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The mechanical behaviour of a cross-weave ceramic matrix composite

Part II *Repeated loading*

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In order to find out whether or not a cross-weave ceramic composite, graphite fibre/SiC matrix, would be prone to fatigue failure, tests in pulsating tension and pulsating compression have been carried out with the weave oriented in the 0/90 configuration. Both types of testing (at fairly high fractions of the ultimate monotonic failure load) cause creep strain, which is frequency dependent in tension, and ultimately complete failure can occur. Damage in pulsating tension is found to consist of cumulative microcracking and spalling, with the final failure mechanism broadly similar to that in monotonic deformation. Damage in pulsating compression appears dominated by delamination.

1. Introduction

The main disadvantage of ceramics in applications for structural components is their brittleness, which limits both their application and reliability. A possible advantage that they may not be prone to fatigue [1] is open to dispute [2, 3]. Substantial advances have been made in the development of ceramics having enhanced toughness during the past ten years. The toughening mechanisms that have been established include transformation, microcracking, ductile phase and fibre reinforcement [4, 5]. All these mechanisms have a common feature that material elements at, or near, the crack surfaces exhibit non-linear behaviour with hysteresis [4]. These toughness approaches have been successfully applied to improve the reliability of advanced structural ceramics or high performance ceramics which have potential applications as high temperature structural materials; e.g. for turbines and heat engines. All the toughened ceramics (including both monolithic and fibre composite types) have the strength and fracture characteristics which are usually associated with metallic materials rather than with brittle ceramics. These properties include nonlinear, irreversible stress-strain response, damage tolerance and the existence of an increasing resistance to crack growth with crack extension rather than single-valued fracture toughness. Therefore, the general concept that ceramics simply are immune to fatigue may not be true with the toughened monolithic ceramics and fibre reinforced ceramic composites. Recently, studies [6] have provided persuasive evidence of true cyclic fatigue-crack growth under tension–tension cycling in a magnesia-partially-stabilized zirconia (MgO-psZ). The results show evidence of crack closure analogous to behaviour in metals.

The most dramatic improvements in fracture properties of ceramics have been obtained by reinforcing with either continuous or discontinuous high strength fibres. In recent years, considerable efforts have been made in the study of the fracture strength of this class of ceramics. However, little is known about the fatigue behaviour under cyclic stress.

Carbon fibre reinforced SiC composite offers an example of a newly developed matrix-dominated ceramic composite. The mechanical properties and fracture characteristics of this composite both in tension and compression have been reported in a previous paper [7]. Preliminary results indicate that inelastic deformation mechanisms prevail in this material, and therefore, cyclic loading, which is associated with hysteresis and energy dissipation during deformation or cracking, can be anticipated to cause degradation of material performance. The object of the present investigation is to study the fatigue behaviour of a C/SiC cross-weave composite under tension–tension or compression–compression cycling. The studies have been based on the same composite material as explored in the previous paper [7].

2. Tension–tension cycling on notched specimen

Since the composite used here is the same as used previously [7], the interested reader is referred to that paper for details of the material, a SiC impregnated and coated cross-weave composite of carbon fibre bundles. Tests under tension–tension cycling were carried out with notched samples of the design shown

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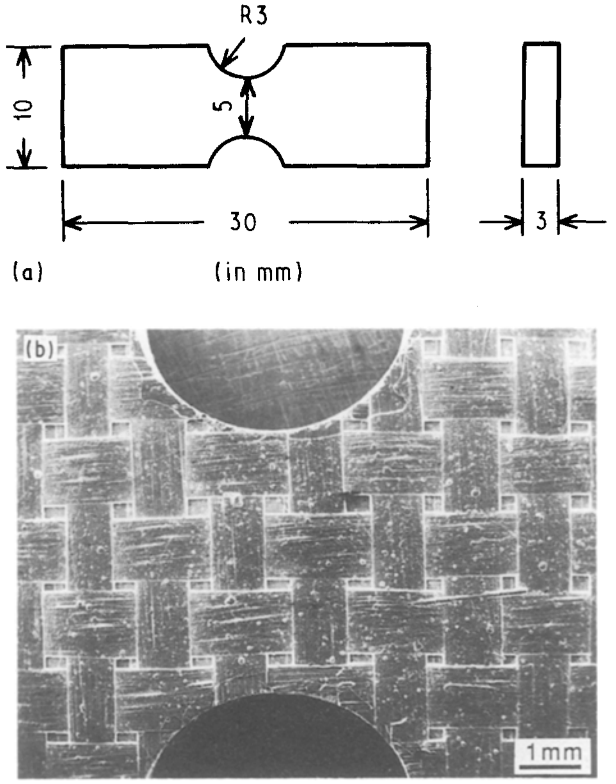

Figure 1 The notched specimen used for cyclic tension–tension tests showing (a) dimensions and (b) appearance.

in Fig. 1 cut from composite strips. An Instron hydraulic machine was used to conduct the tests at frequencies in the range of 0.01 to 0.2 Hz. The specimens were held by friction type grips and the alignment was achieved by using universal joints. Load versus displacement curves of the specimen were recorded during the test using an X–Y recorder with high sensitivity.

Fig. 2 shows the cyclic stress–strain response associated with an exploratory ascending step test. Initially, the test was run at 60% of the fracture load using a low cyclic frequency, 0.01 Hz; further frequency increments were made later. The effects of the first 100 loading cycles were recorded continuously and indicate cyclic creep behaviour. It is seen that the specimen exhibited a continuously decreasing cyclic creep rate with cycling and the cyclic creep behaviour of the specimen was found to be frequency dependent. As can be seen from Fig. 2, a change in frequency from 0.01 to 0.08 Hz led to a transient acceleration of creep deformation at the first cycle (on the basis of elapsed time) and then the elapsed strain rate was found to decrease. There is no obvious explanation so far for this remarkable observation. After 100 cycles of recording, the creep rate was typically observed to decrease and thereafter only occasional hysteresis loops were recorded. For the relatively few cycles of stress applied (1000), there was no sign that the creep finally abated. Fig. 2 shows that two more loading steps were applied at 74 and 80% of the fracture load, respectively. Any load increment had the effect of greatly (if transiently) increasing the inelastic residuum (creep). The particular sample illustrated in Fig. 2 failed at 80% of the monotonic fracture load after a total of 3680 cycles.

The development of cracking associated with cyclic creep was studied by SEM during test interruptions. Fig. 3 shows the appearance of the central part of the sample before repeated loading. There were no surface cracks initially visible at the indicated magnification

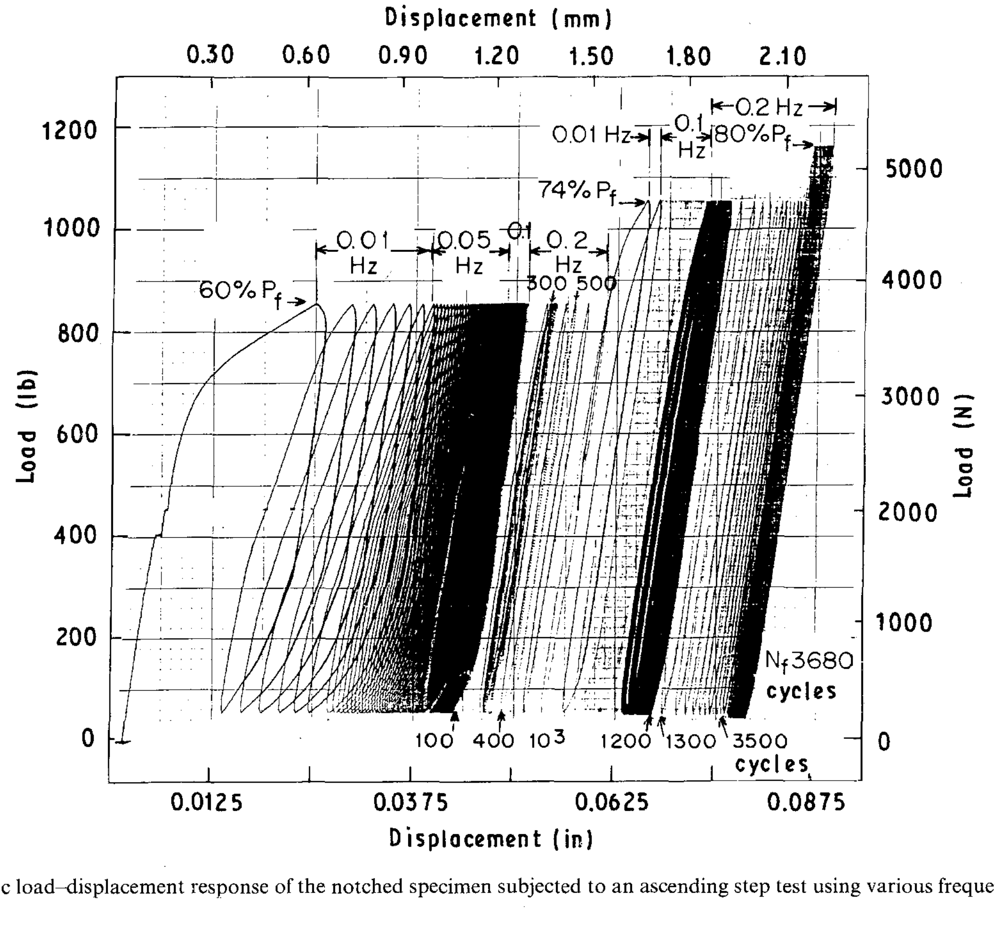

Figure 2 Cyclic load–displacement response of the notched specimen subjected to an ascending step test using various frequencies indicated.

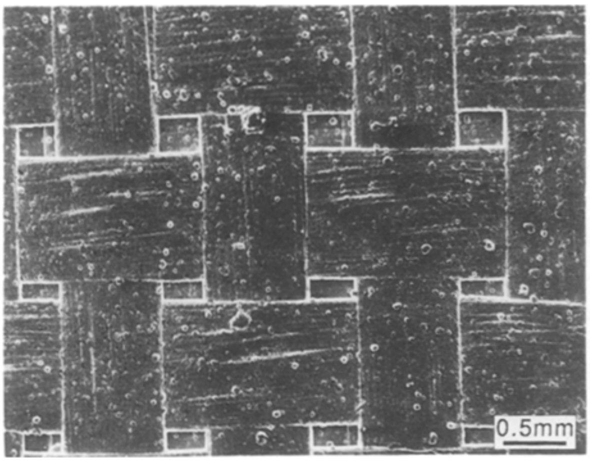

Figure 3 The central surface area of the notched specimen before testing.

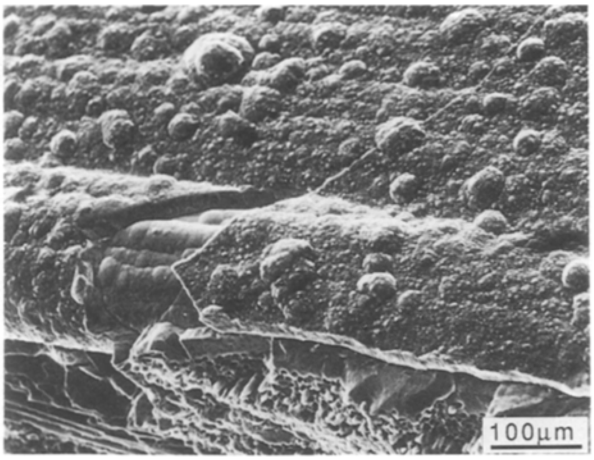

Figure 5 SEM micrograph showing the damage to the specimen edge due to cycling under a load range from 222 to 2783 N for 150 cycles.

or at higher magnification. The specimen was first cycled for 1850 cycles under a tensile load range from 222 to 3782 N (60% of the fracture load), then cycled for 2500 cycles under a tensile load range from 222 to 4672 N (74% of the fracture load) and finally cycled to failure under 222 to 5116 N (81% of the fracture load). The total number of cycles to failure was found to be 4530 cycles. The cycling frequency was 0.05 Hz. The test was interrupted at 150 cycles and 850 cycles for investigation of the damage phenomena by SEM. Fig. 4 indicates the same area as shown in Fig. 3 but after 150 cycles surface cracks can be clearly seen after the specimen was exposed to repeated loading for this short period. Fig. 5 shows the resulting damage to the specimen edge by cycling for 150 cycles. Figs 6 and 7 were taken from the same areas as shown in Figs 4 and 5, respectively, and show the multiplication of surface cracks and spalling episodes associated with a further cyclic exposure for 700 cycles. These cracks were clearly important in the ultimate failure of the specimen since they were ultimately incorporated into the final rupture as shown in Fig. 8a, b, c and d. Normally, the cracks seemed confined to the SiC matrix and appeared to avoid the bundles of carbon fibres. Occasionally, the cracks penetrated the bundles, cutting the fibre in the plane of the matrix crack. In comparison with the development of cracking under monotonic loading as reported in the previous paper [7], it seems here that repeated loading created more surface cracks and spalling. As indicated in Fig. 2, on repeated loading there is a considerable creep. This is attributed to the opening of cracks under load and their failure to close completely on unloading, because they are propped open by debris.

3. Tension–tension cycling on a smooth specimen

In order to exclude the possible contributions to the irreversible strain on repeated loading from factors such as stress concentrations, flaws by machining (the specimen was cut from a strip) and specimen-grip interaction, a smooth specimen was used for tension–tension cyclic loading. A smooth specimen is one tested in the as-received and as-processed condition, which was long enough to avoid possible grip-specimen interactions. The overall length of the specimen was 140 mm and it measured 3.2×8.1 mm^2 in section with a gauge length of 40 mm. The experimental fatigue arrangement was similar to that used for the notched specimen except a clip-on extensometer was attached to the gauge length to measure the strain over the gauge length without involving grip

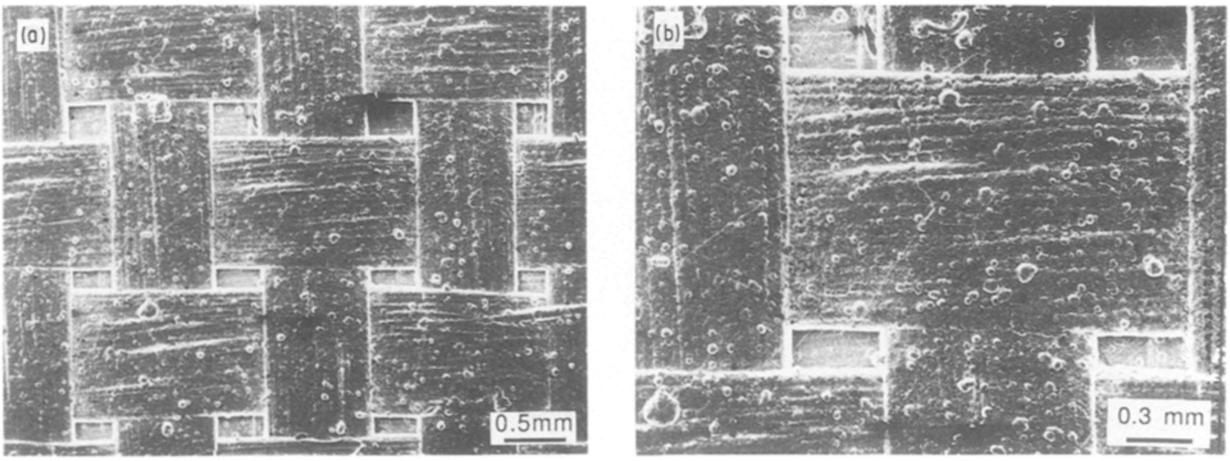

Figure 4 The same area as in Fig. 3 after the specimen was cycled for 150 cycles under a load range from 222 to 3782 N at different magnifications. The loading axis is horizontal.

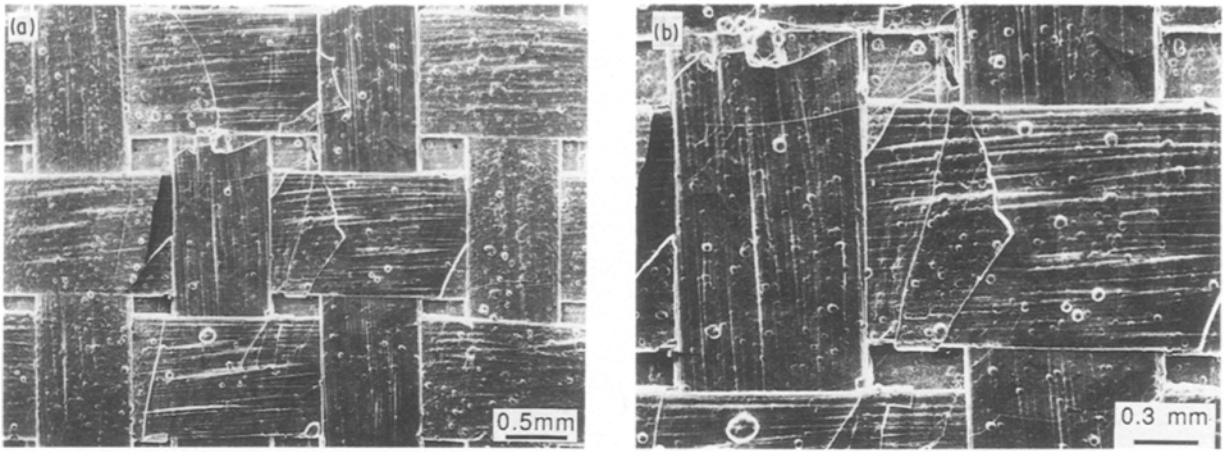

Figure 6 SEM micrographs taken from the same areas as in Fig. 4 after the specimen was subjected to a further cyclic exposure for 700 cycles. (a) and (b) were taken under different magnifications.

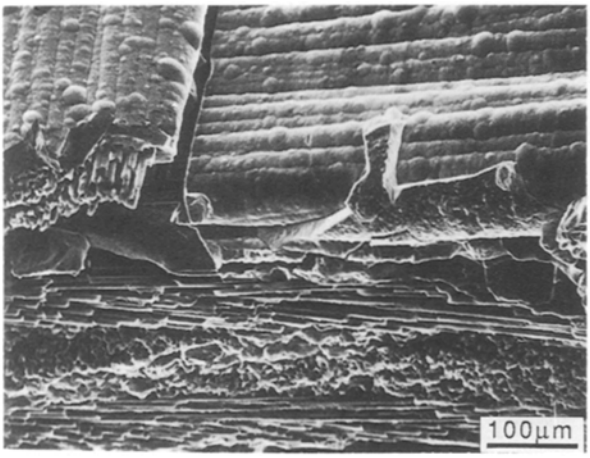

Figure 7 The appearance of the specimen edge after a further cycling for 700 cycles. Notice that the far right area shows the same area as seen in the left lower corner of Fig. 5.

effects. The specimen was first stretched to a stress of about 308 MPa (70% of the fracture stress) and then subjected to repeated loading under a tensile stress range from 8.75 to 308 MPa. The cyclic stress–strain response in the first 100 loading cycles was recorded continuously and is shown in Fig. 9. It is very interesting that on unloading there is a considerable inelastic strain residuum, about 20% of the strain applied. This residuum continues to increase on repeated loading after the first tensile application.

After 100 loading cycles, the test was interrupted and the specimen surface was observed using a stereo binocular microscope. No cracks were visible on the exterior surface of the specimen. A further 900 loading cycles were then imposed, but these caused little irreversible strain. SEM observation at high magnification revealed the existence of microcracks on the specimen surface as shown in Fig. 10. In comparison with the notched sub-specimen, the full size smooth specimen gave much less inelastic strain residuum on repeated loading and fewer surface microcracks as well. Based on the results for both types of the specimen, the nonlinear behaviour is tentatively interpreted in terms of microcracking behaviour as follows: during load application, the existing population of flaws either open as microcracks or stimulate microcracking, the elastic deformation including the sum of the crack opening displacements. During unloading, a certain fraction of these cracks remains open to produce the strain residuum because of wedging, we believe, spalled fragments or crack closure associated with surface roughness [6]. Fig. 9 shows that repeated loading and unloading causes a slight creep strain, possibly by accumulation of these irreversibilities. The difference in behaviour between the notched and smooth specimens reflects an increased population of flaws introduced by the machining of the notch.

From comparison of the fracture surface of a cyclically-failed specimen with that of a specimen broken in monotonic deformation (reported previously [7]), it seems that the mechanism of failure by cyclic loading is similar to that of monotonic loading but tends to be more gradual and somewhat more localized although it is possible that the use of a notched specimen forced the localization. A more definitive study of the failure mechanism will require examination over a wider range of load fractions and within the specimen. The latter represents a difficult problem because of the danger of artificially introducing cracks during specimen sectioning and preparation. Methods are under development to circumvent this problem.

4. Cycling under pulsating compression loading

As reported in the previous paper [7], the matrix dominated composite C/SiC, subjected to compressive loading, mainly shows linear elastic behaviour. Few matrix cracks were believed to be generated until the moment of final failure under compressive loading. It was previously shown [1, 8] that crack extension is often observed in ceramics during unloading from a compressive stress, a phenomenon which could be classified as a fatigue effect or could lead to a fatigue phenomenon. More recently, investigations on polycrystalline alumina indicated [9] that the application of fully compressive cyclic loads can lead to Mode I crack growth at room temperature. Therefore, the

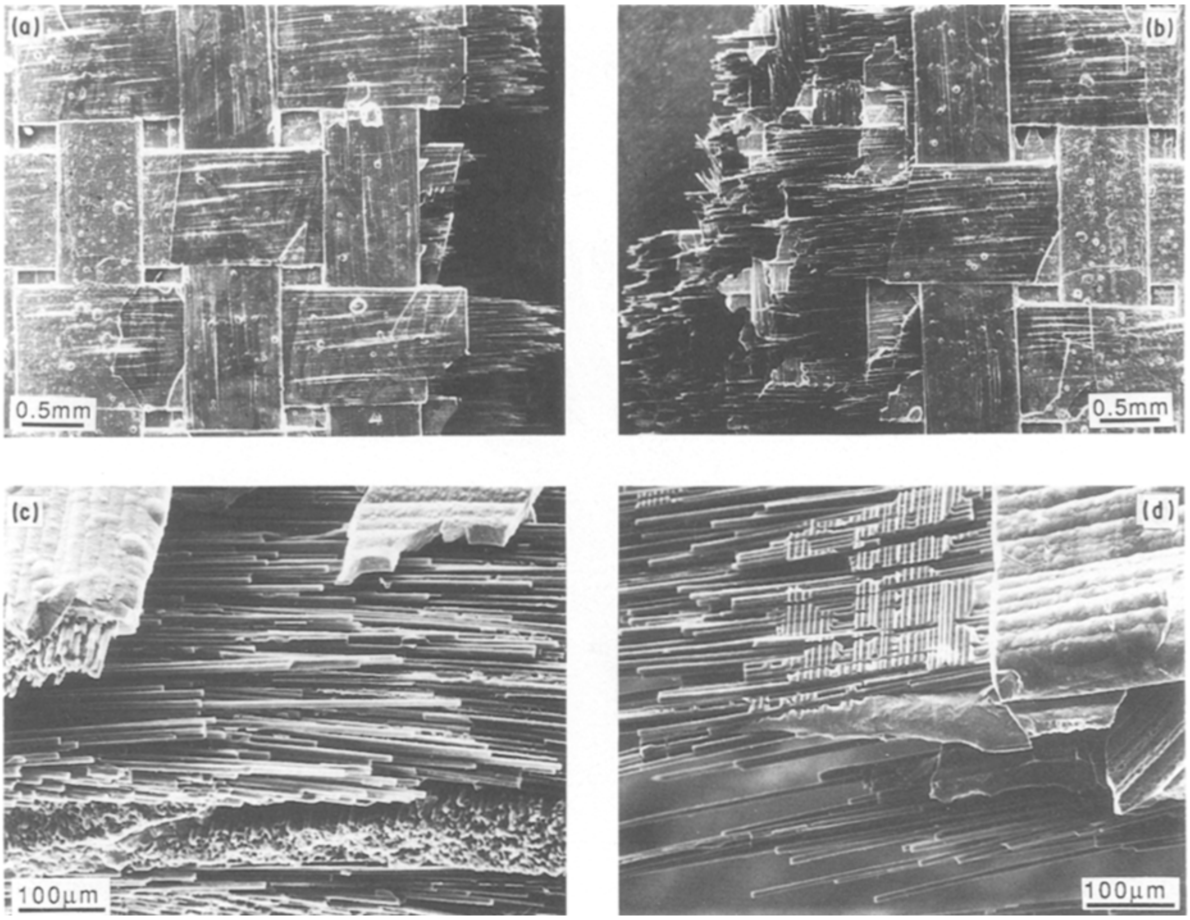

Figure 8 SEM micrographs showing the characteristic features of the totally failed specimen. (a) and (b) are seen normal to the surface. (c) and (d) show the appearance of the specimen edge.

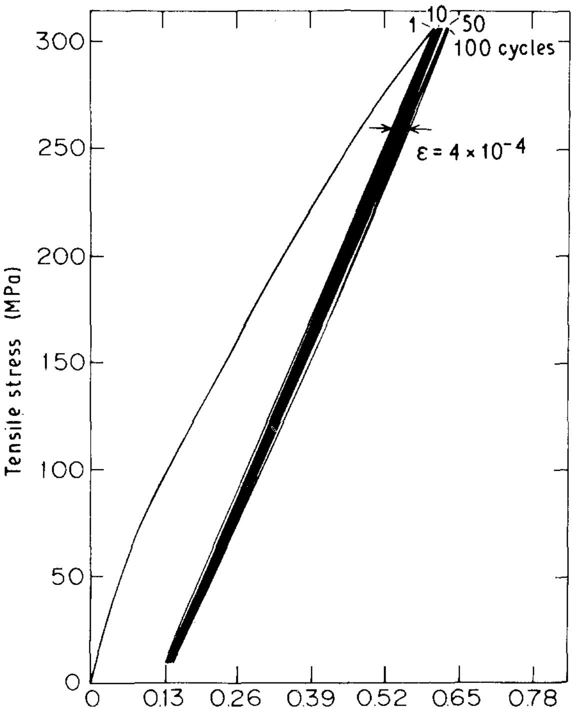

Figure 9 Stress–strain response of a smooth full-size specimen cycled in tension (R = + 0.028) for 100 cycles.

damage accumulation that occurs under cyclic compressive loads is a problem of particular interest and practical importance for ceramics, especially for cross-weave composites which contain ordered arrangements of voids as part of the weave structure. However, the fracture behaviour of both monolithic ceramics and ceramic matrix composites under cyclic compression is not understood. Tests of compression–compression cycling were therefore performed in the present study in a preliminary attempt to analyse the compressive mode of fatigue.

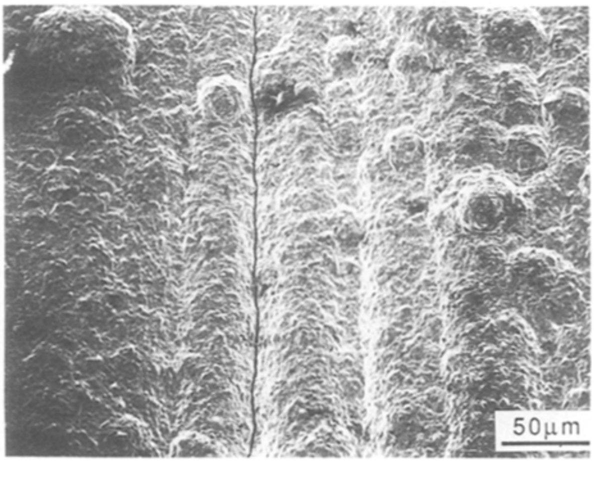

Figure 10 SEM micrograph showing a surface microcrack after the specimen was cyclically loaded for 1000 cycles under a stress range from 8.75 to 3.08 MPa. The stress axis is horizontal.

Tests were carried out in a conventional Instron servohydraulic machine. A specially designed test fixture described elsewhere [7] was used to ensure adequate alignment and to measure the strain of the

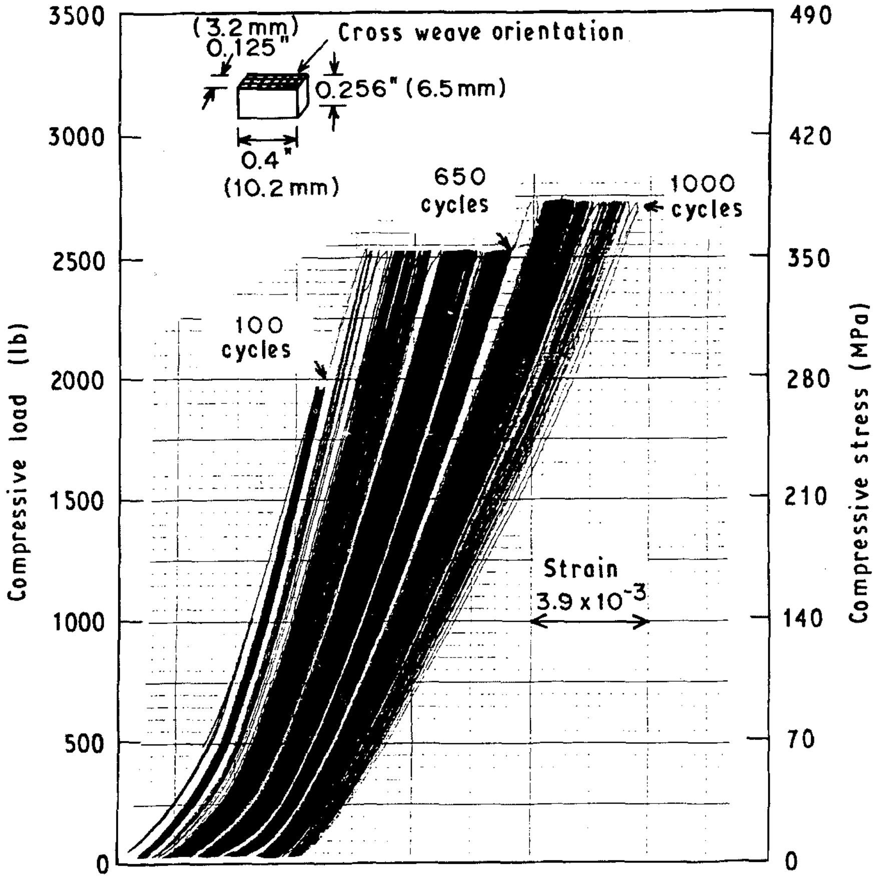

Figure 11 Cyclic compressive stress–strain response of a C/SiC composite subjected to a three-step test. The configuration of the specimen is shown in the insert.

specimen during cyclic compressive loading. Fig. 11 shows the cyclic stress–strain response associated with an exploratory three-step test. The configuration of the specimen, also shown in Fig. 11, indicates that the cross-weave was oriented in a 0/90 arrangement. The specimen was first cyclically loaded under a compressive stress amplitude ranging from -7 to -277 MPa for 100 cycles, then cycled for 550 cycles under a stress range from -7 to -354 MPa, and finally cycled to complete failure under a stress range from -7 to -382 MPa. The total number of cycles to failure was 1 000 cycles exactly (and fortuitously). A triangular wave form with a frequency of 0.2 Hz was used throughout the entire test.

Several interesting points were revealed in the results shown in Fig. 11. Firstly, compressive cycling caused creep deformation, the rate of which declined with cycling and finally reached a saturation stage at a given stress level. Secondly, sudden, large increases of cyclic creep were observed during the cycling. These episodes can be seen as gaps in the generally solid bands of pen recordings traced out during the creep process. This phenomenon is reminiscent of strain burst behaviour observed during cyclic deformation of metals and alloys [10–12]. Thirdly, a continuous acceleration of cyclic creep occurred during the onset of the final failure in the specimen.

A preliminary attempt was made to explain the observed phenomena in terms of the damage of the specimen. Fig. 12 shows a SEM micrograph from a specimen which was cycled for 500 cycles under a loading pattern similar to that shown in Fig. 11. It can be seen that a characteristic feature of damage is edge delamination of the specimen. Combining this result with those obtained from monotonic tests in compression [7], it is reasonable to conclude that edge delamination of the specimen is a major damage process which is responsible for a good part (but not necessarily totally) of the cyclic creep deformation and strain bursts observed during cyclic compression loading. However, it should be pointed out that while the results gathered so far are extremely interesting, the study must be considered far from complete because of the limited number of samples and stresses used, and the great complexity of the phenomena.

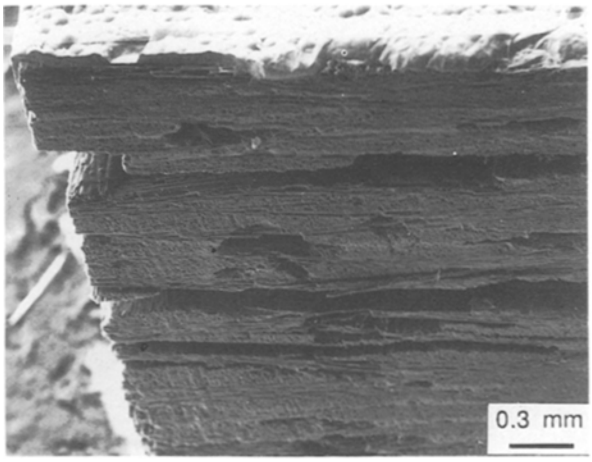

Figure 12 SEM micrograph of the specimen edge delamination after the specimen was cycled in compression–compression for 500 cycles. The stress axis is horizontal.

5. Conclusions

From tests on a C/SiC cross-weave composite carried out under cyclic loading both in pulsating–tension and pulsating–compression, we offer the following conclusions:

1. Tensile cyclic creep behaviour, which seems to be frequency-dependent, was observed when notched specimens were tested in pulsating–tension. Repeated loading in such specimens induced surface cracks and spalling. Repeated loading on a smooth specimen gave much less creep strain and fewer surface cracks and spalling than observed in the notched sample; this behaviour is attributed to the fewer pre-cycling imperfections in the smooth specimen.
2. Compression–compression cyclic loading also caused a significant creep behaviour and a strain burst phenomenon was observed. Edge delaminations which were observed in samples during repeated loading are taken to be mainly responsible for the compressive cyclic creep behaviour.
3. The mechanisms of failure by cyclic loading appear to be similar to those of monotonic loading but tend to occur more gradually and perhaps on a somewhat more localized level, although it is possible that the specimen design forced this conclusion.
4. Fatigue or repeated loading will cause cumulative damage and therefore will degrade the inelastic properties of the ceramic composite materials. Obviously, if ceramic matrix composites are considered to be materials for safety-critical structural applications, evaluation of the materials with respect to cyclic loading will become absolutely essential.

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