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EFFECT OF ENVIRONMENT ON CREEP BEHAVIOR OF SiC/SiC COMPOSITES

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ABSTRACT: Creep behavior of Standard SiC/SiC and Enhanced SiC/SiC composites has been studied. For the Standard SiC/SiC, the creep rates in air are much higher than in argon due to the effects of oxidation on creep. Conversely, the creep rates in argon are evidently higher than those in air for the Enhanced SiC/SiC. This implies that the oxidation resistance of the Enhanced SiC/SiC is much improved on one hand. Although creep rate of the Enhanced SiC/SiC in argon is higher than that of the Standard in argon, the time to rupture of the Enhanced SiC/SiC is still longer than that of the Standard one.

KEYWORDS: creep, oxidation, SiC/SiC, ceramic matrix composite

INTRODUCTION

Continuous fiber ceramic-matrix composites (CFCCs) are specially designed to have a weak interface which causes a crack to deflect along the interface, permitting intact fibers to bridge matrix crack faces for high fracture resistance [1-15]. However, the interface layer (e.g. carbon or BN coating) leads to low oxidation resistance at high temperatures [4-6]. This is a serious problem for non-oxide matrix composites such as SiC fiber reinforced SiC matrix composite (SiC/SiC). A glass-forming, boron-based particulates can be added to the matrix that reacts with oxygen to produce a sealant glass that inhibits oxidation of carbon layer. The modified SiC/SiC in this way was called the Enhanced SiC/SiC composite [7]. This paper presents the effect of environment on creep behavior in both Standard SiC/SiC and Enhanced SiC/SiC at high temperatures.

MATERIALS AND EXPERIMENTAL PROCEDURES

The composites used in the investigation were processed by chemical vapor infiltration (CVI) of SiC into plane woven $0^o/90^o$ SiC-fiber preforms. Before the infiltration the preforms were coated with carbon by CVD in order to decrease the interface bonding between the fibers and the matrix, thereby increasing toughness. The size of as-received composite panels was 200 x 200 mm with a thickness of 3.2 mm, and the composite contained 40 vol% SiC fibers and 9.7 % porosity. The diameter of SiC fiber (Nicalon^{TM}) was about 12 μm and each bundle consisted of 500 fibers.

The tensile specimens were machined from the panels using diamond cutting tools. The shape and dimensions of the specimens for the monotonic tension, creep and cyclic fatigue tests were described in reference [8-10]. The surfaces of the specimens were polished by an 800 grit grinding wheel before testing, thus the specimens were unprotected by a final CVI run after machining.

All the mechanical tests were carried out with a servo-hydraulic MTS 810 testing system. The monotonic tensile tests were conducted in air under constant displacement rate of 0.5 mm/min. Creep tests were conducted under a constant load in both air and argon atmosphere.

Creep strain was measured directly from the gage length of the specimen by a contact extensometer. Periodically partial unloading-reloading was applied to measure the modulus change during creep tests. The specimens were allowed to soak about 30 min before starting the tensile tests and creep tests. After fracture, the specimens were examined by both optical microscopy and scanning electron microscopy (SEM).

RESULTS AND DISCUSSION

The micrographs of the Enhanced and Standard SiC/SiC composites in original state are shown in Fig. 1. The differences between them are that there are glassy phases in the matrix of the Enhanced SiC/SiC (grey phases in the matrix in Fig. 1(c)). The thickness of carbon layer (0.5-0.6 μm) at the interfaces of the Enhanced SiC/SiC is larger than that of the Standard SiC/SiC (0.1-0.2 μm). Moreover, there are more pores in fiber bundles of the Enhanced SiC/SiC than in those of the Standard SiC/SiC.

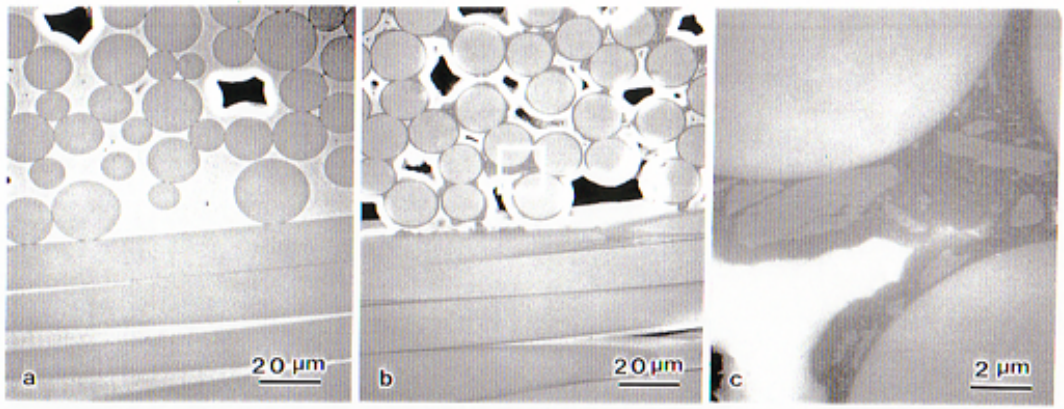

Fig. 1 Microstructures of the Standard SiC/SiC and the Enhanced SiC/SiC. (a) Standard; (b) Enhanced; (c) Glassy phases in the matrix of Enhanced SiC/SiC.

Creep rates of the Enhanced SiC/SiC and Standard SiC/SiC in air and argon are shown in Fig. 2. For the Standard SiC/SiC, the creep rates in air are much higher than in argon due to the effects of oxidation on creep. Conversely, the creep rates in argon are evidently higher than those in air for the Enhanced SiC/SiC. This implies that the oxidation resistance of the Enhanced SiC/SiC is much improved on one hand. On the other hand, this is partly because creep rates of SiC fibers in argon are higher than those in air. Creep rates of the Enhanced SiC/SiC in argon are higher than those of the Standard SiC/SiC due to the low creep resistance of the enhanced matrix. However, in air, creep rates in the Enhanced SiC/SiC are substantially lower than those in the Standard SiC/SiC.

The time to rupture in air is longer than that in argon at a given stress in the Enhanced SiC/SiC, while the time to rupture in air is much shorter than that in argon at a given stress in the Standard SiC/SiC, as shown in Fig. 3. The time to rupture of the Enhanced SiC/SiC is much longer than that of the Standard SiC/SiC in air. Although the creep rate of the Enhanced SiC/SiC in argon is higher than that of the Standard one in argon, the time to rupture of the Enhanced SiC/SiC is still longer than that of the Standard one. This demonstrates that the addition of glassy phase in the matrix of the Enhanced SiC/SiC increases creep rates, but much improves total creep time to failure in argon. The reason for this result may be understood by the match of creep resistance between fibers and the matrix. Creep resistance of SiC fibers (Nicalon^{TM}) is lower than that of SiC matrix. Creep of fibers transfers the stress onto the matrix and

causes matrix cracks. The matrix cracking reload the fibers. As the matrix creep resistance decreases, creep relaxation of the matrix may decrease matrix cracking and stress concentration near large pores, at which creep cracks often initiated.

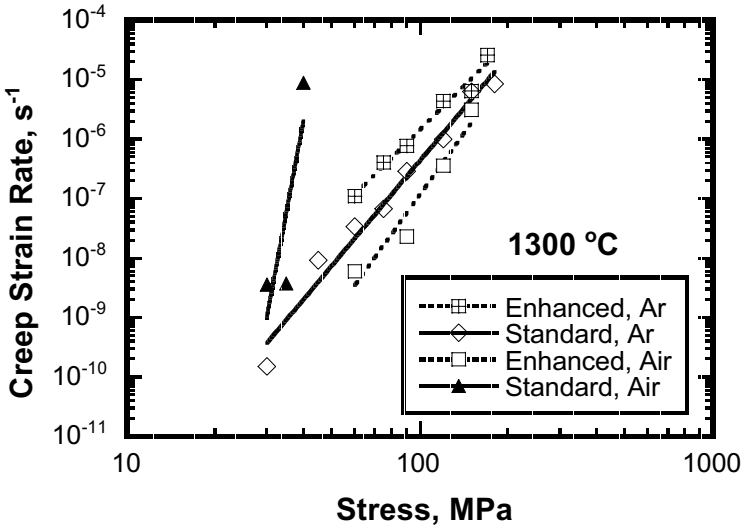

Fig. 2 The minimum creep strain rate as a function of stress in Enhanced SiC/SiC and Standard SiC/SiC at 1300 °C in air and argon.

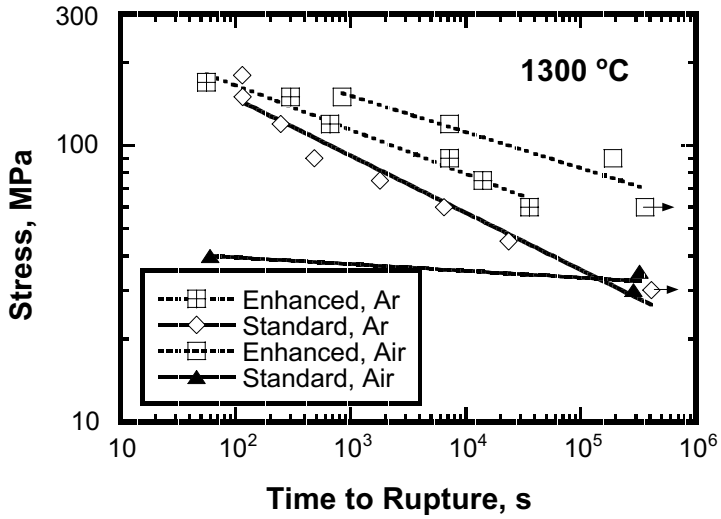

Fig. 3 Time to rupture versus stress in Enhanced SiC/SiC and Standard SiC/SiC at 1300 °C in air and argon.

Young's modulus during creep was periodically measured to examine the cracking evolution. It was found that the variation of the Young's modulus was related to the evolution of matrix cracks (Fig. 4(a)). The modulus decreases with time at stresses (120 and 180 MPa) higher than the matrix cracking stress and remains constant at lower stresses (45 and 60 MPa). However, the modulus in Standard SiC/SiC is almost constant, as shown in Fig. 4(b).

The modulus in Enhanced SiC/SiC decreases with creep time at the high stresses in the same way as in the Standard SiC/SiC [7]. The reduction of the modulus indicates creep damage evolution. If the fibers have a lower creep resistance than the matrix, the gradual decrease in

modulus during creep is because creep of the bridged fibers transfers stress to the matrix and causes matrix cracking and crack growth. Fig. 5 gives the data at the stress of 60 MPa which is lower than the matrix cracking stress. Therefore, the first loading and the early stage of creep do not exhibit reduction of modulus. If the reduction of modulus is thought to reflect multiplication and propagation of the matrix cracks in the specimens, no extensive matrix cracks can be expected according to the constant modulus for Standard SiC/SiC. This is consistent with the observation results of cracks [7]. The reason for the degradation of modulus after an early creep in Enhanced SiC/SiC is considered to be the lower creep resistance in the matrix.

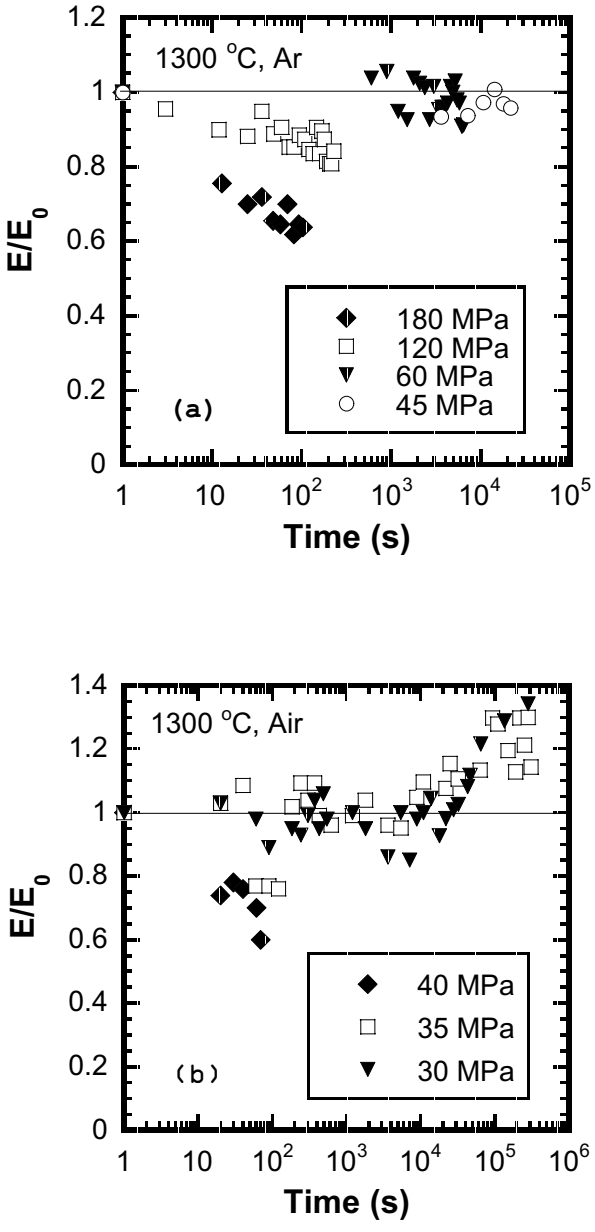

Fig. 4 Variation of Young's modulus measured by partial unloadings with time during creep at 1300 °C, E_0 is the modulus in the linear portion during loading. (a) in argon; (b) in air.

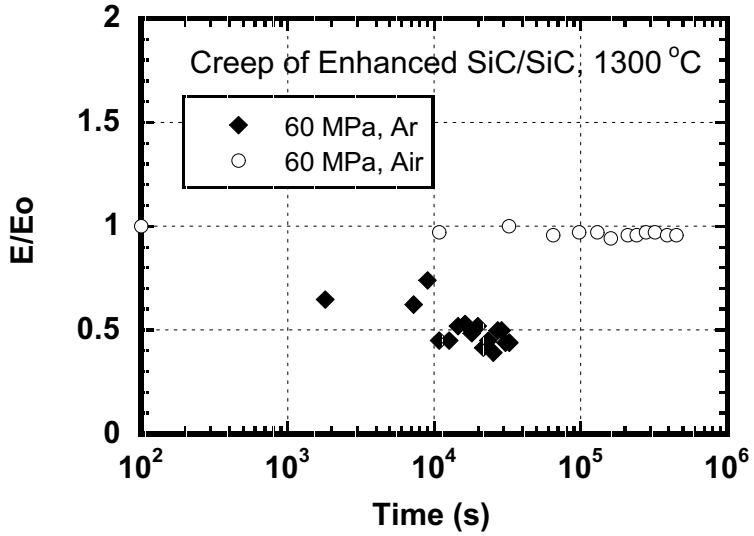

Fig. 5 Young's modulus normalized by the value of the first loading (E/Eo) versus time of the Enhanced SiC/SiC under constant load in air and argon at 60 MPa and at 1300 °C.

It can be seen that the modulus remains constant during creep in air, but decreases with time in argon (Fig. 5). Although the applied stress for creep test is 60 MPa below the matrix cracking stress (70 MPa), the damage led to the degradation of Young's modulus is also the matrix cracking in argon. The observation of the specimen crept at 60 MPa in argon showed matrix cracks initiated from the large pores [7]. This may be because of the lower creep resistance of fibers in argon than in air [16]. The creep of fibers transfers the stress onto the matrix and causes matrix cracking. Moreover, the sealing of cracks by glass may more effective in air than in argon, since there are more oxygens in air to react with the glass-forming particulates than in argon.

Creep cracks are always found at the large pores among the fiber bundles (indicated by arrows in Fig. 6). When cracks meet 0° fibers, debonding of interfaces between fibers and the matrix occurs. The 0° fibers bridge crack faces and therefore decrease the driving force at the crack tip as a bridging component [1-3]. At 1300 °C, the glassy phases become liquid, which flow into cracks. At room temperature they become solid again and situated in the cracks (Fig. 6). Around the large pores between fiber bundles, the matrix is the last coating by the pure SiC (light colored layer), the same as the matrix in the Standard SiC/SiC. Crack propagation is very straight in this layer, but deflected or discontinous in the inner matrix (dark color) (Figs. 6 and 7). This means that creep crack propagation occurs in the matrix of the Enhanced SiC/SiC.

Fracture surfaces under creep in air show that the interfaces between fibers and the matrix become hollow [7]. This is because the pyrolytic carbon layer at the interfaces reacts with oxygen [17-20] and forms gas evaporating into environment. On the matrix, there are many pores or holes. Their size is the same as the glass-forming particulates. Therefore, it is thought that the particulates become glass and flow out to seal cracks at high temperatures. The borosilicate glass did not wet the surface and acted to seal the interior from oxidation. Although carbon interphase oxidation on the fracture surfaces, in most areas in the specimens no oxidative attack exists and carbon remains at the interfaces in Enhanced SiC/SiC.

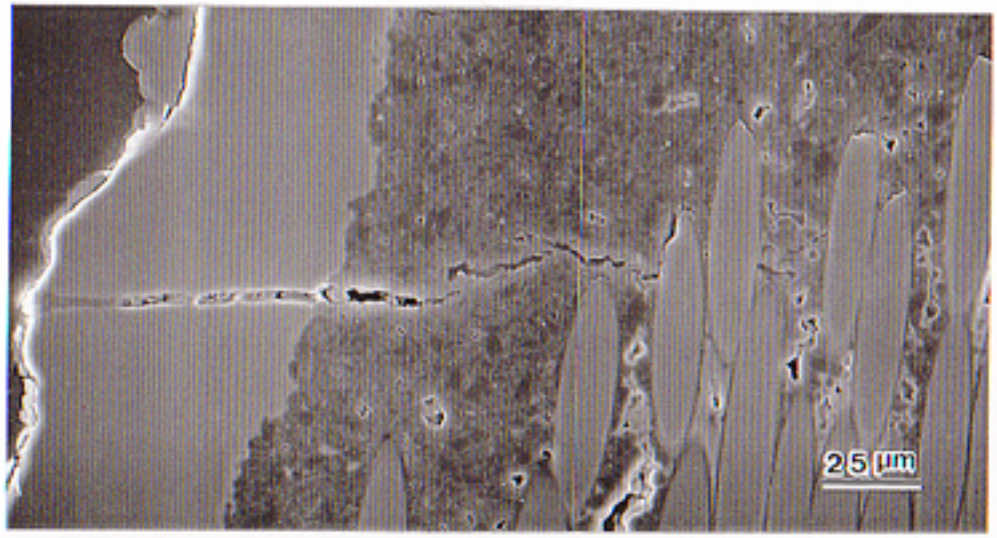

Fig. 6 Crack propagation in the specimens of the Enhanced SiC/SiC crept in air at 1300 °C and 90 MPa for 53 h.

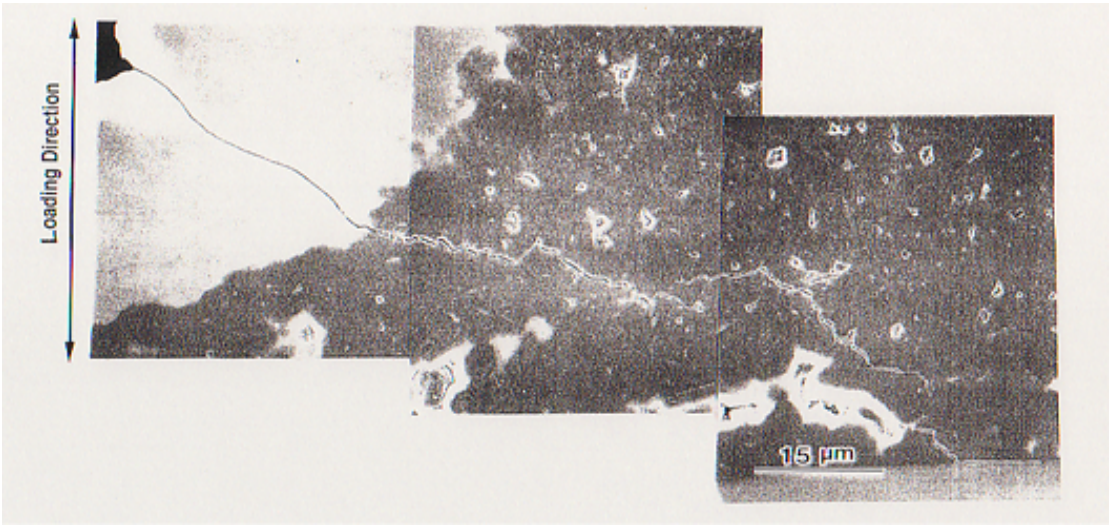

Fig. 7 Creep crack propagation paths in the specimen of the Enhanced SiC/SiC crept in argon at 1300 °C and 90 MPa for 2 h.

For the Standard SiC/SiC, strength and stress rupture life in air are always lower than those in argon or vacuum due to oxidation [19-23]. The oxidation of the Standard SiC/SiC includes two concurrent phenomena: oxidation of the pyrocarbon interphase which creates an annular porosity around the fibers; and silica formation on the free surfaces of the fiber and/or the matrix, which progressively closes the porosity and the access for oxygen towards the interphase, and consequently stops the oxidation processes [17,18]. The annular porosity around the fibers decreases fiber strength, which decreases with an increase in gage length. The silica formation on the free surfaces of the fiber and/or the matrix produces a strong interface which is harmful for both strength and ductility. Therefore, both the annular porosity around the fibers and the silica formation on the free surfaces of the fiber and/or the matrix decrease the strength of the composite. This is the primary reason for the lower creep and resistance of the Standard SiC/SiC in air than in argon (Fig. 2). However, the thermodynamic stability of Nicalon^{TM} fiber at high temperature should be also considered.

The fibers heat-treated in argon at high temperatures showed severe degradation caused by the decomposition of the silicon oxycarbide phase, which resulted in CO and silicon monoxide gas evolution. While, fibers treated in an oxidizing environment (O_2, air) showed slightly less degradation. Only 25% of the initial strength of the fibers was retained after the treatment at 1300°C in argon. The instability of Nicalon™ fiber at high temperature in argon may be the reason for the close creep resistance at low stresses in air and argon.

CONCLUSIONS

(1). For the Standard SiC/SiC, the creep rates in air are much higher than in argon due to the effects of oxidation on creep. Conversely, the creep rates in argon are evidently higher than those in air for the Enhanced SiC/SiC. This implies that the oxidation resistance of the Enhanced SiC/SiC is much improved on one hand. On the other hand, this is partly because creep rates of SiC fibers in argon are higher than those in air.

(2). Creep rates of the Enhanced SiC/SiC in argon are higher than those of the Standard SiC/SiC due to the low creep resistance of the enhanced matrix. However, in air, creep rates in the Enhanced SiC/SiC are substantially lower than those in the Standard SiC/SiC.

(3). Although creep rate of the Enhanced SiC/SiC in argon is higher than that of the Standard in argon, the time to rupture of the Enhanced SiC/SiC is still longer than that of the Standard one.

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