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Fatigue behaviour of 3-d SiC/SiC Composites

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A tension–tension fatigue damage analysis was performed using 3-d silicon carbide fibre reinforced (orthogonal) silicon carbide matrix (SiC/SiC) composites. Two groups of SiC/SiC specimens were tested. The first group consisted of samples without any oxidation protective top layer coating, whilst the latter one contained samples covered with a well fitting, chemical vapour deposited (CVD) SiC system. This coating is necessary for the material to sustain high temperatures. Both the coated and uncoated material had a fibre volume fraction of about 36% equally distributed in three rectangular directions. Load control fatigue tests were conducted at room temperature. The fatigue life was found to decrease by increasing the cyclic stress level. A power-law equation is proposed, which correlates the applied maximum stress during the fatigue test with the number of cycles to failure. In general, the presence of the coating layer decreases the static strength of the material. However, the nominal maximum cyclic stress for which the endurance fatigue limit appeared, remained unaffected by the presence of the oxidation protective SiC coating. Microstructural examination has also been performed on the fractured specimens and it reveals some of the failure mechanisms of the composite that appeared under quasi-static and dynamic loading.

1. Introduction

Ceramic matrix composites (CMCs) are ideal structural materials for high temperature applications due to their outstanding structural properties such as high specific strength, stiffness and toughness combined with crack insensitivity. Therefore, CMC's are already used in space technology in certain applications, that require a material with good mechanical loading and resistance to thermal damage behaviours.

Nevertheless continuous material development is necessary if further improvements in the quality and properties are to be achieved. In particular attention should be focused on optimizing the fibre content and fibre preform structure as well as the manufacturing process and process management, including matrix formation and fibre–matrix interface control. Special emphasis should be concentrated on improving the material toughness, which in contrast to bulk ceramics is relatively high in the case of CMC's [1]. Two main microstructure parameters affect the fracture behaviour of CMC's:

(i) the fibre strength.
(ii) the interfacial properties, particularly the fibre–matrix interface shear stress associated with friction and/or debonding. This is controlled by fibre coating and by residual thermal stresses [2].

In the case of SiC/SiC composites the control of the interfacial properties is currently managed by the application of a chemical vapour deposited (CVD) carbon layer on the SiC fibres.

However, due to the working environment, the material has to withstand corrosion (which consists mainly of attack by oxygen or water vapour) up to the temperature of a particular application. Therefore oxidation resistant coatings have to be developed and applied onto SiC/SiC composites. In the present case this coating was a top layer, well fitting, chemical vapour deposited SiC system [3].

For efficient use of materials in any application the failure mechanisms and damage behaviour under quasi-static and fatigue loading must be well understood. In general, only limited work has been published on the fatigue behaviour of ceramic/ceramic composites [4–8]. Most of this work is related to glass ceramic matrix, SiC fibre reinforced composites. In the case of SiC/SiC composites work has been restricted to unidirectional (1-d) and bi-directional (2-d) materials [2, 8].

In the present work static tensile and tension–tension fatigue damage analysis is performed on 3-d SiC/SiC composites with and without an oxidation protective top SiC layer coating. The degradation of both strength and stiffness after fatigue is also studied in the case of tested coupons that exceeded the endurance fatigue limit.

Finally a power-law equation is proposed which correlates the applied maximum stress during the fatigue test with the number of cycles to failure in both the cases of coated and uncoated material.

2. Experimental procedure

2.1. Description of the material

The 3-dimensional (3-d) SiC/SiC material was produced by Aerospatiale Bordeaux, France as a part

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of a Brite/Euram Project entitled "Development and Characterization of CMC and C/C Composites" (Contract No. BREU 0334-C).

The 3-d SiC/SiC material was made out of Nicalon fibres and a metal-organic based SiC matrix. The woven fibre preform had a 3-dimensional orthogonal architecture shown in Fig. 1. The development of the material includes optimization of all of the seven basic manufacturing steps:

(i) Weaving of an orthogonal 3-d SiC preform.
(ii) Application of a thin (0.1 μm) CVD carbon layer for fibre–matrix interface control.
(iii) SiC, submicron sized powder, infiltration under pressure for predensification of the raw fibre preform.
(iv) Drying.
(v) Impregnation by SiC precursor (PVS) using liquid injection to finally densify the composite material.
(vi) Curing and pyrolysis.
(viii) Final heat treatment at 1000 °C in an inert atmosphere.

The final product has a density of 2.1 g cm^{-3} and an open porosity of about 20%. The fibre volume fraction is 36%, equally distributed in the three rectangular directions.

Plates of 6 mm thickness were manufactured. Due to the material thickness and porosity, there was no possibility of final quality control using ultrasonic scanning. The material can be used up to 1200 °C but an oxidation protection layer is necessary for oxidative application above 500 °C.

Microstructural analysis by optical microscopy was performed on both coated and uncoated specimens. It revealed that the open porosity was not equally distributed or of the same size along the composite. The carbon interface layer was easily recognized.

The SiC coating layer that fills-in part of the porosity, exhibits an almost constant thickness of 200 μm which was deposited in a stepwise procedure, of two pure SiC layers, each of 100 μm thickness. No cracks occur in the SiC coating. The interfacial strength between the SiC coating and the substrate material seems to be very high, since no coating delamination occurs either during quasi-static tensile loading or the fatigue tests.

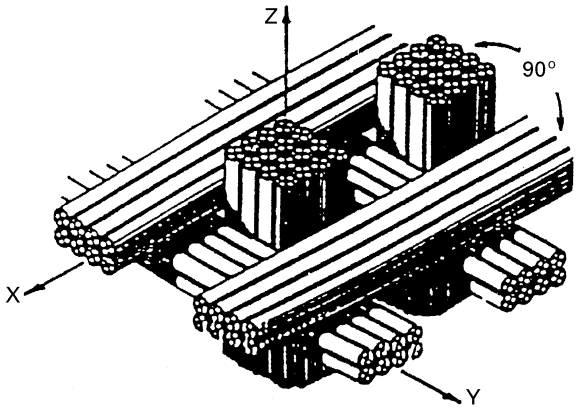

Figure 1 Schematic representation of the 3-d SiC/SiC perform reinforcement.

2.2. Testing procedure

Straight strip specimens of length 200 mm, width 10 mm and thickness 6 mm (the original plate thickness) were cut from the SiC/SiC composite plates, using diamond blade of a low speed saw. Half of them were covered with a double layer SiC coating applied by a CVD process.

Tapered aluminium end-tabs were used for the gripping area of the specimens, and they were bonded using epoxy resin (Ciba LY-564/hardener HY 2954) and processed using the recommended post curing cycle.

All the tensile and tension–tension fatigue tests were carried out on a closed loop servo-hydraulic testing machine equipped with a hydraulic gripping system, at room temperature, in air. The tensile tests were used to measure the static mechanical properties of both coated and uncoated SiC composites. These characteristics, particularly the ultimate tensile strength (UTS), are of specific interest because all the fatigue loads as well as the material endurance fatigue limit are expressed as a percentage of the UTS. In addition any scattering in the material fatigue life would probably arise from the scattering observed for the UTS. Also the degradation of both the ultimate tensile strength and the modulus of elasticity of fatigue loading specimens were used as a measure of the intensity of fatigue effect on the 3-d SiC/SiC composites. For this purpose any test coupons that exceeded the endurance fatigue limit of – no failure after 10^6 cycles were further tested under tensile loading conditions.

The tensile tests were conducted using position control under a cross head velocity of 0.1 mm per min. Special care was taken over the correct alignment of the test specimens, using an in-house developed optical fixture. The strain measurements were performed using a Mayes extensiometer device (gauge length 20 mm), which was removed from the specimen shortly before the final fracture. The application of a strain gauge on the specimen surface proved to be impossible due to the surface porosity and roughness.

Load-control fatigue tests were performed, under a sinusoidal waveform at a frequency of 10 Hz. The ratio of minimum to maximum applied stress during fatigue (R parameter) was 0.1.

Fatigue damage analysis was performed by microstructural examination of longitudinal and transverse cross-sections of the tested specimens.

3. Results and discussion

3.1. Quasistatic tensile behaviour

Typical examples of the axial stress–strain response of 3-d SiC/SiC composites both uncoated and coated are given in Figs 2 and 3 respectively. The materials have a non linear behaviour as a result of the compliant SiC fibre preform which has been stabilized with the poor mechanical performance SiC matrix. This kind of structure permits the development of extensive damage areas within the matrix, which allows the release of the stress concentration around the fibre bundles. The damaged areas increase during the tensile test, leading to a stiffness degradation, which is

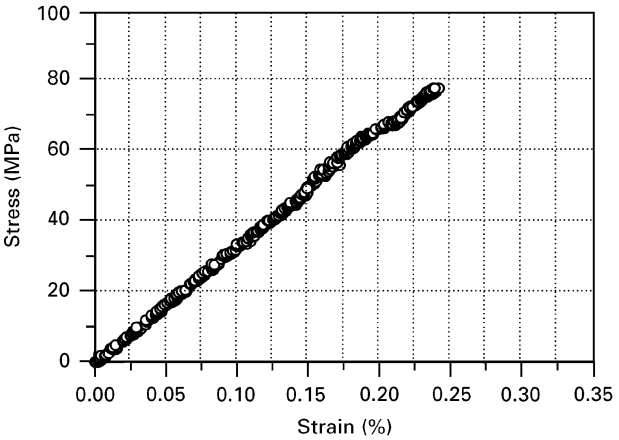

Figure 2 Typical Stress–Strain curve of the uncoated 3-d SiC/SiC Composite with $E = 27$ GPa, $\sigma_{ult} = 161$ MPa and $\varepsilon = 0.6\%$.

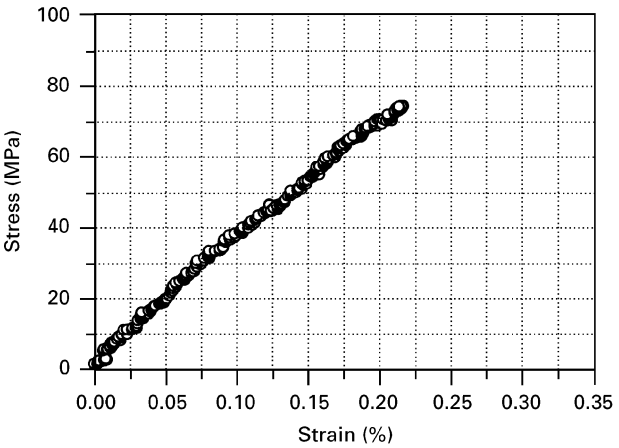

Figure 3 Typical Stress–Strain Curve of the coated 3-d SiC/SiC Composite with $E = 35$ GPa, $\sigma_{ult} = 138$ MPa and $\varepsilon = 0.49\%$.

monitored macroscopically as a non linear stress–strain behaviour.

Table I summarizes the results obtained during the tensile tests on both uncoated and coated 3-d SiC/SiC samples. These results are the mean values of five specimens per group. Two different values are reported for the modulus of elasticity. The first value represents the tangent modulus calculated at the initial part of the stress–strain curve, while the second one represents the secant modulus, typical in the case of a nonlinear behaviour. It has been calculated by applying a linear interpolation onto the stress–strain curve between 10 and 50% of the tensile failure strain.

For the strength calculation, in the case of coated samples, the cross-sectional area used was measured without taking into account the coating thickness this implies the assumption that the coating was not load bearing.

From Figs 2 and 3 it is obvious that both sample types exhibit a common non-linear behaviour over a critical stress. This stress level is about 60 MPa. The uncoated 3-d SiC/SiC material has an ultimate tensile strength of 160 MPa which is 15% higher than that of the coated one. The scattering of the strength data was also higher for the coated samples. Furthermore, the tangent modulus of elasticity of the uncoated SiC/SiC material, calculated at the beginning of the stress–strain curve was 23% lower as compared to the relative modulus of the coated one. This difference falls to approximately 10% when the secant modulus is taken into account.

This more "brittle" behaviour of the coated 3-d SiC/SiC material was reflected in the tensile failure strain which is about 12% lower compared to the failure strain of the uncoated sample.

As a general remark, the coated samples gave broader scattering for all the measured parameters. Optical stereo-microscopy performed on the fracture surfaces of broken coated tensile coupons revealed that the SiC coating intruded within the 3-d SiC preform through the composite open porosity. Thus part of the materal's open porosity has been filled in and the materials ability to arrest crack development has been reduced. This effect also leads to a more "dense" and "stiff" material which is reflected in a higher measured modulus of elasticity.

However, this cannot be the only reason for the observed strength reduction in the case of the coated samples. Measurements of the thermal expansion coefficient of uncoated and coated 3-d SiC/SiC, obtained by using a Thermomechanical Analyzer (TMA) module apparatus (DuPont model 943), may also implicate the residual stress field produced by the coating application as an additional effect that contributes to the strength reduction. More precisely, the thermal expansion coefficient of the coated samples was $3 \times 10^{-6}\,^{\circ}C^{-1}$ in contrast to the relative value of the uncoated samples which was measured to be $1.5 \times 10^{-6}\,^{\circ}C^{-1}$. This difference is reflected in a thermal expansion misfit between the substrate and the coating material. As a result, a tensile residual stress field is produced within the coating layer, which supports the crack initiation at the coating layer.

In the case of coated samples, three different areas could be identified; the dense SiC coating layer, the interphase area (densified 3-d SiC/SiC composite with reduced open porosity) and the unaffected bulk substrate.

TABLE I Summary of tensile tests results for both uncoated and coated 3-d SiC/SiC composites

Properties	Uncoated 3-d SiC/SiC		Coated 3-d SiC/SiC	
	Mean value	SSD	Mean value	SSD
Tensile strength σ (MPa)	161	0.7	138	6.7
Tensile failure strain ε (%)	0.6	–	0.49	–
Tensile tangent modulus of elasticity E_f (GPa)	27	0.7	35	1.65
Tensile secant modulus of elasticity E_{sec} (GPa)	26	1	30	6

It was observed that a tentative surface crack produced by microstructural defects has already present and that it advanced within the SiC coating and the densified interphase when the tensile load was increased.

As the crack approaches the bulk substrate part of the composite it is arrested. This kind of damage leads potentially to a reduction of the loading cross-sectional area of the test coupon and therefore a lower nominal transfer load. Macroscopically this is observed as a strength reduction.

3.2. Tension–tension fatigue behaviour

Different groups, each containing 3 specimens of either uncoated or coated 3-d SiC/SiC composites were tested at different maximum stress levels in order to investigate the fatigue behaviour of the materials. Some tests have also been carried out at high values of the maximum applied stress on single specimens in order to obtain a more representative initial part of the fatigue life curve.

Fig. 4(a and b) present fatigue results obtained under different cyclic stress levels for the uncoated and coated samples respectively. These curves show the maximum applied stress during the fatigue test versus the number of cycles to failure. In both figures the width of the grey zone represents the scattering of the fatigue data.

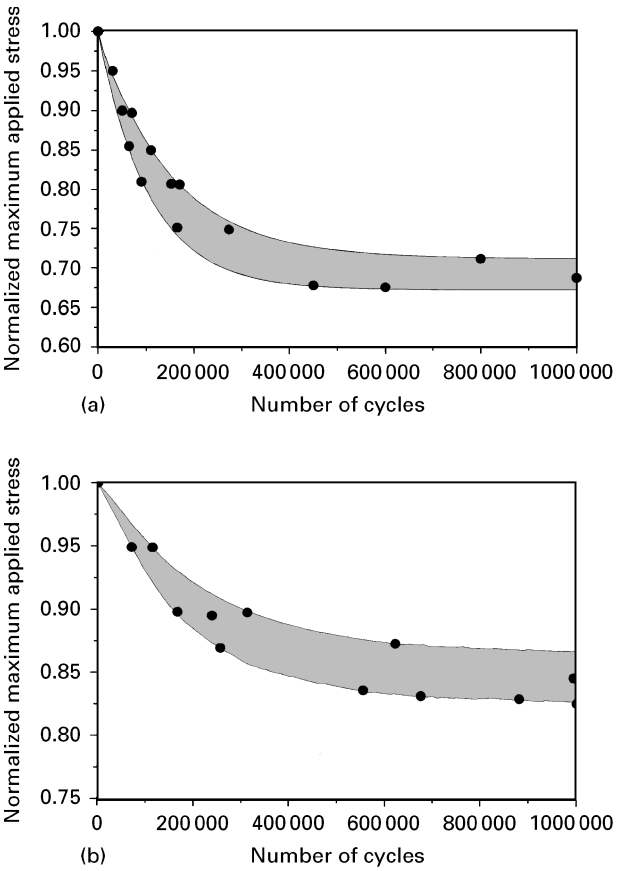

Figure 4 (a) Normalized maximum applied stress versus fatigue life (Wöhler curve) of uncoated 3-d SiC/SiC Composite. (b) Normalized maximum applied stress versus fatigue life (Wöhler curve) of coated 3-d SiC/SiC Composite.

TABLE II Maximum applied stress which corresponds to the endurance fatigue limit both for uncoated and coated 3-d SiC/SiC material

Maximum stress level for endurance fatigue limit	% UTS	MPa
Uncoated 3-d SiC/SiC	70	114
Coated 3-d SiC/SiC	85	117

Table II summarizes in a more direct manner the stress level for which the endurance fatigue limit (EFL) of the uncoated and the coated samples was reached, both as percentage of the ultimate tensile strength and as an actual stress value. The uncoated 3-d SiC/SiC material reaches the EFL for a stress level up to 114 MPa (70% of σ_{UTS}), which is well above the point at which static stress–strain curves deviate from linearity. The coated material attains the EFL at a maximum stress equal to 117 MPa, which represents 85% of its tensile ultimate strength.

Thus, the coated material seems to recover its ability to transfer load under fatigue conditions, which appears to be diminished under static loading.

As fatigue proceeds, in parallel with some very interesting effects which will be discussed later on, the interfacial bonding between the coating and the substrate material continuously decreases due to interfacial wear [2, 9]. This relaxes the residual stress field. This effect also "returns" some of the materials open porosity that had been filled in during the coating application phase.

Heuristic conclusions thus drawn, allow a designer to use the properties of the uncoated material even in the case of the application of coated samples. A power-law correlation between the cyclic stress and the fatigue life was also observed. The corresponding life equation of the uncoated 3-d SiC/SiC composites can be written as;

$$\frac{\sigma_{\text{applied}}}{\sigma_{\text{ult}}} = a^* N_{\text{f}}^{-k}$$

where σ_{applied} is the maximum applied stress during the fatigue test, σ_{ult} is the UTS of the material, N_{f} is the number of fatigue cycles to failure, a is a constant and k is the fatigue strength exponent. The parameters a and k were calculated for the case of the uncoated 3-d SiC/SiC material by fitting the relative life time plot and have values of $a = 2.21$ and $k = 0.04481$.

Fig. 5 shows a more convenient representation of fatigue data in a $\log\sigma - \log N$ form in order to obtain linear plots.

In the case of fatigue tested coupons that exceed the endurance fatigue limit (10^6 cycles), tensile tests were performed in order to investigate any degradation of their stiffness and strength characteristics. The conditions during these tensile tests were identical to those used in the case of non-fatigued specimens.

Depicted in Fig. 6 are the stress–strain curves of both uncoated and coated 3-d SiC/SiC composites. Both materials appear to have an almost identical

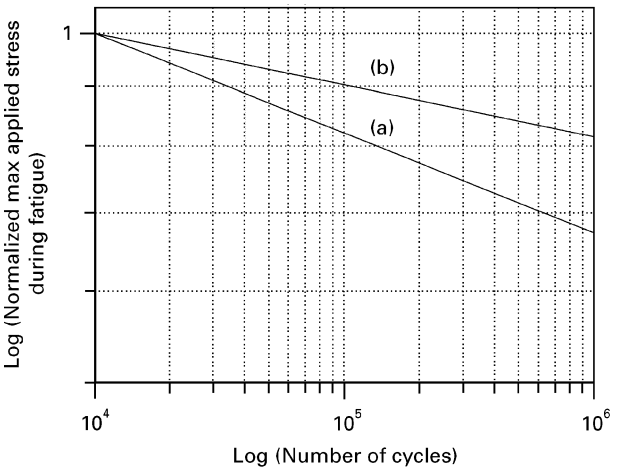

Figure 5 Normalized maximum applied stress versus fatigue life on a logarithmic scale: a) Uncoated 3-d SiC/SiC Composite; b) Coated 3-d SiC/SiC Composite.

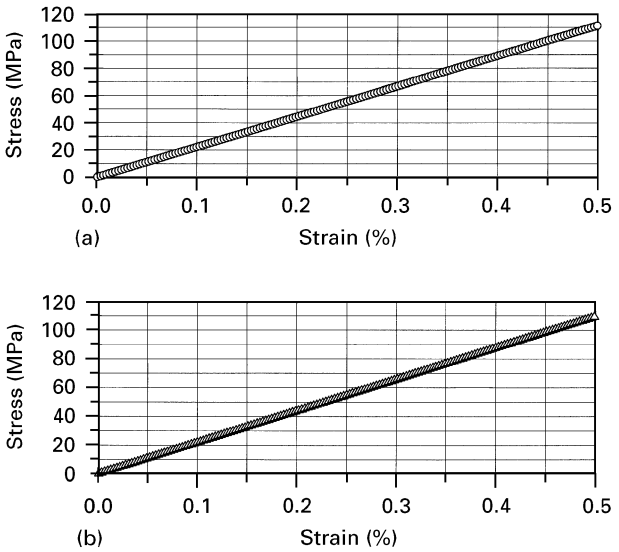

Figure 6 Typical stress–strain curves of 3-d SiC/SiC Composite after 10^6 fatigue cycles: a) Uncoated 3-d SiC/SiC Composite; b) Coated 3-d SiC/SiC Composite with $E = 21.9$ GPa, with $E = 22.3$ GPa.

behaviour. The characteristic results can be summarized as:

(i) The fatigued samples exhibit a linear stress–strain curve in contrast to their response under tensile conditions before the application of fatigue.
(ii) The modulus of elasticity for both uncoated and coated samples is almost the same (22 GPa) which appears to be 18% lower compared to that of the uncoated 3-d SiC/SiC composite before fatigue.
(iii) The relative strength of both materials exhibit very close values (128 MPa) with the coated material appearing to have a wider scattering of the relative data. This value is 18% lower compared to the relative strength of the uncoated sample before fatigue.
(iv) The failure strain in both cases is very close to 0.6% (which is the failure strain of the uncoated material before fatigue) and seems to be governed by the fibre failure strain.

The fatigue effect on the microstructural characteristics of ceramic matrix composites has been discussed recently [2, 9–11] using a 1-dimensional approach. In the present case the 3-d structure of the reinforcement implies more complicated fracture modes.

However, the hypothesis of decreasing the interlaminar shear strength (ISS) in between the fibre–matrix interface [9–11] is a useful concept for qualitative explanation of the 3-d SiC/SiC behaviour of both uncoated and coated materials under fatigue loading.

According to this approach, the ISS continuously decreases as a function of fatigue cycles up to a critical level. This is due to interfacial wear which leads to a residual stress relaxation. On the other hand the ISS has a non-finite limiting value due to friction between the fibre and matrix and additionally the aspirates on fracture surfaces whenever the fibre–matrix interface

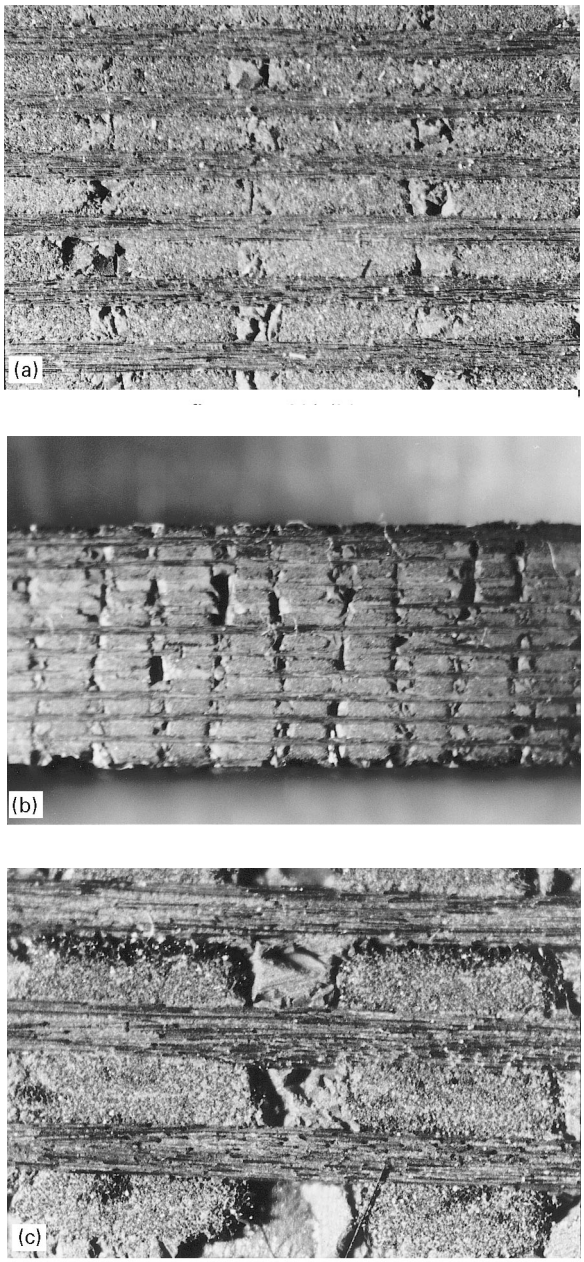

Figure 7 Lateral view of the uncoated test coupons: (a) before fatigue (× 6); (b) after fatigue (× 3, 2); (c) after fatigue (× 12).

fails. Thus, the application of tensile loading to fatigued specimens does not produce additional internal damage up to a certain load level. The load is transferred mainly by the set of fibres parallel to the applied stress direction and the macroscopic linear material behaviour represents the linear elastic behaviour of the SiC fibre reinforcements. More work is required in order to produce a macromechanical model to explain the microscale damage in the case of 3-d SiC/SiC material. The production of such a model is further hampered by the porosity of the sample.

3.3. Microstructural examination

Fig. 7a shows a lateral view of an uncoated test coupon before fatigue. It is easy to distinguish the longitudinal and normal SiC reinforcements as well as the SiC matrix material, which is entrapped within the longitudinal and the normal fibre bundles. No cracks appear in the matrix material. Fig. 7(b and c) show, for two different magnifications, the same lateral view of a fatigued specimen. In both pictures, matrix cracking and fibre–matrix debonding appear. In view of these, the linear behaviour of the fatigued specimens, which exceeded the endurance fatigue limit, could be explained in terms of a fibre dominated phenomenon. Finally, in Fig. 8(a and b) pictures of the fracture surface are given for the cases of an uncoated, fatigue failed specimen. An extensive fibre pull out is observed in both loading and normal to the loading directions, which supports the concept of the wear of the fibre–matrix interfacial zone during the fatigue process.

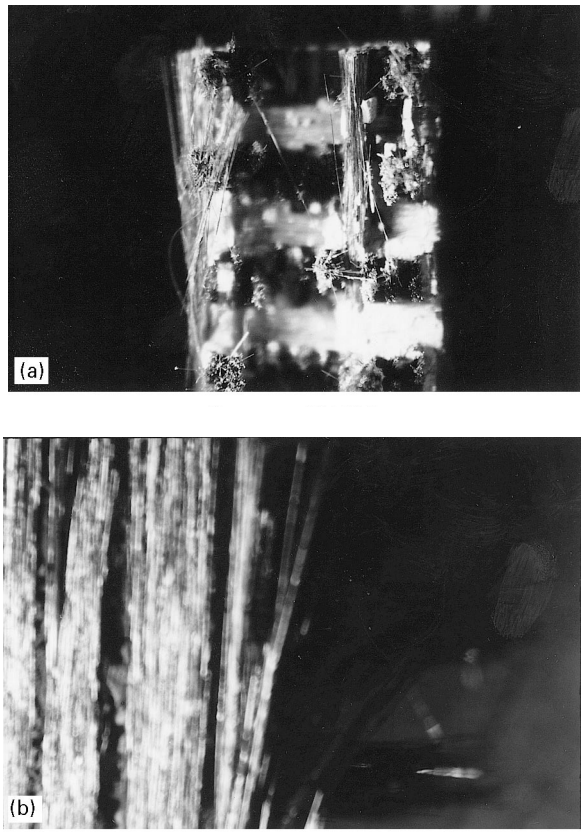

Figure 8 Detail view of the fracture surface of fatigue failed uncoated specimens: (a) ×25; (b) ×100.

4. Conclusions

Tensile and tension–tension fatigue tests were conducted on both coated and uncoated 3-d SiC/SiC composites at room temperature. The main results of this study are as follows:

(i) Both the coated and non-coated 3-d SiC/SiC composites have non-linear stress–strain curves.
(ii) The SiC coating influences the tensile behaviour of the material.
(iii) The SiC coating does not affect the actual maximum stress at which the endurance fatigue limit is approached. In other words the coated material recovers its ability to transfer load during the application of cyclic loading.
(iv) The post-fatigue tensile behaviour of both uncoated and coated materials is identical.
(v) For both materials the design limits should be placed according to uncoated material properties.

A more detailed analysis should address the description of the fracture behaviour of the 3-d SiC/SiC material and its fracture resistance (R-curve). This should also be very helpful for micromechanical modeling of the tensile response of such a material.

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