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The importance of damage and slow crack growth in the creep behavior of ceramic matrix composites

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Abstract—Three ceramic matrix composites (CMCs) were investigated after creep tests: a 2D SiC_f–MLAS, a 2D SiC_f–SiC and a 2.5D C_f–SiC. The damage features of each one are identified, using optical and scanning electron microscopies (SEM), and the complete damage sequences are presented. As a result of its time-dependence, damage in CMCs may be considered as slow crack growth in the temperature and stress fields investigated. But the quantification of damage, through the classical damage mechanics, appears as a complex issue, due to the architecture effects in composite materials.

Keywords: Damage; creep; CMCs; slow crack growth; architecture effects.

1. INTRODUCTION

The classical creep mechanisms of metals and ceramics result in a plastic deformation mainly due to flows of vacancies and motions of dislocations. Creep can then involve diffusion of atoms through the volume or along the grain boundaries, grain sliding by shear along the grain boundaries through diffusion processes, or climb or glide dislocation [1, 2]. But all these mechanisms are thermally activated. So, if the temperature is too low, none of them can be activated. Then, the only way for the material to creep is to develop an array of microcracks through different damage mechanisms. Such mechanisms are especially encountered in ceramic matrix composites (CMCs) due to their highly refractory properties. For these materials a so-called 'damage-creep' mechanism has been proposed, which involves damage mechanisms accompanied sometimes by classical creep mechanisms [3, 4]. Therefore, using CMC parts for industrial applications is a challenging issue, as it is required to control or/and to heal the microcracks and their growth.

This paper highlights the role of damage during creep tests for three different CMCs: one with a glass–ceramic matrix (2D SiC_f–MLAS) and two with a ceramic one (2D SiC_f–SiC and 2.5D C_f–SiC).

2. MATERIALS AND EXPERIMENTAL DEVICE

The 2D SiC_f–MLAS (magnesium lithium alumino silicate) composite is fabricated by Aérospatiale Company. Nicalon NLM 202 SiC fibers are embedded in a slurry containing a glass powder (0.5 MgO–0.5 Li_2O–1 Al_2O_3–4 SiO_2) obtained by a sol–gel route. The cross-ply $(0–90)_6$ composite is obtained by hot pressing. A double 90° ply is located in the middle of the specimen (Fig. 1).

Both silicon carbide matrix composites (i.e. 2.5D C_f–SiC and 2D SiC_f–SiC) are fabricated by SEP, Division de SNECMA, according to the chemical vapor infiltration process [5]. For the 2D SiC_f–SiC composites, woven Nicalon NLM 202 SiC fibers were used, while for the 2.5D C_f–SiC composites, the weaving involves high strength ex-PAN carbon fibers.

Creep tests were performed in three-point-bending for 2D SiC_f–MLAS (1273–1473 K, vacuum), on small specimens ($20 \times 5 \times 2.5$ mm^3), and in tension for 2D SiC_f–SiC and 2.5D C_f–SiC (1273–1673 K, argon), on large specimens (200 mm long, 15 mm width in the gauge section and 3 mm thick).

Damage characterization was achieved by optical and scanning electron microscopies (SEM), using, respectively, an Olympus BH2-UMA microscope (Tokyo, Japan) and a Jeol JSM 6400 microscope (Tokyo, Japan).

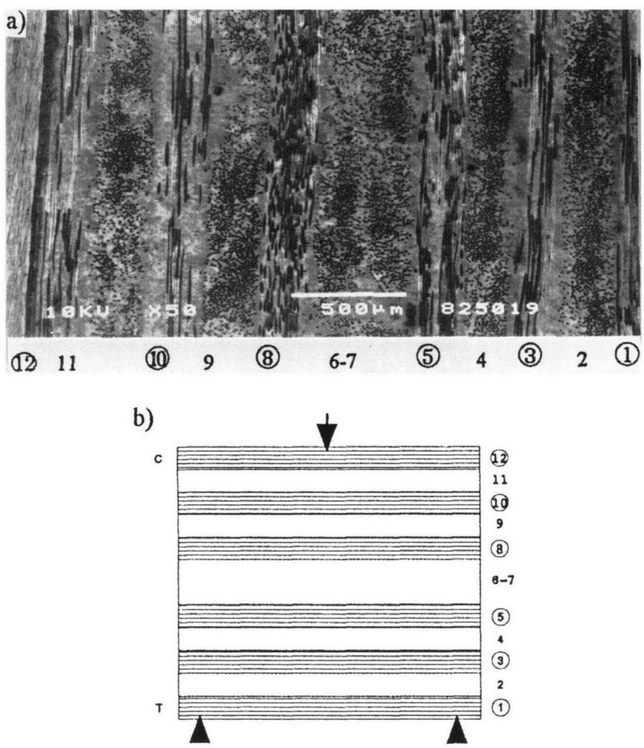

Figure 1. (a) SEM micrograph of a as-received 2D SiC_f–MLAS specimen; (b) nomenclature of the different plies.

3. DAMAGE DURING CREEP TESTS

In the temperature fields investigated for the different materials of this study (1273–1673 K), it can be considered that there is no creep of the fibers or the SiC matrix. Nevertheless, for 2D SiC_f–MLAS composites, tested at the highest temperatures (i.e. 1373–1473 K), a viscous flow of the glass–ceramic matrix may be encountered. Therefore damage mechanisms are very likely to occur.

3.1. 2D SiC_f–MLAS composites

An analysis of the state of internal stresses, using finite elements modelling, has revealed that shear stresses dominate within the specimen when a flexural load is applied [6, 7]. The areas of pure tension and compression are very small. In the area of compression a remarkable absence of cracks is noticed. In the meantime, perpendicular microcracks are observed in the area in tension. When temperature and stress are increased, the opening of these cracks increases, while the spacing distance between cracks decreases.

In fact, most of the damage in bending creep of 2D SiC_f–MLAS composites is due to shear microcracks as observed in Fig. 2. Following damage evolution from the as-received state, until the fractured state, the chronology of damage upon creep tests has been settled. The double 90° ply in the middle of the specimen (ply 6–7)

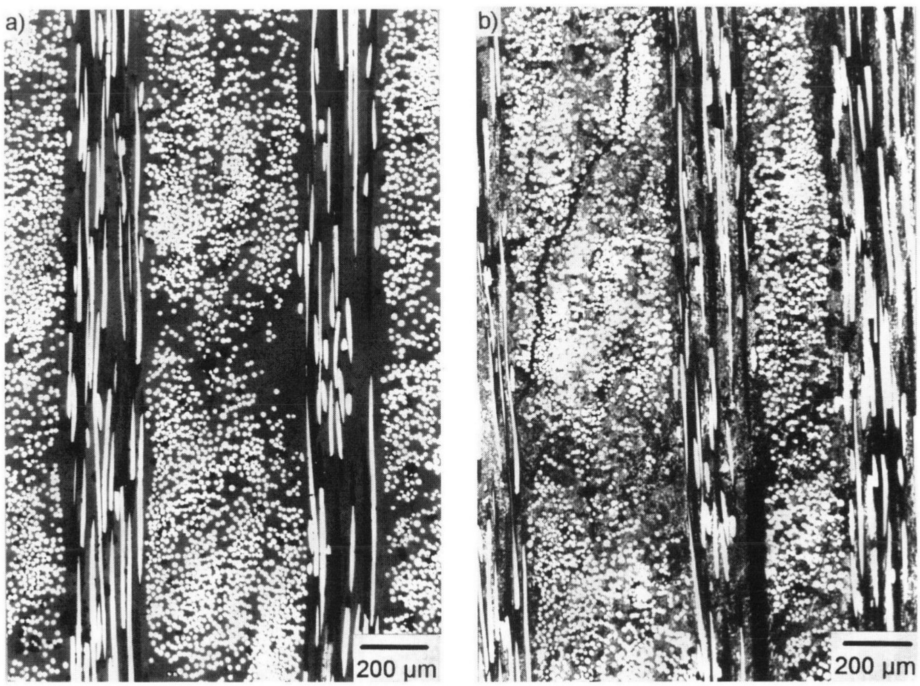

Figure 2. (a) Optical micrographs of a 2D SiC_f–MLAS specimen in the as-received state; (b) creep tested in bending, under vacuum, at 1373 K, under 220 MPa (same area as in (a)).

is affected first. Then, symmetrically, plies 4 and 9 (90°) are concerned, followed by plies 2 and 11 (90°). At last, when all these 90° plies are fractured, the 0° plies become affected also, leading to the tertiary creep stage and the rupture of the specimen [8].

3.2. 2D SiC_f–SiC composites

In the case of 2D SiC_f–SiC composites creep tested in tension, only some transverse microcracks (i.e. perpendicular to the loading direction) are observed. At the edge of the specimen, the microcracks initiate at the macropores and progress through the thickness of the sample [9]. When the saturation of the cracks density is achieved, the opening of these matrix microcracks occurs, due to fibers and bundles/matrix interfacial sliding. Bundles and fibers bridging the matrix microcracks are observed (Fig. 3), which induces load transfer from the matrix to the fibers. When the reloading of the fibers is too high, as opening of the matrix microcracks goes on, the fracture of the specimen takes place with large fiber pull-out length [10].

3.3. 2.5D C_f–SiC composites

For 2.5D C_f–SiC composites, as for 2D SiC_f–MLAS composites, the evolution of damage upon creep tests has been followed from the as-received state until rupture

Figure 3. SEM micrograph of a 2D SiC_f–SiC specimen creep tested in tension, until rupture, under argon, at 1373 K, under 125 MPa.

on both the edge and the surface of the samples (Fig. 4) [11]. From a microstructural point of view the difference between 2D SiC_f–SiC and 2.5D C_f–SiC composites, in the as-received state, lies in the presence of pre-existing transverse microcracks

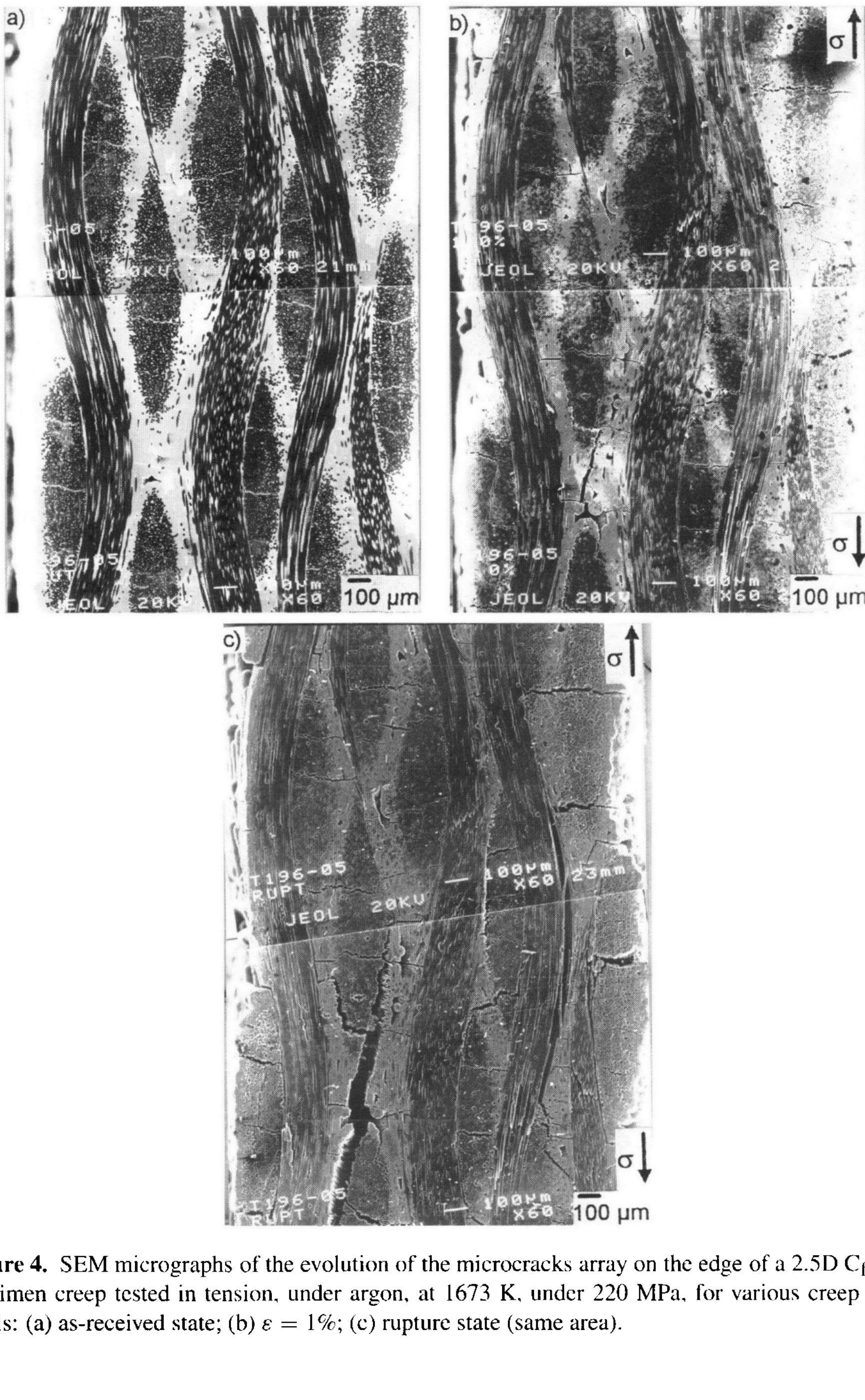

Figure 4. SEM micrographs of the evolution of the microcracks array on the edge of a 2.5D C_f–SiC specimen creep tested in tension, under argon, at 1673 K, under 220 MPa, for various creep strain levels: (a) as-received state; (b) $\varepsilon = 1\%$; (c) rupture state (same area).

in 2.5D C_f–SiC composites, due to the coefficient of thermal expansion mismatch between the fibers and the matrix [12].

During creep tests, the opening and the development of the pre-existing transverse microcracks are responsible for a first damage accumulation [13]. Then, the initiation of interply cracks, parallel to the loading direction, is observed at the macropores. After connection between interply cracks, the opening of both transverse and interply microcracks occurs until rupture.

Considering the surface of the specimen, the formation and the opening of transverse and longitudinal microcracks have been observed too, which gives a better idea of the volumic damage.

Then, a first attempt of damage quantification, using automatic image analysis, has been performed, only considering the transverse microcracks on the edge of a specimen. Using a specific algorithm involving some classical functions of the mathematical morphology (threshold, skeleton, pruning and reconstruction), the occurrence of the opening of the pre-existing transverse microcracks has been confirmed quantitatively [14].

4. DISCUSSION

These three examples highlight that 'damage-creep' is a well-suited concept for the creep mechanism of CMCs, as damage brings a major contribution to the macroscopic creep strain in the temperature and stress fields investigated.

Nevertheless, the complex development of the microcracks arrays appears to be time-dependent. Thus, lifetime prediction can be based on the damage state of the material by considering the microcracking pattern and its evolution during creep. For example, in 2D SiC_f–MLAS composites, the damage state can be evaluated by the presence and the opening of shear cracks in each 90° ply. Nevertheless, this example underlines also the complex influence of the composite architecture on the development of matrix microcracking.

Moreover, the case of 2.5D C_f–SiC composites confirms the importance of such an effect. In fact, five modes of matrix microcracking have been identified in these composites:

- transverse cracks in the transverse bundles (pre-existing cracks);
- transverse cracks in the longitudinal bundles;
- longitudinal cracks in the longitudinal bundles (pre-existing cracks);
- longitudinal cracks in the transverse bundles;
- interply cracks.

Considering these different types of cracks, straightening of the longitudinal bundles parallel to the loading direction is admitted to be the driving force for damage, as proposed by Shuler *et al.* (Fig. 5) [15]. Such a straightening induces the initiation of interply microcracks and the subsequent appearance of flexural forces in the transverse bundles, leading to a further opening of the pre-existing transverse microcracks and the formation of longitudinal cracks in the transverse bundles, observed at the surface of the tested specimen [16].

As a result, such distortions in the architecture of the composite can dramatically affect the damage parameter, D, defined by Kachanov as [17]:

$$D = 1 - E_t/E_0,$$

where E_0 is the elastic modulus of the as-received material and E_t the elastic modulus of the damaged material at a time t.

In fact, the evolution of the damage parameter for a 2.5D C_f–SiC composite tested at 1673 K under 220 MPa (Fig. 6) is not, of course, in agreement with the observations made on the tested specimen, which testify of a continuous damage evolution (Fig. 4). But after the first damage accumulation, the reduction of the damage parameter is attributed to the architectural distortions, which prevent microcracks from closing upon unloading, leading to a reduction of the elastic

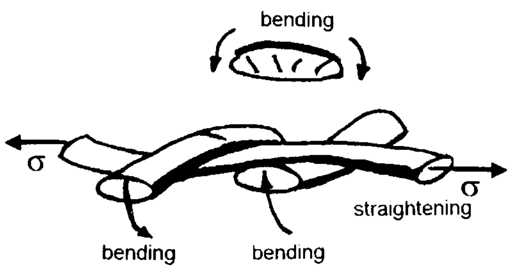

Figure 5. Illustration of the matrix microcracking induced by straightening of the longitudinal bundles parallel to the loading direction and the subsequent bending forces in the transverse bundles (after Shuler *et al.* [15]).

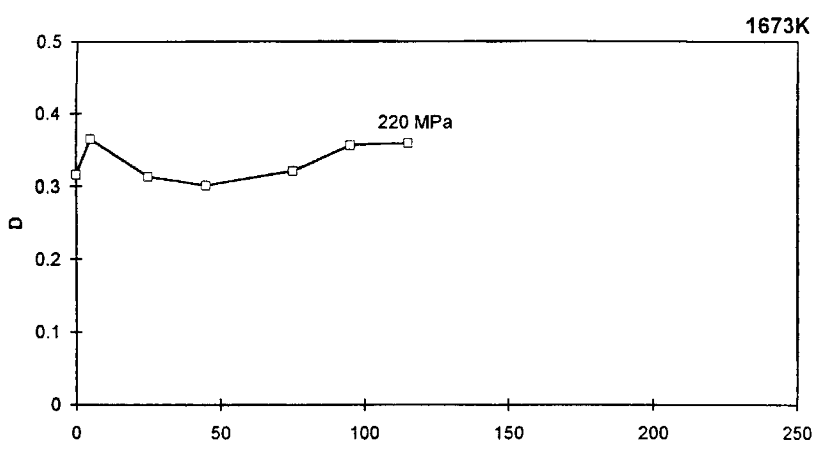

Figure 6. Evolution of damage as a function of time, $D = f(t)$, during creep test of a 2.5D C_f–SiC composite tested, under argon, at 1673 K, under 220 MPa.

strain and the subsequent apparent stiffening of the composite [18]. The second damage accumulation can result from the large architectural distortions achieved at that point, which are partly reversible and lead to a new increase of the elastic strain and the damage parameter.

Therefore, Kachanov's parameter, D, does not seem to be a well-suited indicator for damage in materials with so important architecture effects, such as in CMCs. However, the evolution of D as a function of time illustrates the time dependence of damage, which may be considered as slow crack growth (see Fig. 2). To further investigate this hypothesis towards modelling of the creep behavior of CMCs, a more accurate and realistic damage indicator has to be used and can be brought by quantitative automatic image analysis.

5. CONCLUSION

This paper illustrates the reality and the importance of damage in the creep behavior of three different CMCs. The damage processes observed are time dependent and, subsequently, can be considered as slow crack growth mechanisms. The controlled evolution of these damage processes can lead to lifetime prediction, as far as the development of microcracks can be followed by a well-suited damage indicator. Due to the presence of architecture effects in CMCs, Kachanov's damage parameter, defined for isotropic and homogeneous materials, cannot be used successfully. Damage quantification by automatic image analysis appears as a promising route.

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