

CREEP OF A C/C-SiC COMPOSITE UNDER INTERLAMINAR SHEAR LOADING AT HIGH TEMPERATURES

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SUMMARY: A laminated fiber-reinforced ceramic was investigated with respect to possible creep effects. Because creep effects, if at all, were expected to be caused by the matrix properties, a predominant interlaminar shear loading was applied. It was realized by the compression-shear-test using unsymmetrically grooved specimens. First indications for interlaminar creep were found above 1450°C. At 1800°C and loads of 60% and 70% of the failure load nonlinear deformations can be measured. In order to check whether the properties of the test material have changed due to creep loading, additionally, the residual strengths for the interlaminar shear (ILSS) were determined at the temperature considered and the structure of the material was investigated microscopically in the states before and after thermomechanical loading.

KEYWORDS: C/C-SiC, High Temperature Testing, Creep, Interlaminar Shear Strength, ILSS

INTRODUCTION

The C/C-SiC material investigated belongs to the class of Ceramic Matrix Composites (CMC) which show advantageous thermomechanical properties, e.g. the strength values of these materials increase at higher temperatures [1, 2].

Up to now, to the authors' knowledge, high temperature strength measurements are usually performed under short term quasistatic loading conditions. In this study investigations are presented which were carried out under constant static loading for longer times similar as it was reported recently for a pure C/C-material [3]. Creep deformations were measured under *interlaminar shear* loading. This approach was primarily chosen because the shear failure of C/C-SiC is predominantly determined by the matrix strength. At high temperatures the matrix also behaves rather like ceramics than the fibers so that under shear loading the highest creep effects has to be expected. Additionally, these investigations could give first indications which might explain the excellent mechanical properties of C/C-SiC at high temperatures. This expectation is based on the fact that the strength and the failure behaviour of anisotropic and heterogeneous fiber-reinforced composites are influenced in general and under all mechanical loading conditions by the properties of the weakest links (matrix, interfaces). Possible explanations for the improvement in strength of C/C-SiC with increasing temperature might be for example: an increasing

insensitiveness of the material-inherent cracks to stresses caused by toughening of the matrix, and/or thermal induced structural changes or crack closure mechanisms.

TEST MATERIAL

The C/C-SiC composite considered is manufactured by the Liquid Silicon Infiltration (LSI) process, which was developed for the fabrication of Ceramic Matrix Composites of structural aerospace applications. Heat shields for reentry capsules, intake flaps for hypersonic aircraft and brake discs for light weight automotive cars have already been successfully realized.

Basically, the LSI process consists of three manufacturing steps [4]. Within the first step a green body of Carbon Fibre Reinforced Plastic (CFRP) is formed. Commercially available woven fabrics of high tenacity fibres (AKZO=s HTA) and one-part thermosets (XP-60) as precursors have been used to fabricate 2D-laminates with a fibre volume content of about 60 %.

After curing, the CFRP composite is pyrolyzed at 900 °C in nitrogen in a second step, leading to a porous carbon/carbon (C/C) preform with a pattern of translaminar cracks. In the third step, a silicon carbide matrix is formed by infiltrating liquid silicon into the C/C preform at temperatures above the melting point of silicon (1420 °C) under vacuum. The infiltration is accompanied by a simultaneous chemical reaction between silicon and carbon to form SiC, whereby the silicon preferably reacts with the amorphous carbon matrix.

The LSI process leads to C/C-SiC materials consisting of load carrying carbon fibres and matrices of silicon carbide, carbon and some free silicon (s. Fig. 1). Generally, these composites are characterized by a low density ($< 2 \text{ g/cm}^3$), low thermal expansion, high thermomechanical properties and an extreme thermoshock resistance. In comparison to commercially available carbon/carbon materials the C/C-SiC composites show a low amount of porosity ($< 5 \%$) and a higher oxidation stability due to the internal SiC matrix.

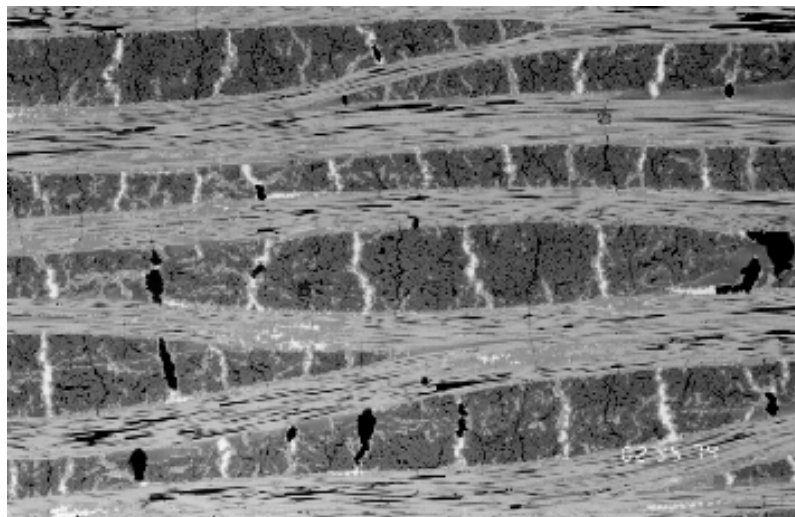


Fig. 1: Microstructure of the C/C-SiC considered (provided by DLR/Stuttgart)

CREEP EXPERIMENTS AT HIGH TEMPERATURES

Test arrangement and geometry of specimens

In order to examine the creep behaviour of C/C-SiC under interlaminar shear loading a compression-shear-test was applied which was proposed by the authors earlier [1, 5]. The geometry of the unsymmetrically grooved specimens used is shown schematically in Fig. 2. If the laminae are oriented parallel to the loading direction, the interlaminar failure occurs under compressive loading along the desired path between the grooves (dashed line).

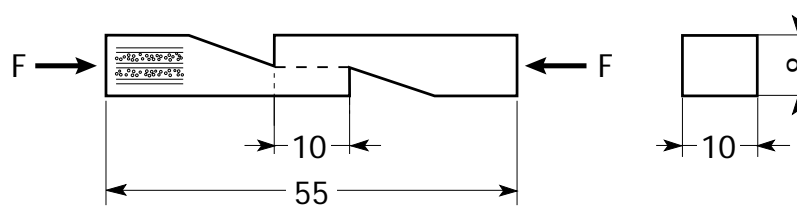


Fig. 2: Geometry of the unsymmetrically grooved compression-shear-specimens

Test procedure

The principal testing procedure and the devices for high temperature measurements of the ILSS were already described in [3, 6]. The test set-up is mounted in a universal testing machine and consists of a vacuum chamber in which the furnace and the loading devices are placed. The whole sample is positioned in the heated zone so that thermal gradients are minimized. All tests - except those at room temperature - were performed under inert gas atmosphere because the heating as well as the loading devices are made of carbon materials which are not protected against oxidation.

At first, the chamber was evacuated to about 4×10^{-5} mbar to remove most of the oxygen, then it was heated to 500°C and flooded with pure nitrogen to a pressure of about 1050 mbar corresponding to the external environment. After holding these conditions for 15 min the heating was continued. When the desired test temperature was reached, a preload of about 100 N was applied and kept constant until a thermal equilibrium is achieved. By following this procedure it is guaranteed that the whole test equipment is actually well positioned so that the further loading to the final test level does not yield to undesirable displacements caused by setting effects or by temperature variations. As soon as the desired load level is reached the actual creep test begins and the displacement is measured at constant loading for one hour if possible.

In order to achieve correct and reproducible results the temperature of the test set-up has to be exactly controlled and must be kept constant within narrow bounds. A temperature variation of one degree, for example, leads to a thermal expansion of the testing device in the order of the creep effects expected. To avoid such influences, an additional cooling system for the loading piston is integrated which adjusts the temperature within about 0.1 degree.

The creep deformation has to be measured outside the test chamber because no displacement measurement system was available which can directly be applied to the specimens at the high test temperatures considered. Therefore, two measurements were performed at each temperature and load level in which the displacement of the loading system during static loading was determined: 1. with the grooved compression-shear-specimens (s. Fig. 2) and 2. with a dummy. The dummy consists of the same C/C-SiC material and has the same dimensions as the compression-shear-specimen, but without the grooves. In this second test all parts of deformation were registered which result from the testing device (machine, piston, loading stamps ...) and from the axial

compression of the sample. Consequently, the difference between the displacement curves with the grooved specimens and the dummy should represent the creep which arises in the interlaminar plane between the grooves of the compression-shear-specimen.

Results

First, the quasistatic value of ILSS was determined at room temperature to obtain a reference value for the C/C-SiC batch investigated here. The mean value of three tests amounts to $= 27,3 \pm 3,5$ MPa. This is in very good agreement with the data of another batch which was tested earlier to determine the ILSS for the whole temperature range until 2000°C [1]. Following these results the creep loading levels of this study were selected and given as percentages of the quasistatic strengths at the temperature considered.

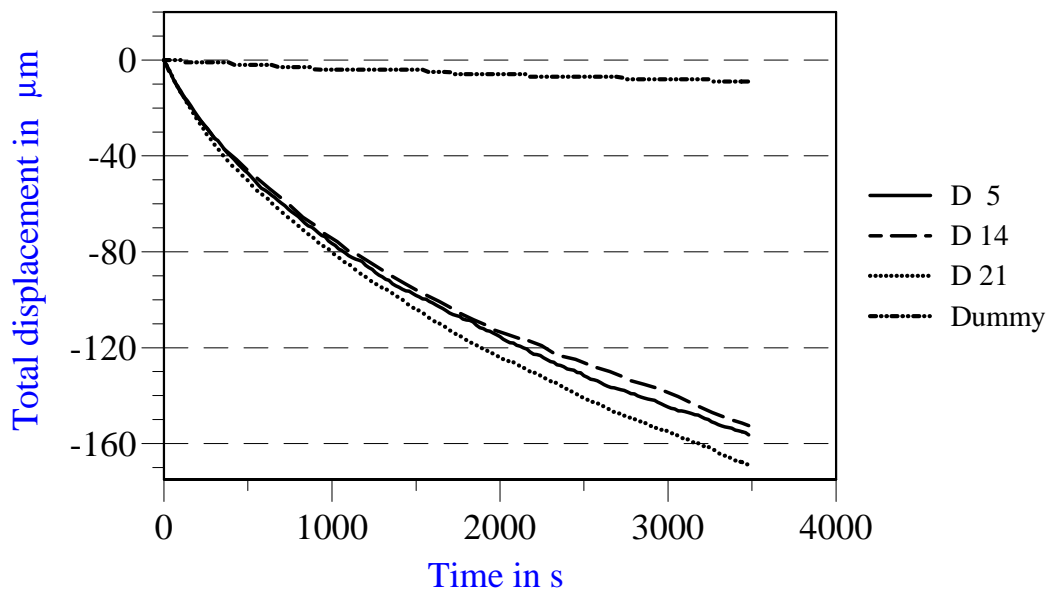


Fig. 3: Externally measured total displacements of three compression-shear-specimens (D 5, D 14, D 21) and of a dummy (all made of C/C-SiC) under static loading at 70% of the strength value at 1800°C

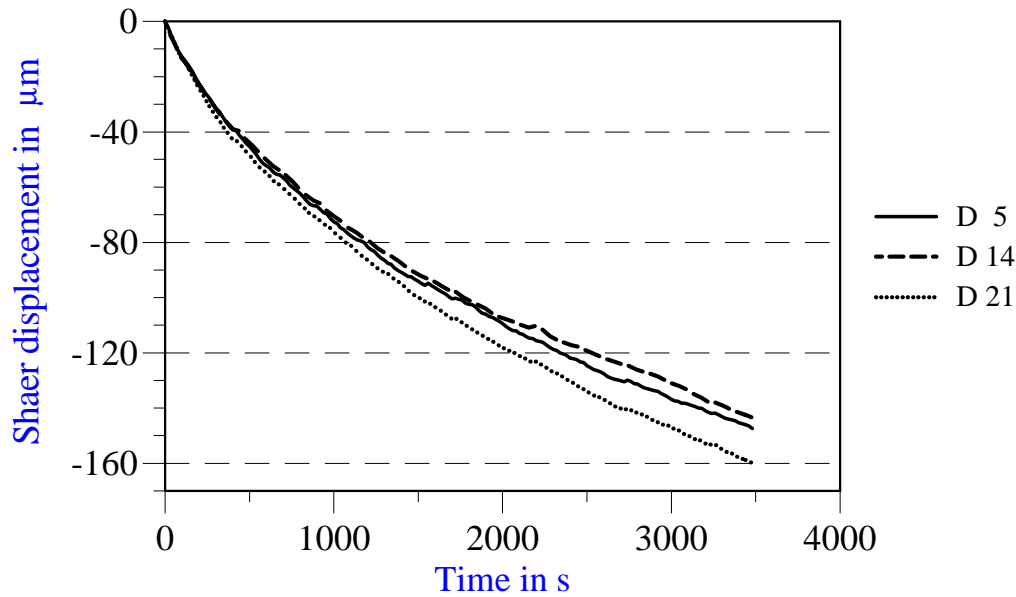


Fig. 4: Interlaminar creep deformation of the compression-shear-specimens of Fig. 3, obtained by the subtraction of the dummy-displacement

The results of the creep tests are plotted in the Figs. 3 to 5. First, as an example in Fig. 3 the total displacement curves of the compression-shear-specimens and of the dummy are shown for the most critical loading situation chosen (1800°C, 70% of fracture load F_F).

In Fig. 4 the dummy curve is subtracted from the curves of the corresponding compression-shear-specimens. Therefore, in this plot the true interlaminar creep deformations of the C/C specimens are shown.

In the same way three specimens each were investigated at four further loading levels: 70% of F_F at 1600°C and 1450°C; 60% of F_F at 1800°C and 1600°C.

The loading at 70% of F_F and 1600°C show an unexpected behaviour in comparison with all the other series: these specimens failed after a short loading time of only few minutes in the reduced cross section near the grooves by a brittle fracture perpendicular to the loading direction. In order to avoid this premature failure, additionally, specimens were tested which have reduced shear lengths of only 5 mm. In these specimens the same interlaminar shear strength arises by only half the load in comparison to the 10 mm specimen. Furthermore, possible bending stresses in the residual cross section behind the grooves which probably initiate the brittle fracture also can be reduced. This smaller type of specimen loaded at 70% and 60% of F_F at 1800°C and 1600°C, respectively, survived the test duration of about one hour.

All the results obtained including these from Fig. 4 are summarized in Fig. 5. In this diagramm the average creep curves calculated from three individual curves of each loading level are shown for comparison.

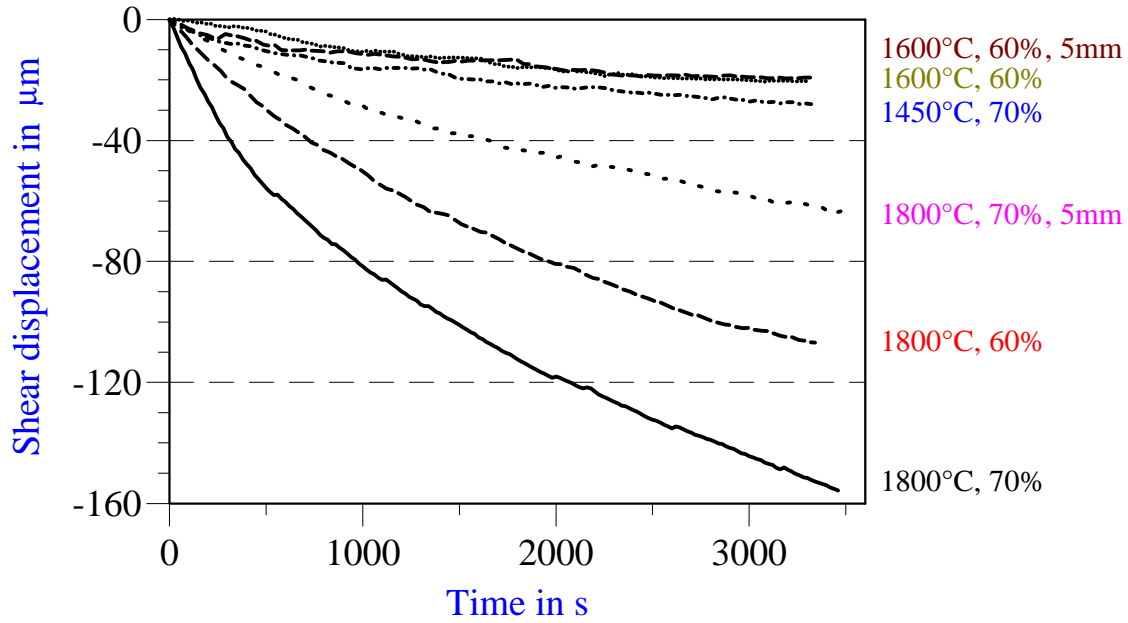


Fig. 5: Influence of the load and of the temperature level on the creep deformation of the C/C-SiC considered under interlaminar shear loading

Residual strength measurements and structural investigations

In order to investigate whether the mechanical properties and/or the structure of C/C-SiC were changed due to the thermomechanical treatment during creep experiments, additionally, the residual interlaminar shear strengths were measured at creep temperature and structural investigations were carried out by Transmission Electron Microscopy (TEM) .

In a first approach two series of strength measurements were considered in which the specimens exhibit a smaller distance between the grooves (5 mm) which guarantees an interlaminar shear failure. In Table 1 the ILSS values measured after creep are compared to the values of the reference material investigated in the state as delivered. No differences were found at 1600°C, however, the ILSS of the specimens with the maximum thermomechanical preloading (creep at 1800°C and 70% F_F) increases by about 15 % compared to non pretreated specimens. Therefore, microscopical investigations were performed to study possible influences of thermomechanical loading on the microstructure. An assessment of these results has to be considered under the restriction that the creep specimens were taken from another batch as the non pretreated specimens (s. above).

Table 1: Influence of the creep loading on the high temperature ILSS of C/C-SiC

Pretreatment	ILSS at 1600°C in MPa	ILSS at 1800°C in MPa
non [1]	37,3	38,0
creep at 1600°C, 60% F_F *	37,0	-
creep at 1800°C, 70% F_F *	-	43,8

* s. Fig. 5

For the TEM investigations appropriate samples were taken from three differently preloaded compression-shear-specimen: tested at room temperature (RT), at 1600°C and at 1800°C. The room temperature specimen was fractured under quasistatic loading without thermal treatment so that this material represents the state as delivered. The other two specimens, however, were loaded at 1600°C and 1800°C, respectively, and at 60% of the fracture load over about 80 min (creep).

All specimens show the typical microstructure with carbon fibers and crystalline matrix which consists of carbon, silicon carbide and free silicon. The grain size differ in a wide range, e.g. two types of silicon carbide were found: fine-grained (15-540 nm) and coarse-grained (0,5 - 6 μ m). Because of this scattering and the complex heterogeneous microstructure of the C/C-SiC considered each TEM image represents a random detail. Even TEM samples taken from the same compression-shear-specimen show different individual details each. In consequence, no significant differences could be identified between the structure of the thermomechanical preloaded creep specimens and the RT specimens of the state as delivered.

CONCLUDING DISCUSSION

Between the creep curves of the compression-shear-specimens tested at the same loading level only a small degree of scatter was found (s. Figs. 3 and 4). This finding shows that the properties of the test material are relatively uniform within the plate investigated and, furthermore, that the test conditions and the measurements are reproducible.

The creep experiments show that the interlaminar deformation behaviour depends on the load level and on the test temperature applied (s. Fig. 5): At 1800°C a reduction of the load from 70% to 60% reduces the creep displacement during the measurement time considered by about 30%.

In both cases the curves are not linear. Furthermore, the creep curves measured at the same load levels but at different temperatures (1450°C, 1600°C) demonstrate that the shear deformation is clearly diminished with decreasing temperature. However, even at the relatively high temperatures of 1450°C and 1600°C and load levels of 70% and 60% of F_F , respectively, the specimens withstand this high static loading with rather small deformations. Only at extremely high temperatures, e.g. of 1800°C, creep clearly effects remarkable nonlinear deformations.

The reduction of the shear length or of the distance between the grooves of the compression-shear-specimens (s. Fig. 2) from 10 mm to 5 mm yield an unexpected creep behaviour. On the one side it has to be expected that the shear deformation should be in the same order for both geometries because shear stresses of the same level were applied in each case. At 1800°C, however, for the 5 mm specimens the deformation amounts only about 40% of that one of the 10 mm specimens. On the other side this result indicates that the deformation at this extremely high temperature is not only induced by interlaminar shear stresses. It has to be assumed that bending stresses are superimposed caused by the unsymmetrically arranged grooves. This assumption can be supported by the change of failure behaviour in the quasistatic tests after creep loading: the specimens of the series 60% F_F /1600°C/10 mm show a brittle fracture perpendicular to the loading direction in the reduced cross section near the grooves whereas those of the series 60% F_F /1600°C/5 mm failed as requested by interlaminar shear along the path between the grooves.

Although the results of the creep experiments show that the matrix allows viscoelastic deformations at extremely high temperatures, the continuous increase of the quasistatic strengths with

increasing temperature (it was found between RT and 1450°C [1]) can not be explained by this effect. Therefore, the additional residual strength measurements and the TEM investigations do not give reliable indications for structural changes which might contribute to the mechanical improvement of C/C-SiC at high temperatures. Possibly, a further phenomenon by which the improvement of strength at high temperatures might be explained, could be an increasing insensitiveness of the material-inherent cracks to stresses caused by thermal induced crack closure mechanisms. This effect was not investigated in the frame of this work.

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