

Stress, Strain and Elastic Modulus Behaviour of SiC/SiC Composites during Creep and Cyclic Fatigue

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Abstract

Creep and fatigue tests of Hi-NicalonTM/SiC (SiC matrix contains glass-forming, boron-based particulates), Standard SiC/SiC (SiC matrix is pure SiC) and Enhanced SiC/SiC (SiC matrix contains glass-forming, boron-based particulates) were carried out in air at 1300°C. The stress–strain hysteresis loops during fatigue and creep were studied. The change of Young's modulus during creep and fatigue was analysed and compared among the three kinds of materials. Creep strain rates of Hi-NicalonTM/SiC in air were similar to those of Enhanced SiC/SiC, but much lower than those of Standard SiC/SiC. Consequently, the time to rupture at a given stress in Hi-NicalonTM/SiC was similar to that in Enhanced SiC/SiC, but much longer than in Standard SiC/SiC. Fatigue resistance of Hi-NicalonTM/SiC was similar to that of Enhanced SiC/SiC, but much better than Standard SiC/SiC. © 1998 Elsevier Science Limited. All rights reserved

1 Introduction

In recent years, creep and fatigue of continuous fiber reinforced ceramic matrix composites (CMCs) have been hot topics,^{1–20} since these properties are indispensable for considering the application of CMCs. To get high fracture toughness and thermal shock resistance, CMCs were designed with weak interface between fibers and matrix, e.g. the interface

in SiC/SiC composite coated by carbon. The weak interface can cause crack to deflect along the interfaces, permitting intact fibers to bridge crack faces.¹ However, although the use of weak interfaces can increase fracture toughness and thermal shock resistance,² it is not compatible with creep and fatigue resistance at high temperature, which demands strong interfaces resisting the nucleation and growth of cavities.⁴

Carbon coating layer in SiC/SiC leads to low oxidation resistance at high temperatures. A glass-forming, boron-based particle can be added to the matrix that reacts with oxygen to produce a sealant glass that inhibits oxidation.⁶ This technology is applied to SiC/SiC composites. The modified SiC/SiC was called Enhanced SiC/SiC[†] composite.^{6,10} The Enhanced SiC/SiC composites exhibited good high temperatures (up to 1300°C) properties in air.¹⁰

Since matrix microcracking occurs during initial application of a creep load, fiber bridging of matrix cracks operates in creep of Standard SiC/SiC at high stresses, although creep resistance of SiC fibers is lower than that of SiC matrix.⁷ This is undesirable for environmental resistance of the composites, if they are exposed to air. Because creep of fibers controls matrix crack growth,^{21,22} increasing creep resistance of fibers can improve creep behavior of the composite. Moreover, the decreasing creep resistance of the matrix by the addition of oxides is also expected to increase creep resistance²³ and simultaneously improve environmental resistance. It was found that the addition of glassy phases in the SiC matrix increased the creep and oxidation resistance in Enhanced SiC/SiC composite at 1300°C, compared with Standard SiC/SiC.¹⁰

Cyclic fatigue behavior of CMCs at high temperatures is not yet well understood. Environment factors, creep of constituents, thermally induced stresses at interfaces and interfacial sliding resistance, etc., may cause the reduction of fatigue life

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[†]In this paper, standard SiC/SiC indicates NicalonTM fibers reinforced SiC composite, in which the SiC matrix is pure SiC. Enhanced SiC/SiC indicates NicalonTM fibers reinforced SiC composite, in which the SiC matrix contains glass-forming, boron-based particulates. Hi-NicalonTM/SiC indicates Hi-NicalonTM fibers reinforced SiC composite, in which the SiC matrix contains glass-forming, boron-based particulates.

at high temperatures.^{8,18–20} Creep of fibers and the degradation of the interfacial sliding resistance were considered to be the reasons for decreased fatigue resistance at high temperatures in a Standard SiC/SiC composite.^{8,9,19}

The presence of SiC_xO_y amorphous phase in NicalonTM fibers is responsible for the low creep resistance owing to a viscous flow at temperatures as low as 1000–1200°C.²⁴ The silicon oxycarbide phase decomposes, forming SiC and gaseous species such as CO and SiO, whose diffusion through the fiber and reaction with the free carbon are believed to create pores and other defects in the fiber structure.²⁵ Such a decomposition causes strength and Young's modulus degradations and affects the fiber creep behavior even in inert atmospheres.^{26,27} The elimination of SiC_xO_y from the fibers can improve creep resistance by electron irradiation in vacuum instead of curing in air.²⁷ The Young's modulus is also increased.^{27–30} The modified NicalonTM fibers are called Hi-NicalonTM fibers. To increase the mechanical properties of SiC/SiC, Hi-NicalonTM fibers were used to reinforce the enhanced SiC matrix.

Both creep and fatigue tests of Hi-NicalonTM/SiC, Enhanced SiC/SiC and Standard SiC/SiC were carried out in air at the same maximum stresses to compare time-dependent deterioration with cyclic-dependent damage. The mechanical behavior of Hi-NicalonTM/SiC was compared with Enhanced SiC/SiC and Standard SiC/SiC to investigate the effects of improving fibers and the matrix on creep and fatigue resistance at high temperature.

2 Materials and Experimental Procedures

The composites used in the investigation were processed by chemical vapor infiltration (CVI) of SiC into plain woven 0°/90° NicalonTM fiber preforms for Standard SiC/SiC and Enhanced SiC/SiC, while satin woven 0°/90° Hi-NicalonTM fiber preforms for Hi-NicalonTM/SiC (made by Du Pont Lanxide Composites Inc., DE). Before the infiltration the preforms were coated with carbon by CVD in order to decrease interface bonding between fibers and the matrix, thereby increasing toughness. The thickness of the carbon coating layer is 0.2~0.5 μm for the Standard SiC/SiC, the Enhanced SiC/SiC and the Hi-NicalonTM/SiC. The composites, processed as 200×200 mm panels with a thickness of 3.2 mm, contained 40 vol% SiC fibers and 9.7% porosity. The diameter of a fiber 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 Refs 8–10. The surfaces of the specimens were machined by an 800 grit grinding wheel before testing. The specimens were not protected by a final CVI run after machining.

All the mechanical tests were carried out with a servo-hydraulic MTS 810 testing system (MTS System Corporation, MN) at 1300°C in air. The fatigue tests were performed with sinusoidal loading frequency of 20 Hz. The stress ratio (*R*), which was defined as the ratio of minimum stress to maximum stress, was 0.1 for fatigue tests. Creep tests were conducted under constant load. Creep strain was measured directly from the gage length of the specimen by a contact extensometer (MTS Model 632.53-F71, MTS System Corporation, MN), which has measuring range of ± 2.5 mm over its gage length of 25 mm. Repeated unloading–reloading with a rate of 50 MPa s^{−1} was applied to measure the modulus change during the creep tests. The specimens were allowed to soak over 30 min at 1300°C before starting creep or cyclic fatigue tests. After fracture the specimens were examined by both optical microscopy and scanning electron microscopy (SEM).

The alignment between the upper and lower grips of the load unit was verified using the steel dummy specimen for verification supplied by MTS Corporation to allow the bending strain less than 5% in accordance with ASTM Standard E 1012-89. Analytical and empirical analysis studies have concluded that for negligible effects on the estimates of the strength distribution parameters (for example, Weibull modulus and characteristic strength) of monolithic advanced ceramics, allowable bending as defined in ASTM Practice E 1012 should not exceed 5%. Similar studies of the effect of bending on the tensile strength distributions of continuous fiber reinforced ceramic matrix composites do not exist. ASTM Practice C 1275-94 adopts the recommendations for tensile testing of monolithic advanced ceramics. Since CMCs have inelastic deformation, which can redistribute stress state and sometimes lead to notch insensitivity, 5% of bending strain should not affect the strength distribution.

3 Results and Discussion

3.1 Microstructures

The plain woven fiber structures in both Standard SiC/SiC and Enhanced SiC/SiC, and satin woven fiber structures in Hi-NicalonTM/SiC composite are shown in Fig. 1. There are glassy phases in the enhanced SiC matrix of both Enhanced SiC/SiC

and Hi-NicalonTM/SiC [dark phases in the matrix in Fig. 1(d)].

The ultimate tensile strength of the Hi-NicalonTM/SiC is 235 MPa and the proportional limit is 80 MPa. The tensile strength of the Enhanced SiC/SiC is 230 MPa and the proportional limit is 80 MPa.¹⁰ The tensile strength of the Standard

SiC/SiC is 230 MPa in argon and 110 MPa in air, while the proportional limit is 90 MPa in argon and air.⁸

3.2 Fatigue and creep

The cyclic fatigue life versus the maximum stress of Hi-NicalonTM/SiC is similar to that of Enhanced

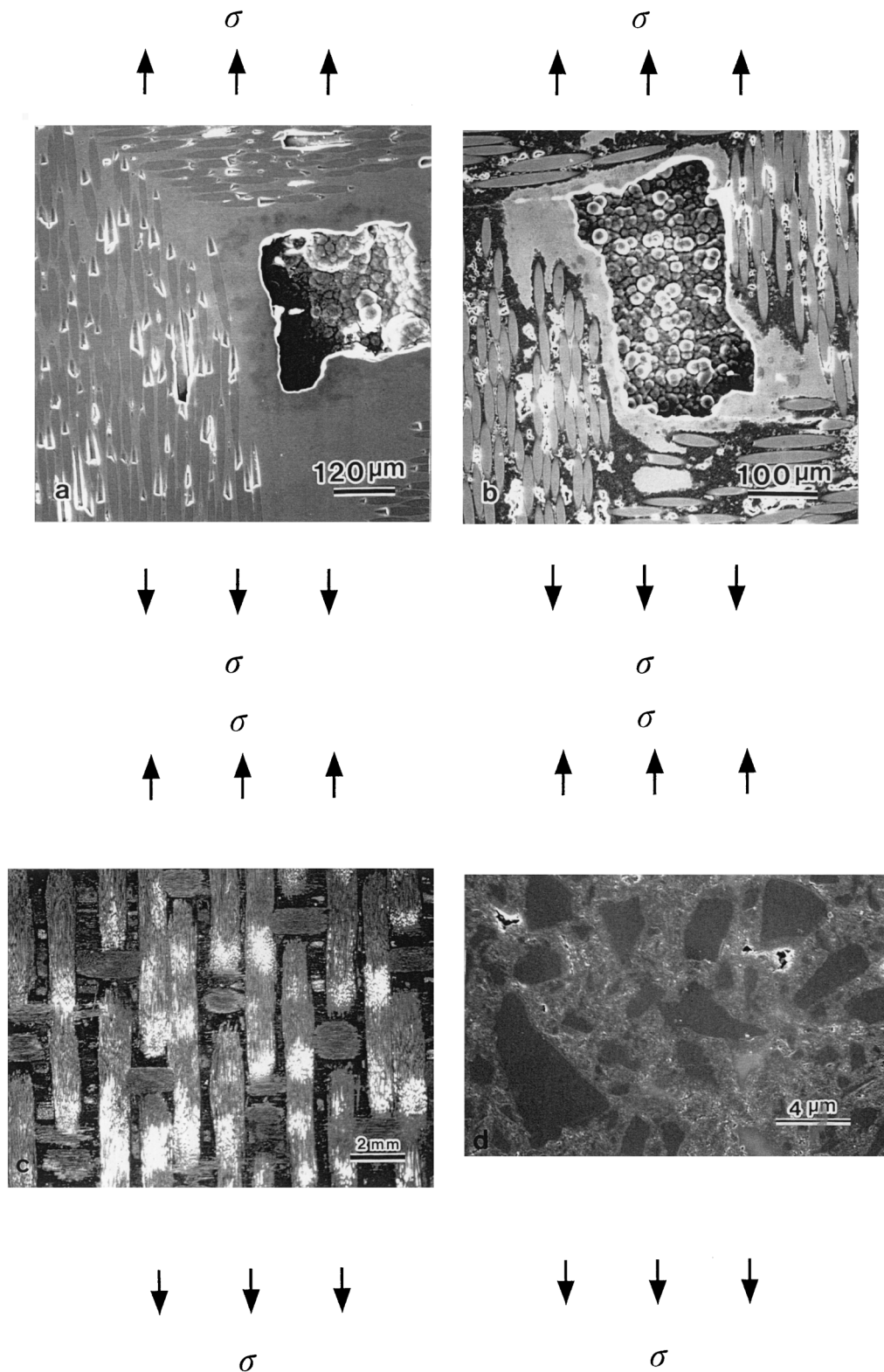


Fig. 1. (a) Plain-woven structure of the Standard SiC/SiC; (b) plain-woven structure of the Enhanced SiC/SiC; (c) satin-woven structure of the Hi-NicalonTM fiber bundles in the Hi-NicalonTM/SiC composite; (d) glassy phases (dark blocks) in the matrix of the Hi-NicalonTM/SiC.

SiC/SiC, but much longer than that of Standard SiC/SiC at 1300°C in air (Fig. 2).

The minimum creep rate of Hi-Nicalon™/SiC at 1300°C in air is much lower than that of Standard SiC/SiC and is similar to that of Enhanced SiC/SiC (Fig. 3). The time to rupture of Hi-Nicalon™/SiC is much longer than that of Standard SiC/SiC, and is similar to that of Enhanced SiC/SiC (Fig. 4).

The creep behavior of the composites depends on the creep of the matrix, the fibers and the interphases and interfaces between the fibers and the matrix. At present, not only the in-situ creep behavior of the matrix and fibers is not known, but also the creep behavior of the individual fibers and matrix is not well understood. In Hi-Nicalon™/SiC, there is not any creep data of the enhanced matrix available for analysing creep behavior of the composite. The amount of the additives in the matrix is also not known. Therefore, it is impossible to compare creep resistance of the matrix to that of the fibers to know the load transfer direction during creep. However, all the testing stresses

for creep of Hi-Nicalon™/SiC in air are higher than the proportional limit, which can be thought as an approximate stress of the matrix cracking. The matrix microcracking occurs during initial application of a creep load, therefore fiber bridging of matrix cracks always operates whether creep rate of fibers is higher or lower than the matrix. In the crack bridging mechanism, matrix crack

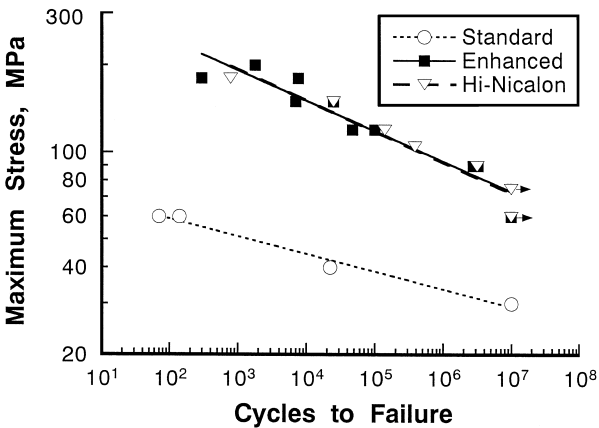


Fig. 2. The maximum stress versus cycles to failure for fatigue in Hi-Nicalon™/SiC, Enhanced SiC/SiC, and Standard SiC/SiC in air at 1300°C.

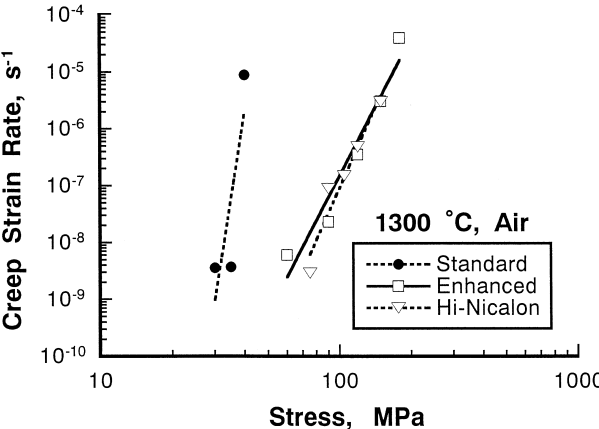


Fig. 3. The minimum creep strain rate as a function of stress in Hi-Nicalon™/SiC, Enhanced SiC/SiC and Standard SiC/SiC at 1300°C in air.

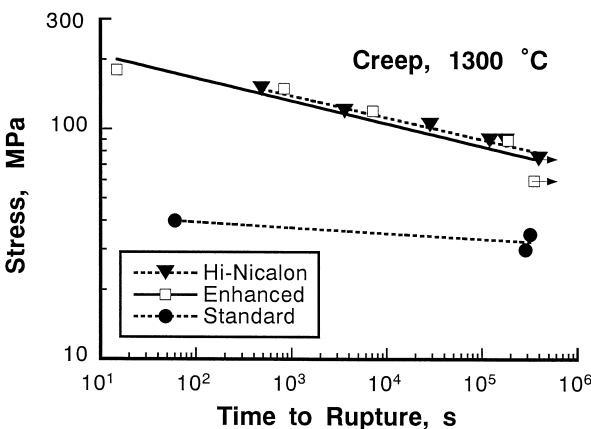


Fig. 4. Time to rupture versus stress in Hi-Nicalon™/SiC, Enhanced SiC/SiC and Standard SiC/SiC at 1300°C in air.

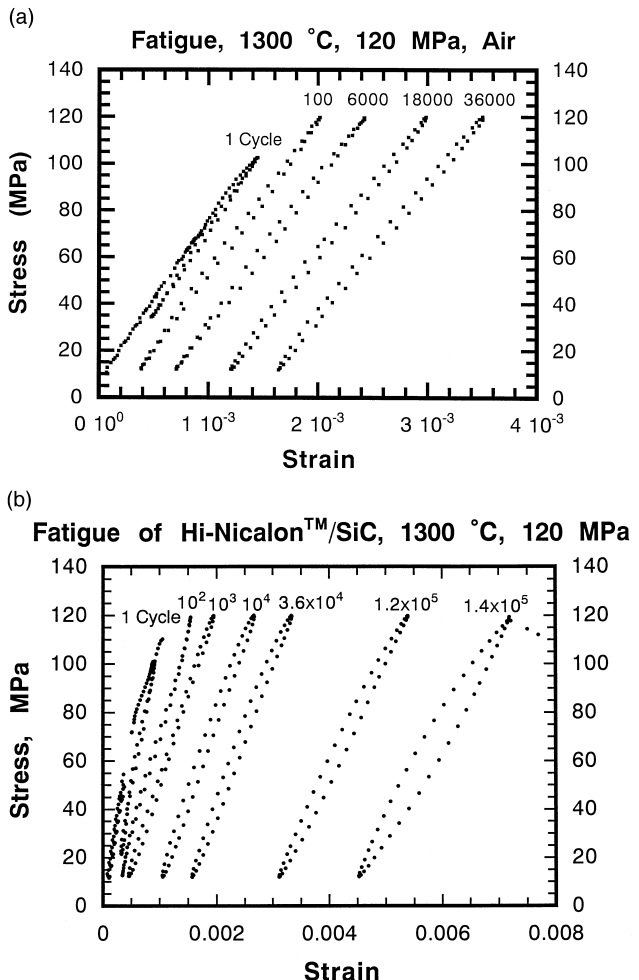


Fig. 5. Evolution of the hysteresis loops during fatigue at 1300°C under the maximum stress of 120 MPa in air: (a) Enhanced SiC/SiC; (b) Hi-Nicalon™/SiC.

growth rate is governed by a process in which the increase in the crack length is accompanied by an increase in the number of fibers bridging the crack. This continues up to a steady state condition produced by the competition between creating more bridged fibers as the crack length increases and the fracture or creep of these fibers as more stress is transferred to them by the increased crack-opening displacement. Therefore, the effects of creep resistance of the bridged fibers on creep behavior of the composite are expected to be important.

Although the greater creep resistance (one order of magnitude) of Hi-NicalonTM fibers was found compared to the normal NicalonTM fibers,²⁷ creep resistance of Hi-NicalonTM/SiC is slightly higher than Enhanced SiC/SiC in which the matrix is the same as in Hi-NicalonTM/SiC but the fiber is normal NicalonTM fiber. This seems that Hi-NicalonTM fiber does not improve creep resistance of the composite, if the difference in fiber architectures (satin structure in Hi-NicalonTM and plain-woven structure in Enhanced SiC/SiC) is neglected. In fact, the effects of fiber architectures on creep of the composites may be not small. The bending extent of the fibers in satin structure is

smaller than in plain-woven structure. This may lead to less damage in fiber bundles and is beneficial to fiber pullout.

3.3 Modulus change

The gradual modulus degradation in cyclic fatigue has been reported for the unidirectional and laminated ceramic composites at room temperature^{31–35} and elevated temperatures.^{5,16,17,36} It has been shown that the gradual damage growth accompanies modulus decrease in the CMCs under fatigue loading.^{33,35}

To understand the damage evolution and degradation mechanism during fatigue and creep, Young's moduli were measured. Figure 5 shows the evolution of the stress–strain hysteresis loops. The slope decreases and the width of the loops increases with cycles. The former indicates the decrease of the modulus and the latter means the decrease of the interfacial sliding resistance. The hysteresis loops move to the right along the strain axis, which is known as ratchetting due to time-dependent deformation (e.g. creep). The modulus normalized by the value from the linear part during the first loading versus cycles is shown in Fig. 6. At

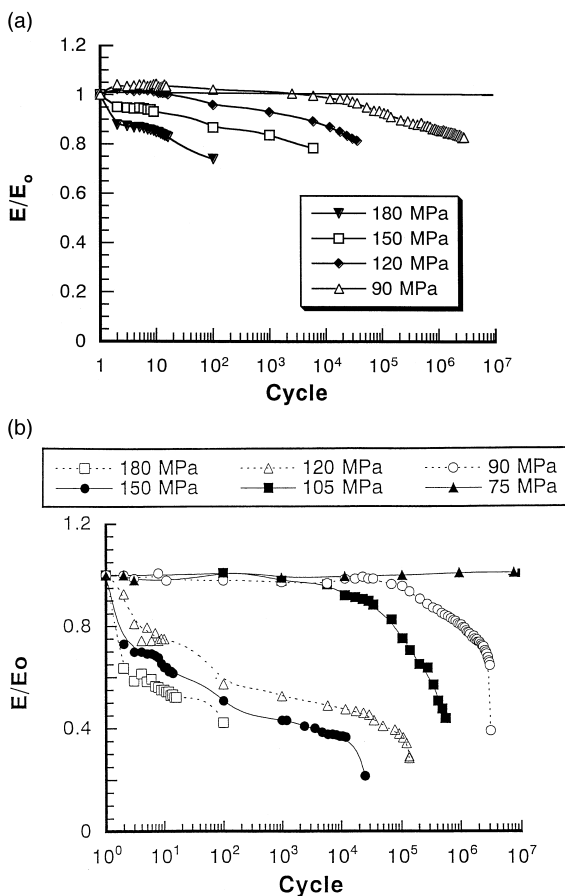


Fig. 6. The elastic modulus normalized by the value of the modulus under the first loading (E/E_0) versus cycles for fatigue in air at 1300°C under the different maximum stresses. (a) Enhanced SiC/SiC; (b) Hi-NicalonTM/SiC.

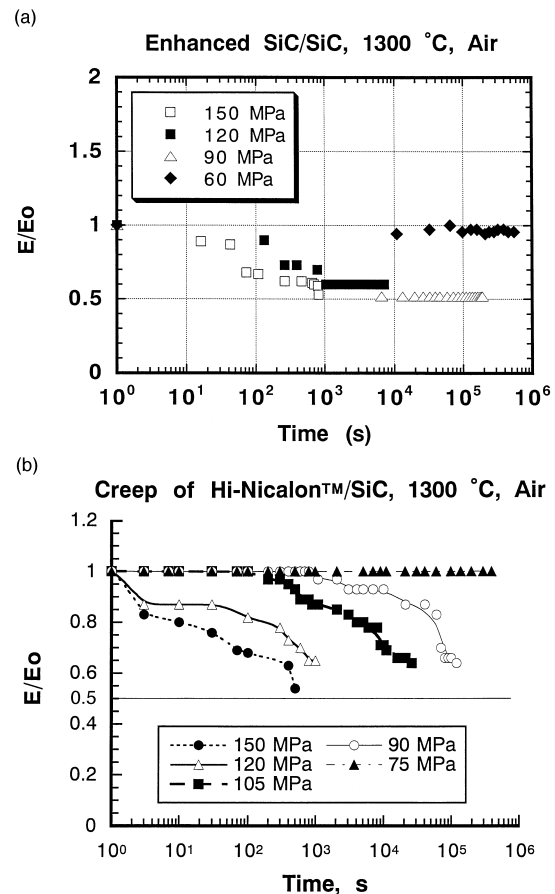


Fig. 7. The elastic modulus normalized by the value of the modulus under the first loading (E/E_0) versus time for creep in air at 1300°C under the different stresses: (a) Enhanced SiC/SiC; (b) Hi-NicalonTM/SiC.

the stresses ≥ 120 MPa, the modulus decreases rapidly within 10 cycles, and then goes to gradual decrease stage, and finally drops fast up to fracture. At the stresses ≥ 105 MPa, the modulus first keeps constant up to 10^4 cycles and then monotonously decreases. At 75 MPa, the modulus keeps constant up to 10^7 cycles, at which the test was stopped. When the modulus decreases to 20–40% of the original value, the specimens fracture.

The change of the modulus during creep with time in air (Fig. 7) is similar to that during fatigue (Fig. 6). However, the limit modulus for fracture is 50–60%, higher than under fatigue. This is the same as the results in Standard SiC/SiC,⁹ in which it was explained by the longer debonding of the interfaces under fatigue.

If the fibers have a lower creep resistance than the matrix, the gradual decrease in modulus during creep is because the creep of the bridging fibers transfers more stress to the matrix and causes matrix cracking and crack growth.^{37–39} However, it is not known whether Hi-NicalonTM fibers have a higher or lower creep resistance than the SiC matrix with the additives. Since the reduction of

Young's modulus reflects multiplication and propagation of the matrix cracks in the specimens under fatigue tests,³³ the first loading did not produce extensive matrix cracks at the stresses ≤ 105 MPa according to the constant modulus stage (Fig. 7). During this stage, creep occurs, incubating damage for propagation of the matrix cracks. At the stresses ≥ 120 MPa, extensive matrix cracks are bridged by fibers. Therefore, creep of the fibers promotes propagation of the cracks, leading to the decrease of the modulus.

3.4 Microscopic damage and fracture

Creep and fatigue cracks are always found at the large pores between fiber bundles [(Fig. 8(a) and (b)]. 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 general bridging mechanism [Fig. 9(a)]. 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. 9 (b)]. The oxidation occurred on the interfaces and the fibers and the matrix (Figs 8,9). Fibers can be severely

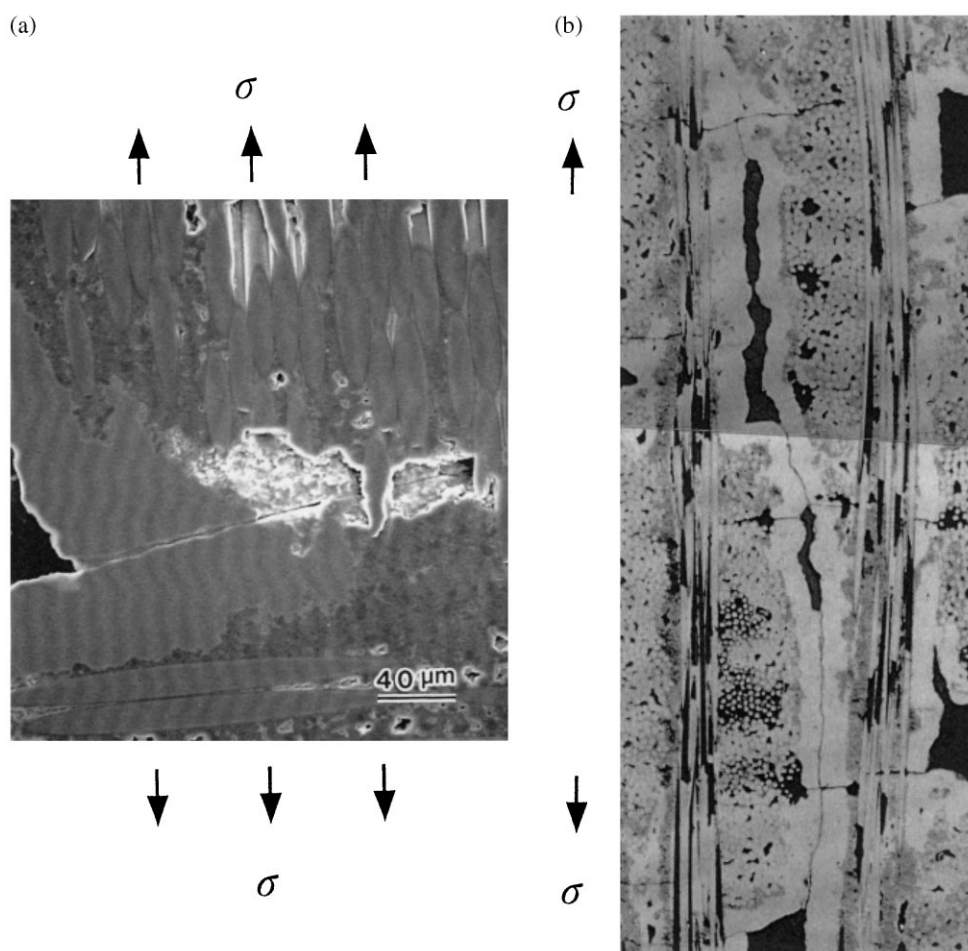


Fig. 8. (a) A crack initiated at a large pore in the specimen of the Enhanced SiC/SiC fatigued in air at 1300°C and 75 MPa for 10^7 cycles; (b) cracks in the specimen of Hi-NicalonTM/SiC fatigued in air at 1300°C and 150 MPa for 2.5×10^4 cycles.

damaged by oxidation in the places near the edge of specimen or close to the large pores. However, such a severe oxidation is not widely distributed in the specimens tested in air, because the fill of the

glassy phases in the cracks prohibits the diffusion of oxygen along the crack paths. Around the large pores between fiber bundles, the matrix is the last coating by the pure SiC (light colored layer), the

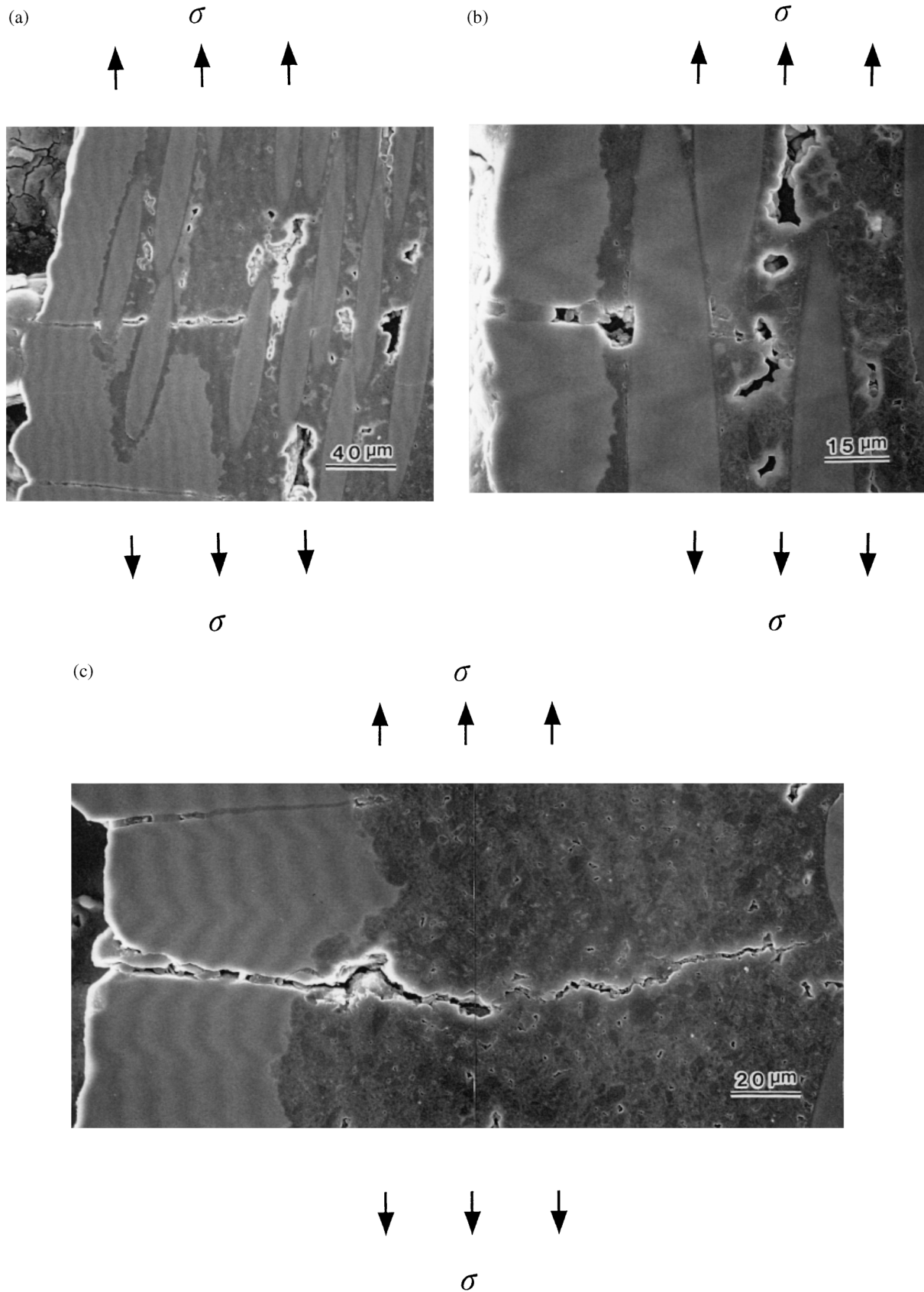


Fig. 9. Cracks in a crept specimen of the Enhanced SiC/SiC fatigued in air at 1300°C and $75\ \text{MPa}$ for 53 h: (a) cracks bridged by 0° fibers; (b) the interfaces between fibers and the matrix being debonded (oxidation damage on fibers can be seen); (c) crack propagation in the matrix.

same as the matrix in the Standard SiC/SiC. Crack propagation is very straight in this layer, but deflected or discontinuous in the inner matrix (dark colored zone), as shown in Fig. 9(c).

The fiber pullout under fatigue is longer than that under creep (Fig. 10). The longer fiber pullout of fatigue fracture (Fig. 10) demonstrate that the cyclic unloading promotes the debonding of the interfaces by decreasing the interfacial sliding resistance.^{8,12} On one hand, as the debonding length increases, the associated model II crack (interface crack) propagation will dissipate more energy. Thus, at the same applied load, the net model I crack-tip driving force decreases due to the consumption of energy by debonding of the interfaces, leading to lower crack growth rate under fatigue. On the other hand, the increase of the debonding length should increase the contribution of fibers creep on the crack growth and the probability of fiber fracture depending on gage scale.¹² This promotes matrix crack growth and the fracture of the composite. Creep of fibers relaxes the bridging force. Therefore, creep of fibers will decrease the debonding length.

4 Conclusion

Creep strain rates of Hi-NicalonTM/SiC in air were similar to those of Enhanced SiC/SiC, but much lower than those of Standard SiC/SiC. Consequently, the time to rupture at a given stress in Hi-NicalonTM/SiC was similar to that in Enhanced SiC/SiC, but much longer than in Standard SiC/SiC. Fatigue resistance of Hi-NicalonTM/SiC was similar to that of Enhanced SiC/SiC, but much better than Standard SiC/SiC. It should be pointed out that no data of creep and fatigue of the CVI SiC matrix are available in literatures. To understand creep and fatigue mechanism in SiC/SiC, the mechanical behavior of the matrix is needed to be studied in the future.

Acknowledgements

The authors are very grateful for the assistance of Mr S. Ogawa, Mr Y. Nagano and Dr J. Cao. This work is a part of the automotive ceramic gas turbine

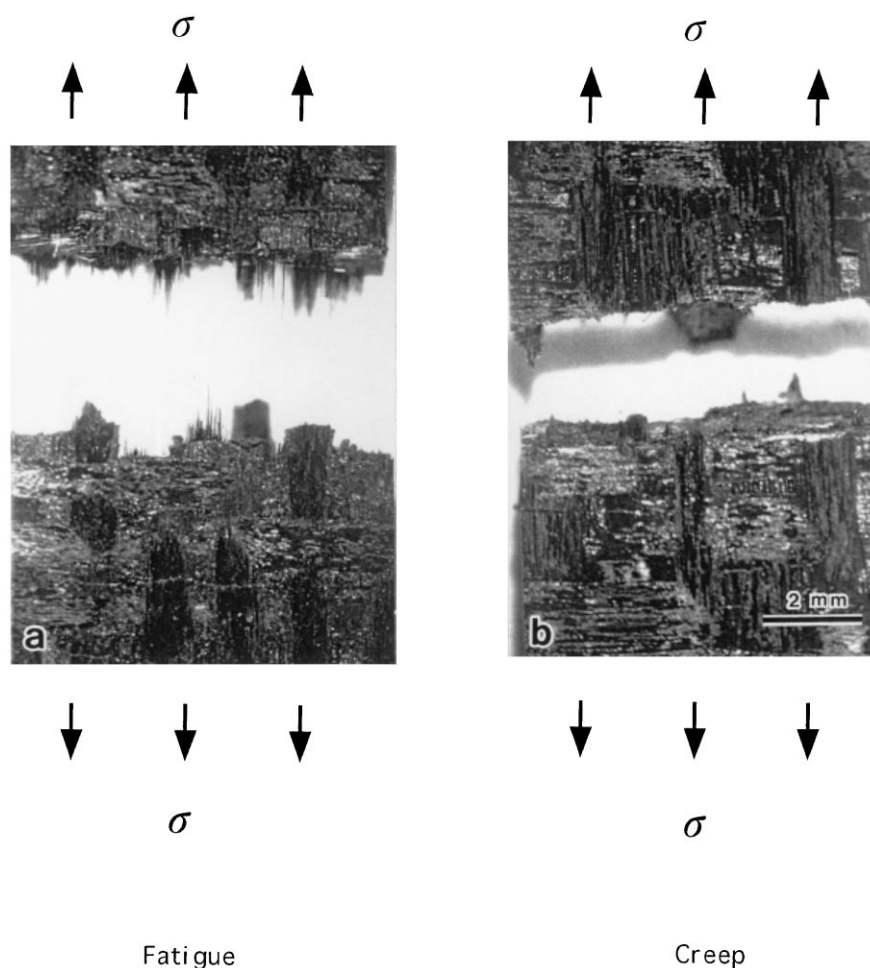


Fig. 10. Fiber pullout in Hi-NicalonTM/SiC at the maximum stress of 120 MPa and 1300°C in air: (a) fatigue; (b) creep.

development programs conducted by Petroleum Energy Center (PEC) in Japan.

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