

# **FAILURE OF 2.5D C<sub>f</sub>-SiC COMPOSITES CREEP TESTED IN TENSION**

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**SUMMARY:** This paper concerns the creep behavior of 2.5D C<sub>f</sub>-SiC composites. Creep tests have been performed in tension under a ‘low argon pressure at 1273K-1673K between 110 and 220 MPa. From the evolution of the damage parameter vs time, a damage creep mechanism can be assumed. In order to identify such a mechanism, a microstructural investigation has been performed at different scales (by SEM and HREM). Under high testing conditions ( $T>1473K, \sigma>160\text{ MPa}$ ), it appears that the microcrack array, composed of five types of matrix microcracks, is settled upon loading and during the transient stage. During the steady state, the evolution of the damage is mainly due to the opening of these microcracks. So, the lifetime of the ceramic matrix composite will depend mostly on the ability of the interphase to protect the fibers, in other words its capability to deflect microcracks. At the microscopic scale, different types of inter-facial sliding have been evidenced: at 1473K a dry friction between two rough solids, and at 1673K a viscous flow.

**KEYWORDS:** ceramic matrix composite, tensile-creep, matrix microcracking, damage, fiber/matrix interphase, fiber bridging, inter-facial sliding, fracture, lifetime, 2.5DC<sub>f</sub>-SiC.

## **INTRODUCTION**

Ceramic matrices reinforced with long ceramic fibers - ceramic matrix composites, CMCs - are a class of materials with potential long term applications in the field of 1273-1473K under high stresses, compared to superalloys (1-3). Many different architectures are developed from different ceramic fibers to obtain specific directional reinforcement or/and to avoid a certain delamination.

This paper deals with the creep behavior of a woven 2.5D C<sub>f</sub>-SiC material which has been studied in our laboratory using multiscale investigations and different approaches (4).

## **MATERIALS AND EXPERIMENTAL DEVICES**

The material under investigation is a C<sub>f</sub>-SiC composite fabricated by chemical vapor infiltration (CVI) of a pure SiC matrix within a 2.5D preform of high strength ex-PAN carbon fibers (SEP

Division de SNECMA, Saint Médard en Jalles, France), (5). The so-called 2.5D architecture corresponds to a stack of five plain woven clothes with a certain interlocking between them. A pyrocarbon interphase has been deposited on the fiber prior to SiC infiltration. A micrograph of such a material is presented in Figure 1.

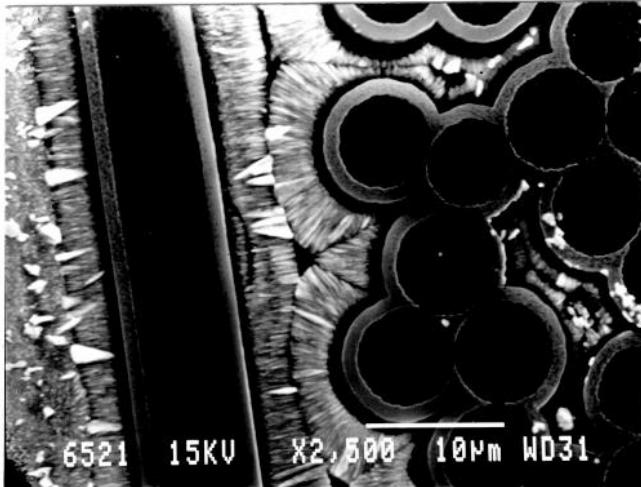


Fig. 1 : SEM micrograph showing the different phases in a 2.5D C<sub>f</sub>-SiC composite.

High temperature thermomechanical characteristics published on ceramics and ceramic matrix composites are often very dispersed. That can be due not only to the well-known statistical dispersion of the intrinsic defects in ceramics, but also to the fact that experimenters are not careful enough with some experimental and technical errors which lead to biased results. For example, some published results of lifetime prediction for silicon nitride can vary over one order of magnitude for measurements performed on exactly the same material and experimental conditions (6) which cannot be only attributed to the heterogeneity of the material and/or the presence of defects.

From the experience in our laboratory, we made technological choices in order to develop and optimize a high temperature testing device, based on a critical study of the different possible parts involved (7) : load frame alignment, thermal gradient and stability in the furnace, thermocouple position and temperature measurements, pressure fluctuations and strain measurements.

Tests were performed with an hydraulic Schenck PSB 100 machine, equipped with an airtight fence (AET) and two hydraulic grips (Instron). The dog-bone tensile specimens (200 mm long, 25 mm broad, 15 mm broad in the gage, and 3 mm thick) were heated by an induction furnace (Célés) with a graphite susceptor. Temperature was measured with 2 WRe 5/26 thermocouples, and deformation from two mechanical contact extensometers (Schenck) with SiC arms (Fig. 2).

Tests were conducted in tension under argon partial pressure (50 mbar) for temperatures between 1273 and 1673K, at stress levels of 110 and 220 MPa.

A Jeol JSM 6400 scanning electron microscope (SEM) was used for damage characterization. Transmission (TEM) and high resolution (HREM) electron microscope characterizations were performed to investigate changes in the different phases and the inter-facial features at the fiber/matrix interface, using three microscopes operating at 200 kV: a Jeol 200 CX, a Jeol 2010 and a Topcon EM 002B, all equipped with EDX analysis (Link). For such localized investigations, disc shaped samples ( $\varnothing = 3\text{mm}$ ) were cut from the 2.5D C<sub>f</sub>-SiC specimens, then

mechanically ground and dimpled to a  $\sim 10 \mu\text{m}$  thickness. The final thinning was achieved by ion-milling (Ar', 5kV).

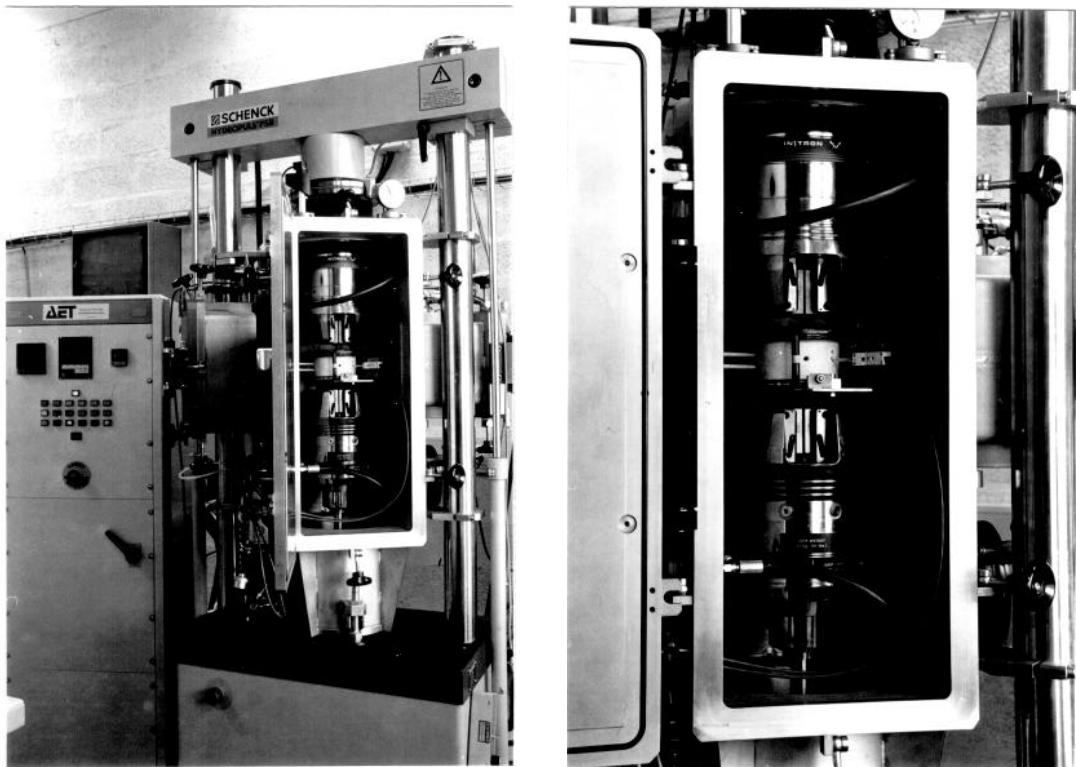


Fig. 2 : Creep equipment used for the 2.5D C<sub>f</sub>-SiC samples.

## RESULTS

Some strain-time creep curves,  $\epsilon$ -t, are presented in Figure 3 (specimen ruptures are indicated by vertical arrows). The noise-free shape of the curves underlines the quality of the creep experiments. The undulations observed time to time are due to unloading-reloading cycles in order to follow the change in the elastic moduli.

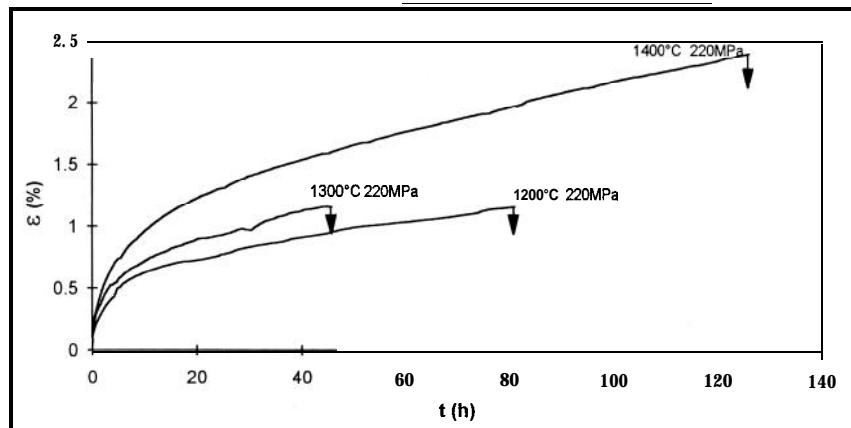


Fig. 3 : Strain-time curves,  $\epsilon$  - t, for 2.5D C<sub>f</sub>-SiC specimens creep tested in tension at different temperatures.

These curves present a short (some tens hours) transient state, followed by a correct stationary state where specimen fracture can appear. In no case, a tertiary state is observed which is a simple indication of the ruin of the material.

Creep starts at temperature as low as 1273K, even though it is very small. It can be seen that at 1473K and under 220 MPa strain and time to rupture are much lower than at 1673K for the same stress conditions. That can perhaps presume the existence of different mechanisms which must be confirmed at the microscopic scale.

Dorn's formalism was used to plot creep rate as a function of temperature,  $\dot{\varepsilon} = 1/T$ , even though composite materials do not confirm the required hypotheses of homogeneity and isotropy. Therefore, the data obtained for the stress exponent (i.e.  $n = 1.8-2$ ) and the activation energy (i.e.  $Q \sim 60-80 \text{ kJ.mol}^{-1}$ ) are not exploitable in terms of creep mechanism of the material and cannot be related to the values for the components (carbon fibers and chemical vapor deposited silicon carbide), (8-10). As a consequence another approach has been conducted through the damage mechanics as proposed by Kachanov (11) and Rabotnov (12) in order to identify the mechanism(s) responsible for creep strain.

In a first approach of damage quantification, the damage function D was used, as  $D = 1 - E_t/E_0$  (with  $E_t$  the bulk elastic modulus of the composite at time t, and  $E_0$  the initial modulus of the undamaged composite).  $E_t$  was determined from periodic unloading-reloading loops during the creep tests. Damage curves as a function of time, D - t, are presented in Figure 4. From the evolution of the damage parameter observed: a damage creep mechanism can be assumed (13, 14). To confirm such an hypothesis, a microstructural investigation has to be performed at different scales from the macroscopic one to the nanoscopic one (4).

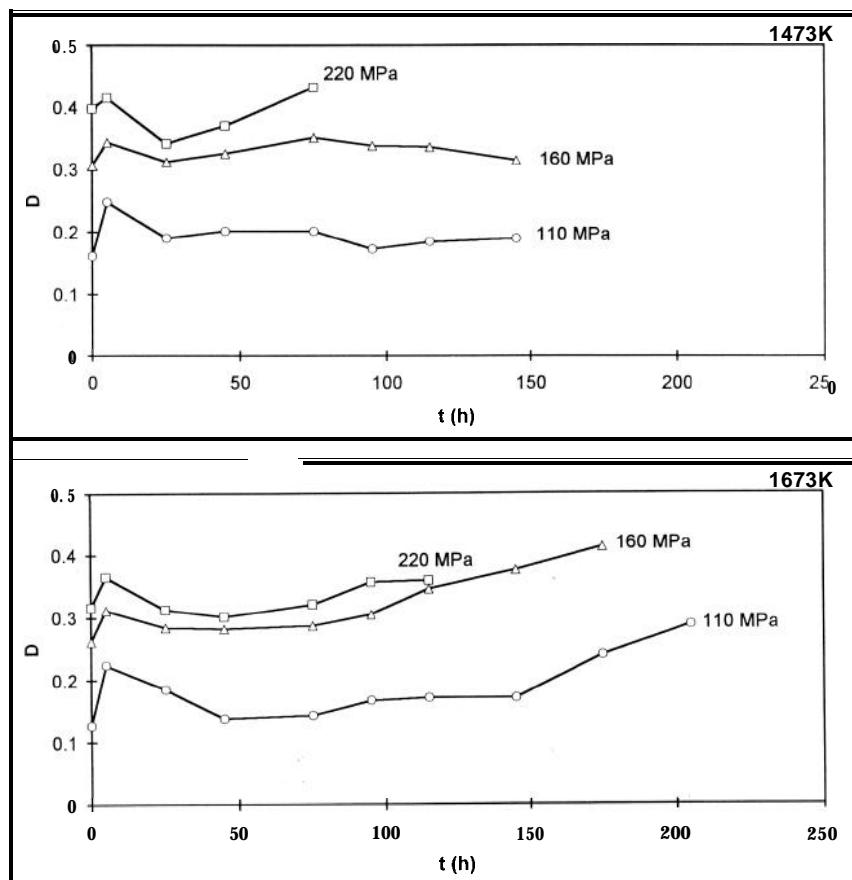


Fig. 4 : Damage curves as a function of time, D - t, for 2.5D C<sub>f</sub>-SiC tensile creep specimens tested at different temperatures and stresses.

## DISCUSSION

The observation of the creep tested specimens reveals, in fact, many damages, such as fiber/matrix debonding, matrix microcracking, fiber bridging, fiber fracture. Figure 5 testifies to effective damage mechanism through matrix microcracking. Following the development of the microcrack array via SEM, it appears that this array is settled upon loading and during the transient stage as far as the testing conditions are high enough ( $T \geq 1473K$  and  $\sigma \geq 160 MPa$ ). Nevertheless at that mesoscopic scale, five types of matrix microcracks are evidenced : either parallel or perpendicular to the loading direction in longitudinal and transverse bundles, as well as interply cracks. Considering the microcrack architecture, one assumes, as proposed by Shuler et al. (15), that the straightening of the longitudinal bundles parallel to the loading axis is the driving force for microcracking.

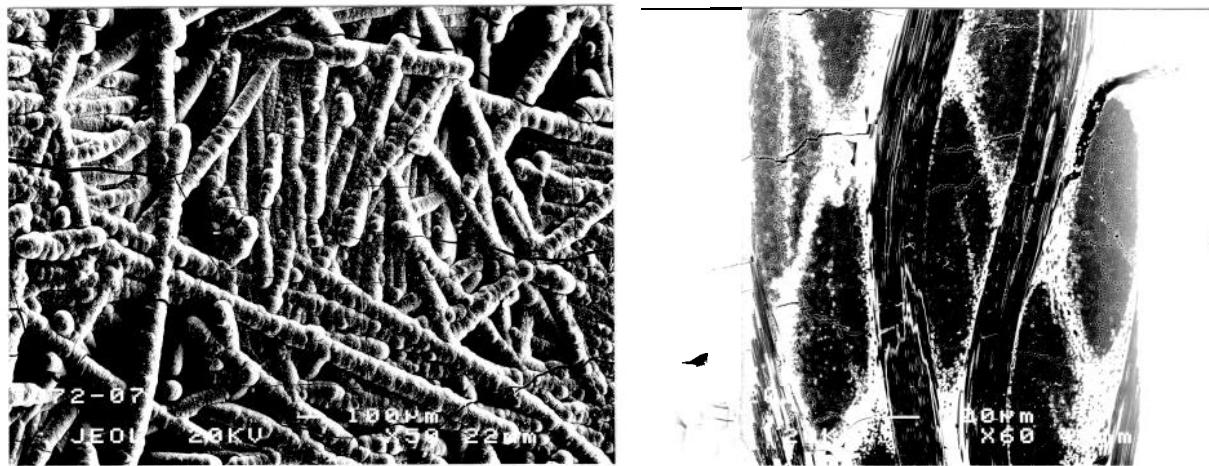


Fig. 5 : Damage observed during creep tests of 2.5D C<sub>f</sub>-SiC specimens.

At this point it can be considered that most of the applied stress is transferred from the matrix to the fibers at the end of the transient stage. Thus in the steady state one observes mainly the opening of the matrix microcracks (Figure 6). As a consequence the lifetime of this CMC seems to depend mostly on the ability of the pyrocarbon interphase to avoid any notch effect on the fibers and the subsequent rupture, in other words its capability to deflect microcracks. That can only be highlighted by TEM observations and analysis.

Through such an investigation at the microscopic scale, on the fracture surfaces, two types of interfacial sliding have been evidenced, only depending of the test temperature (16, 17) : i) at 1473K a dry friction between two rough solids (Figure 7a) ; ii) at 1673K a viscous flow (Figure 7b).

These interfacial characteristics are in agreement with the damageable behavior evidenced at 1473K and the viscoplastic behavior at 1673K.

In sufficiently high creep conditions ( $T \geq 1473K$  and  $\sigma \geq 160 MPa$ ), the stationary stage is governed by microcrack opening controlled by inter-facial sliding, which can be considered as a slow crack growth. This leads to an increasing reloading of the fibers until a threshold value, corresponding to the rupture.

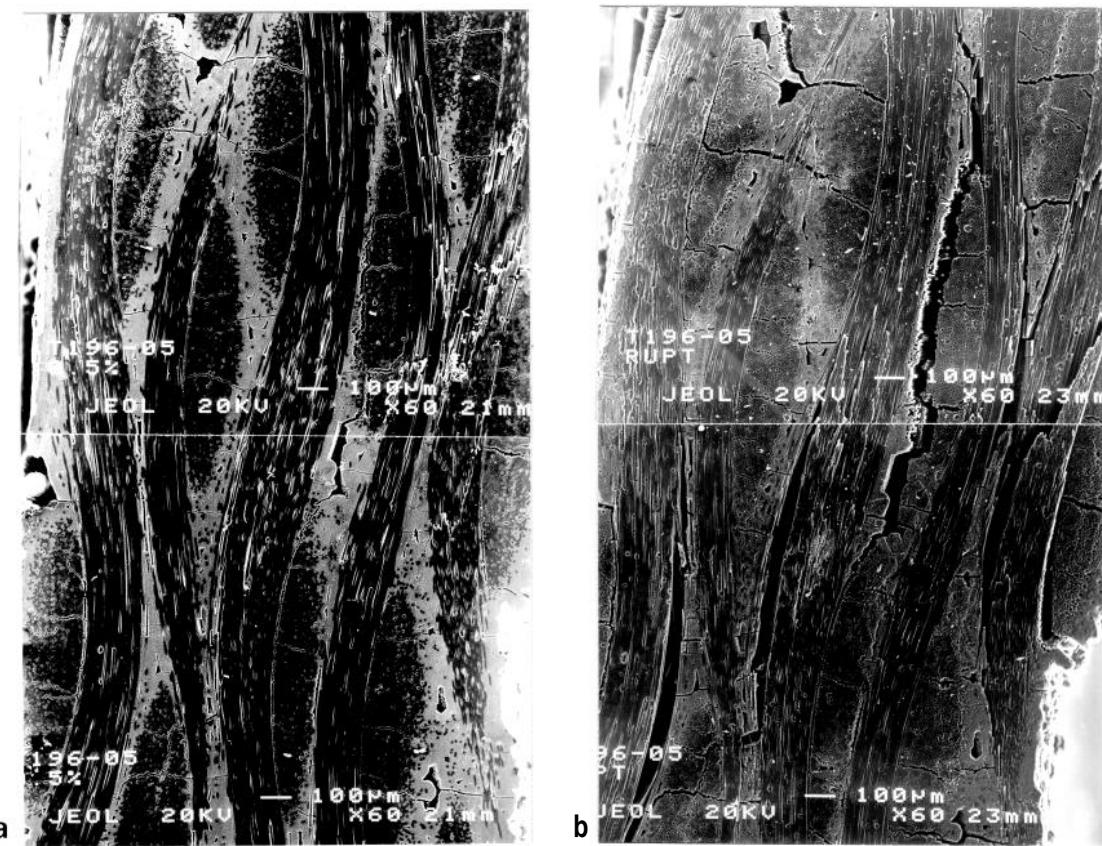


Fig. 6 : Opening of some matrix microcracks in a 2.5D Cr-SiC specimen tensile creep tested at 1673K under 220 MPa: a)  $\epsilon = 0.5\%$  ; b) rupture. The two micrographs correspond to the same area.

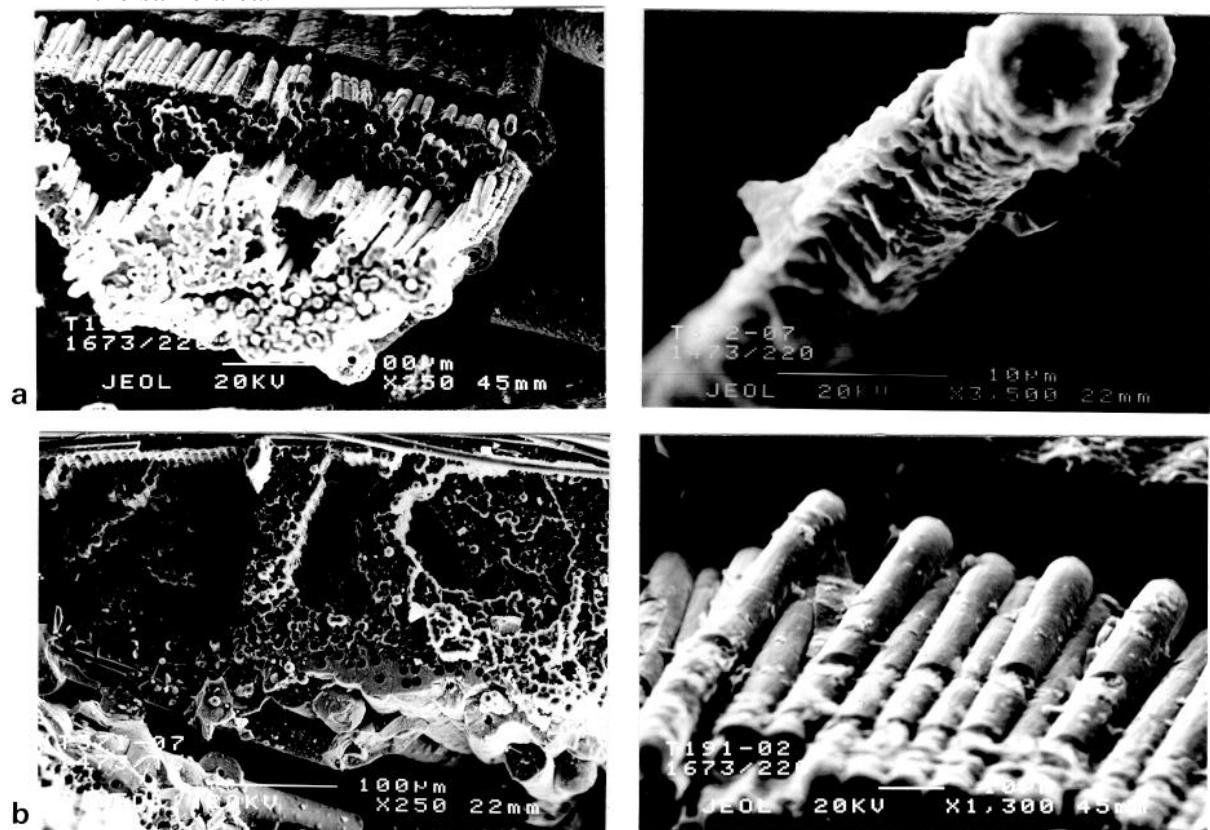


Fig. 7 : SEM micrographs of 2.5D Cr-SiC specimens creep tested at : a) 1473K and b) 1673K.

As a result of this discussion the lifetime of the 2.5D Cr<sub>x</sub>SiC composite may be considered rather as function of a strain to failure. When this threshold strain is reached the catastrophic rupture of the composite occurs which leads to the absence of a tertiary stage as observed on strain-time curves (Figure 3).

## CONCLUSION

This ceramic matrix composite presents a good creep resistance compared to classical metallic materials. In the experimental field investigated, the creep is only controlled by a damage creep mechanism, as it has been confirmed not only by the evolution of the damage parameter, but also by the mesoscopic and microscopic observations.

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## CAPTIONS

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Fig. 2 : Creep equipment used

Fig. 3 : Strain-time curves,  $\varepsilon - t$ , for tensile creep tested specimens of 2.5D C<sub>f</sub>-SiC at different stresses and temperatures.

Fig. 4 : Damage curves as a function of time, D - t, for 2.5D C<sub>f</sub>-SiC tensile creep specimens tested at different temperatures and stresses.

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