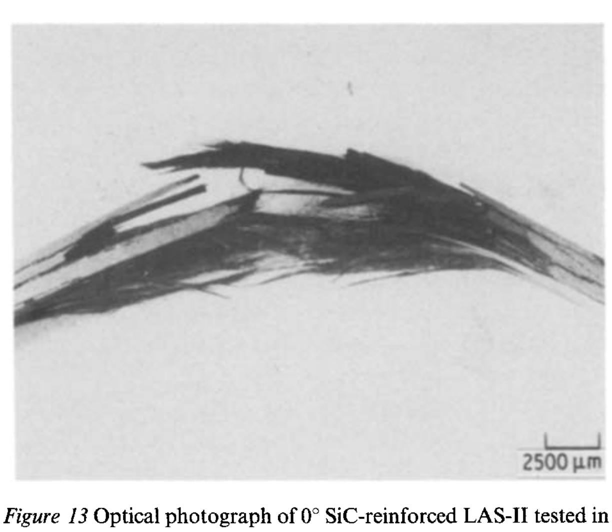

Figure 13 Optical photograph of 0° SiC-reinforced LAS-II tested in air at 900° C after 66 h at 364 MPa (residual strength = 620 MPa).

that the mechanism of degradation operative under low stress at 600° C has been effectively stopped. The mechanism for this superior 900° C behaviour is not evident at this time; however, as shown in Fig. 13, the desirable tough fibrous fracture shown by these composites is retained despite prolonged high-temperature testing.

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