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Anomalous stress-strain behaviour of CFCCs: an extreme form of scatter

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Anomalous stress–strain behaviour of CFCCs: an extreme form of scatter

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Abstract—Continuous fibre reinforced ceramic matrix composites (CFCCs) show a scatter in parameters such as first matrix cracking stress, initial elastic modulus, ultimate strength, etc. which characterise their mechanical behaviour. This scatter can be attributed to a large extent to variations in the axial residual stress state from specimen to specimen. Under some circumstances even negative strain accumulation with increasing stress has been observed during tensile loading of CFCCs. Examples of such anomalous stress–strain behaviour are shown, and its occurrence is explained by the presence of an axial residual stress state which differs from that induced by the thermal expansion mismatch between the fibres and the matrix. Microstructural features leading to the establishment of such an abnormal residual stress state are indicated. Combining these observations it appears that variation in the residual stress state explains both the scatter observed in tests exhibiting normal behaviour, and in tests with anomalous stress–strain behaviour. The latter is hence only a manifestation of extreme scatter in the axial residual stresses.

Keywords: Continuous fibre reinforced ceramic composite; stress–strain behaviour; scatter; residual stress.

1. INTRODUCTION

Continuous fibre reinforced ceramic composites (CFCCs) contain residual stresses caused by the thermo-elastic mismatch between the fibres and the matrix. These residual stresses superimpose on the applied stress and hence affect the mechanical response. In contrast to particulate- or whisker-reinforced ceramics, where residual stresses average out on the scale of the ceramic matrix grains, in CFCCs they are of longer range and are expected to have a more dramatic effect on the mechanical behaviour. This paper focuses on a particular aspect of the tensile behaviour of

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CFCCs, namely, reverse stress axial strain accumulation under tensile loading, and links it to the scatter in the axial residual stress state.

2. RESIDUAL STRESSES AND SCATTER IN TENSILE RESPONSE

Variation in axial residual stresses has been identified as a source of the observed scatter in mechanical properties of multidirectionally reinforced CFCCs prepared by chemical vapour infiltration (CVI) [1]. In particular, the first matrix cracking stress, the stress for fibre failure and the initial elastic modulus scatter widely between batches and between specimens within a single batch. Because the scatter cannot be explained on the basis of variations in fibre volume fraction and in porosity level, the variability of the axial residual stress state has to be invoked to rationalise the scatter. This approach suffers from the fact, however, that quantification of the residual stresses in CFCCs is difficult and cannot reasonably be done for every test specimen.

Recently, a new method has been proposed to determine the average axial residual stress state in CFCC specimens by performing unloading–reloading cycles during mechanical testing [2]. The coordinates of the common intersection point of the regression lines to consecutive unload–reload cycles represent the average axial residual strain and stress in the fibres (corrected for the volume fraction V_f oriented in the direction of load application). An example for a plain-weave carbon fibre reinforced silicon carbide composite, C(f)/SiC, tested at room temperature is shown in Fig. 1. As expected on the basis of the thermal expansion mismatch between the

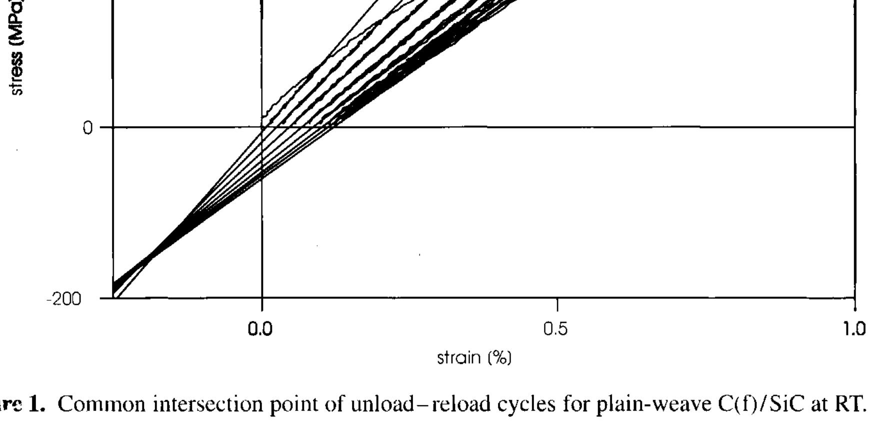

Figure 1. Common intersection point of unload–reload cycles for plain-weave C(f)/SiC at RT.

fibres and the matrix, the average residual strain in the fibres is compressive and compensated by a tensile residual matrix strain.

Comparison of the results obtained by this technique with the residual strains determined experimentally by neutron [3] and X-ray diffraction [4] shows that equivalent results are obtained at room temperature. The results from the loop evaluation also agree with the outcome of the analysis of the same loops by the inverse stress tangent modulus method [5], and with those from finite element calculations, e.g. [6]. Also unloading–reloading presents an experimental advantage in that it can be readily applied at high temperatures.

When the variation in axial residual stress state is taken into account, the scatter in the aforementioned properties is largely reduced, implying that the mechanical behaviour of CFCCs is to a large extent deterministic. Moreover, the temperature dependence of the residual stress state can explain why the mechanical behaviour of the CFCC is temperature dependent, notwithstanding the fact that the strength properties of the fibres and of the matrix do not depend on temperature [1].

3. REPRESENTATION OF THE VARIATION IN AXIAL RESIDUAL STRESS STATE

The coordinates of the common intersection point of the regression lines to consecutive unloading–reloading loops obtained during tensile testing of three CVI-processed CFCCs are shown in Fig. 2. The tests at high temperature have been performed under vacuum in order to eliminate any environmental influence which

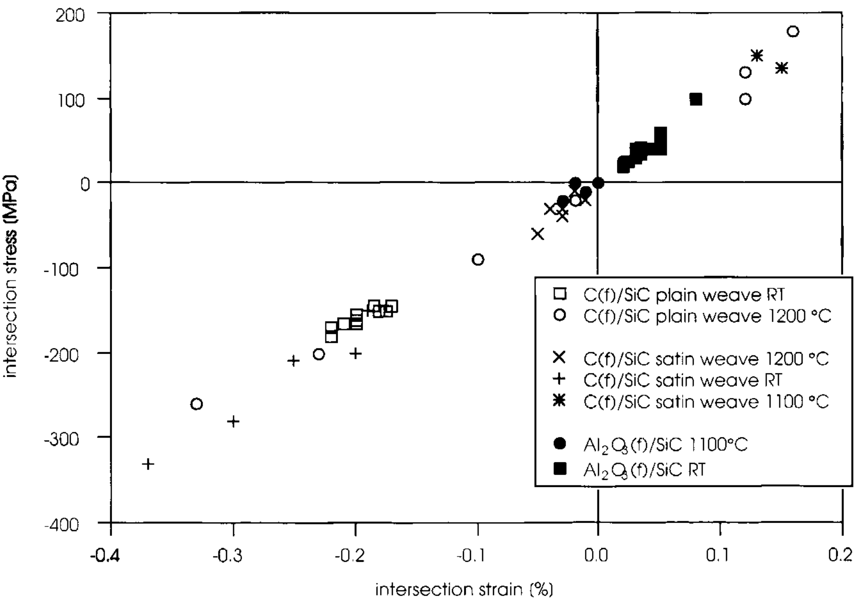

Figure 2. Intersection point coordinates for three investigated CVI-processed CFCCs.

may affect the intrinsic mechanical behaviour. Each point in Fig. 2 represents the axial residual stress and strain in a single test specimen, averaged over the volume of the specimen. For a given CFCC at a given temperature, the scatter in the axial residual stress state is clearly apparent and is obviously expected to induce large variations in the tensile loading response.

At room temperature the residual stress state observed in the three composites, although subjected to a considerable scatter, agrees with that expected from the thermo-elastic mismatch between the fibre and the matrix. However, because the axial residual stresses at high temperature are affected by the amount of interfacial debonding which has occurred during prior cooling from the manufacturing temperature, the residual stress state after reheating to a temperature above the manufacturing temperature does not always correspond to that expected from the thermal expansion mismatch. Indeed, when substantial interfacial debonding has occurred during prior cooling, the fibres and matrix are only loosely bonded and hence only slightly mutually constrained during reheating. Hence, the *magnitude* of the room temperature residual stress state decreases at high temperatures, but its *sign* may be preserved.

4. ANOMALOUS STRESS–STRAIN BEHAVIOUR: PHENOMENOLOGY

In a number of load-controlled tensile tests on the three CVI-processed CFCCs, an anomalous behaviour has been observed when unloading–reloading cycles were applied [7]. This behaviour consists of decreasing strain with increasing stress or of abnormal stress–strain traces upon load reversal, and was observed for all CFCCs over the complete investigated temperature range. Examples are shown in Figs 3 and 4. Extensive verification on the adequate performance of extensometry could not reveal any malfunction. It thus had to be concluded that the reverse axial strain accumulation was an intrinsic material phenomenon.

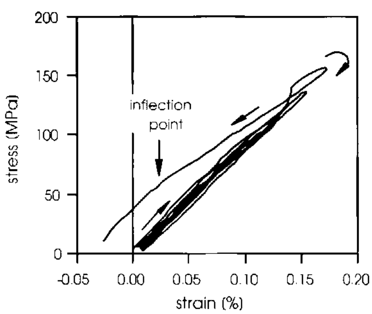

Figure 3. Anomalous stress–strain behaviour in Al_2O_3(f)/SiC at RT.

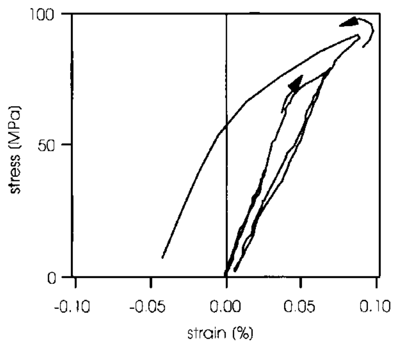

Figure 4. Anomalous stress–strain behaviour in Al_2O_3(f)/SiC at 1100°C.

5. ANOMALOUS BEHAVIOUR: DEVIATION FROM 'NORMAL' RESIDUAL STRESS STATE

When the axial residual stresses from tests with anomalous behaviour are plotted in a stress–strain coordinate system such as that of Fig. 2, it appears that the residual fibre *strains* fall within the range of those corresponding to 'normal' tests, whereas the residual fibre *stresses* are more tensile, and may even be of opposite sign to that expected from the thermal expansion mismatch. An example is shown for the investigated Al_2O_3(f)/SiC composite in Fig. 5. In this respect it is worth noting from Figs 2 and 5 that for normal behaviour the residual stress states for a given CFCC over a range of temperatures can be fitted to a straight regression line through the origin and with a slope which corresponds to the average value of the initial elastic modulus observed for each specimen. This indicates that when normal stress–strain behaviour prevails there is no geometrical constraint to the development of the residual stresses based on the thermal expansion mismatch. The deviation from the regression line for tests exhibiting anomalous behaviour indicates a phase lag between the residual stresses and strains in these tests, i.e. the residual stresses and strains are not exclusively related through the elastic modulus. The reason for this lies in the constraint offered by the microstructure to the free development of the 'normal' residual stress state either during cooling from the processing temperature (anomalous behaviour at room temperature, Fig. 3), and/or during reheating to the test temperature (anomalous behaviour at high temperature, Fig. 4).

For this CFCC the thermal expansion coefficient of the fibres is much larger than that of the matrix. Consequently, after unrestrained cooling down from the manufacturing temperature the matrix is in compression and the fibres are in tension. However, *even in this unrestrained case*, compressive residual stresses can only develop in the matrix regions where the matrix volume fraction is sufficiently high, such as in the matrix deposited at the *outside* of the fibre bundles (*inter*bundle matrix). *Within* the fibre bundles, the matrix volume fraction is too small; the *intra*bundle matrix is therefore under tension, and some matrix cracking is already apparent in the as-received condition [8]. These microcracks extend into the

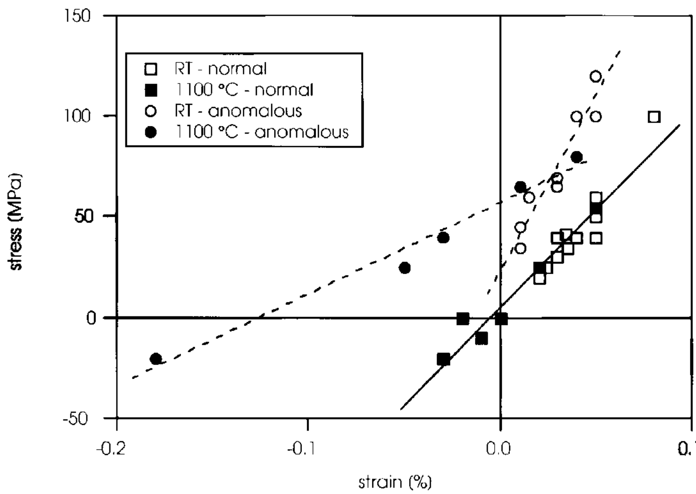

Figure 5. Axial residual stress state for tensile tests on $Al_2O_3(f)/SiC$ specimens showing normal (closed symbols) and anomalous (open symbols) stress–strain behaviour.

interbundle matrix, where they are closed because of the compressive residual stress in the matrix. Upon tensile loading at room temperature, both crack systems open up, allowing unhindered residual stress relief which in turn leads to normal stress–strain behaviour. In the regions where thermal expansion and contraction are unconstrained, heating from room temperature to high temperature causes the interbundle matrix cracks to open, and the compressive residual stress in the matrix as well as the tensile residual stress in the fibres decreases. At high temperature a residual stress state characterised by relatively high axial residual stresses is hence not expected.

Restrained cooling from the manufacturing temperature results in interbundle matrix cracks which are not fully closed at room temperature. Consequently, the bridging fibres experience a higher tensile, and the interbundle matrix a larger compressive residual stress. This shifts the room temperature data points in Fig. 5 upwards. Upon heating, matrix crack opening in the constrained regions is prevented because the matrix blocks between cracks are forced in contact to each other. The residual stress state prevailing at room temperature is hence 'frozen in' upon heating. This causes the axial tensile residual stress in the fibres, respectively compressive residual stress in the matrix to increase in magnitude. As a result, it is even possible for the residual stresses to become of opposite sign of the residual strains, as shown in Fig. 5.

We conclude that anomalous stress–strain behaviour is caused by an anomalous residual stress state which originates from geometrical incompatibilities during cooling. As shown before, also the scatter in mechanical response in tests showing *normal* stress–strain behaviour is caused by the variation in residual stress state. Hence, anomalous stress–strain behaviour is nothing else but an extreme manifestation of scatter.

6. ANOMALOUS STRESS–STRAIN BEHAVIOUR: RESIDUAL STRESS RELIEF

In the previous section, anomalous stress–strain behaviour has been linked to an axial residual stress state different from that based on the thermal expansion mismatch. We now explain how the anomalous behaviour is caused by residual stress relief. During loading, the residual stresses change as a consequence of matrix cracking, of progressive interfacial debonding, and of fibre failure. When the matrix cracks are open *and* the fibres are under residual compression (matrix under tension), the relief causes a contraction of the matrix blocks and an expansion of the fibres bridging the matrix cracks. Consequently, the matrix crack opening increases further, and the relief of residual stress occurs unhindered, resulting in normal tensile behaviour. In the less likely case of open matrix cracks *and* the matrix under *high* average residual compression ('higher than normal' tensile residual stresses in the fibres, Fig. 5), residual stress relief causes an expansion of the matrix blocks and a contraction of the fibres. Because when matrix cracks are open the strain measured on the CFCC equals the fibre strain, stress relief which causes fibre contraction results in a *reduction* of the measured strain, even under increasing tensile stress. Upon unloading, an inflection point then appears because the matrix blocks touch each other, whereas it was not present before. Figure 3 shows an example of such anomalous behaviour.

When matrix cracks are closed the strain measured on the composite is equal to the matrix strain. When in this case the axial residual stress in the matrix is compressive, residual stress relief would cause an expansion of the matrix blocks and a contraction of the fibres. Because this is geometrically impossible, stress relief cannot take place and the magnitude of the residual stress may even increase (which may result in tensile fibre failure). Upon unloading, because matrix blocks are touching, unloading occurs initially with the same tangent modulus as the previous loading. This shifts the unloading curve to the left and negative strain accumulation is again observed, as shown in Fig. 4. At the minimum load reversal, matrix cracks are still closed, and hence the unloading and reloading moduli are also identical, causing the reloading path to lie below the unloading path (counterclockwise hysteresis). In the less likely case of closed matrix cracks *and* matrix under residual tension, residual stress relief reduces the amount of crack closure. When cracks remain closed, a similar situation as shown in Fig. 4 is obtained. When stress relief induces (partial) crack opening the strain measured on the CFCC switches from that corresponding to the matrix strain to that of the bridging fibres which want to expand. This results in a 'fast' forward strain accumulation. Upon load reversal, the unloading path then cuts the previous loading curve, and anomalous strain accumulation is observed.

7. CONCLUSION

The conditions for anomalous stress–strain behaviour are that the residual stress state differs from that based on the thermal expansion mismatch, and the presence of a favourable matrix cracking state. Both are affected by microstructural features which hinder the relative movement of fibres and matrix and thus obstruct the development of the equilibrium stress–strain distribution between the fibres and the matrix. In view of the complex microstructure and reinforcement architecture of current CFCCs, anomalous stress–strain behaviour may be more than just a case of exceptional behaviour.

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