Fatigue evolution of cyclic loaded LSI-based C/C-SiC composites

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Abstract

The fatigue behavior of plain-weave Cf/C–SiC composites prepared by liquid silicon infiltration (LSI) was studied under cyclic tensile stress at room temperature. The specimens were loaded with stress levels of 83% and 90% of the mean static tensile strength for 105cycles. The cross-sections and fracture surfaces of the fatigued specimens were examined by optical microscopy (OM) and scanning electron microscopy (SEM), respectively. The results show that the specimens can withstand 105 fatigue cycles with a stress level of 90% of the static tensile strength. The retained strengths after fatigue for 105 cycles with stress levels of 83% and 90% are about 19% and 11% higher than the static tensile strength. Due to the observation of the microstructures a relief of the thermal residual stress (TRS) caused by stress-induced cracking is probably responsible for the enhancement. Furthermore, the fracture surfaces indicate that the fatigue stress results in interfacial debonding between the carbon fiber and matrix. Additionally, more single-fiber pull out was observed within the bundle segments of fatigued specimens.

Keywords

C/C–SiC; Tensile fatigue; Matrix cracking; Residual strength.

1. Introduction

Currently, ceramic matrix composites (CMC), such as C/C–SiC,is one of the most promising candidates for high temperature structure materials [1-2]. The LSI-based carbon fiber C/C–SiC composites can be manufactured with a suitable efficiency and relatively low cost [3–5]. Short fiber reinforced C/C–SiC (Csf/C–SiC) with resin-derived carbon matrix are already introduced in commercially available advanced friction systems, especially for sedan cars [6]. As one of the most common type of fiber reinforcement, the plain-weave carbon fabric is widely applied for C/C–SiC composites (2D C/C–SiC). These 2D C/C–SiC composites are successfully developed and find their applications in different structural systems, including heating shields and high performance braking systems [6-7]. Generally, structures made of CMC will inevitably suffer fluctuations in loads during operation in the most potential applications. The cyclic stress could lead to the degradation of strength and catastrophic failures. Hence, it is essential to get a comprehensive investigation for the mechanical

degradation due to fatigue for C/C–SiC composites under dynamical loads. So far, the attention was mainly focused on the fatigue behavior of Cf/SiC or SiCf/SiC composites prepared by CVI [8–10]. It has also been reported that the strength of CMC prepared by CVI could be enhanced after fatigue cycling [11,12].Nevertheless, the origin of this behavior is still not well understood, but the probable fatigue mechanism for CMC including the generation and propagation of cracks, interfacial-debonding/sliding, fiber rupture, etc. were proposed [13–17]. Generally, the properties of CMC vary with the different microstructures including fiber architectures, matrix content, fiber/matrix interface, etc.Although, the fatigue research of CMC has been carried out for years, there is almost no investigation about the fatigue behavior of LSI derived C/C–SiC composites with phenolic resin-derived matrix. The aim of this work is to study the damage evolution and the effects on the retained tensile strength of these C/C–SiC composites under cyclic stresses, in order to promote the further design and application of C/C–SiC components with higher reliability. Therefore, the tensile fatigue behavior (S–N curves),the fatigue damage, residual mechanical properties, microstructures and fracture surfaces were investigated.

2. Experimental

The open porosity and bulk density of the as-prepared C/C–SiC composites were measured by Archimedes method in distilled water according to DIN EN 1389. All samples were characterized as-fired without surface polishing. The surface roughness (Ra) was measured by a perthometer (Garant Perthometer Company). According to the DIN EN 658-1, the static tensile tests were carried out with dog-bone shaped specimens prepared by wire eroding, as shown in Fig. 1.

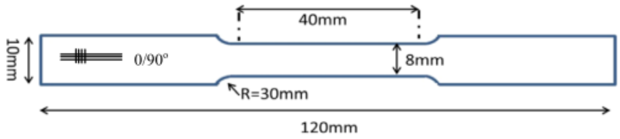

Fig. 1. Geometry of the tension specimens.

3. Results and discussion

Fig. 2(a) shows the tensile fatigue life S–N curve of C/C–SiC composites at room temperature. It is clearly visible that the asprepared C/C–SiC composites exhibit a high resistance to fatigue.When the applied stress level is 95%, three-fifth of the test samples failed before achieving the desired 105 cycles. However,when the applied stress level decreases to 90% and 83%, all samples (5 samples per stress level) exhibit a run-out after 105 cycles. Hence, the fatigue limit of C/C–SiC composites is about 90% of the initial tensile strength. The hysteresis loop modulus(HLM) versus cycles under 83% and 90% of tensile stress are shown in Fig. 2(b). The initial decrease in HLM correlates with the rapid initiation and growth of matrix cracks and fiber breakage. Subsequently, the HLM is stabilized, which indicates fewer crack formations and propagations (near crack saturation)during fatigue. Higher applied fatigue stress introduces more fatigue damages which are corresponding to a decreased HLM.Generally, the HLM could fluctuate slightly due to the opening–closing effects of cracks. Fig. 2(c) shows four stress–strain curves of the C/C–SiC composites after 11104, 33262, 55493 and 100000 cycles under the fatigue stress of 83%. The modulus E* in the 11,104th hysteresis loop, calculated by a strict linear-fit as the dash line shown in Fig. 2(c), is approximately 20.4 GPa. With increasing fatigue cycles up to 55493 and 100000 cycle, the hysteresis loops show no significant variation in the modulus E*.With the increasing number of fatigue cycles, there are only slight changes of the hysteresis loop areas, which indicates a near crack saturation. Due to the strong fiber/matrix bonding, the interfacial degradation is low and almost independent of the fatigue cycles,resulting in minimal hysteresis and accumulation of permanent strain.

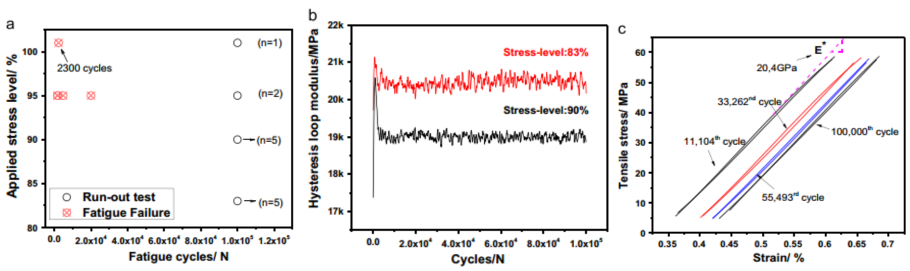

Fig. 2. (a) Tensile fatigue life S–N curve of the C/C–SiC composites; (b) Hysteresis modulus versus cycles; (c); Stress–strain hysteresis loop with stress level of 83%. The horizontal arrows indicate the samples that achieved all 105 cycles, n in the bracket indicates the number of valid samples and E* is the hysteresis loop modulus.

Fig. 3 shows the microstructure of C/C–SiC before/after fatigue. Cracks which are shown in Fig. 3(a) result from the mismatch of the coefficient of thermal expansion (CTE) between the fibers and matrix, during the cooling procedure from processing temperature to room temperature. In general,these cracks can propagate towards to the 901 orientated fibers when fatigue stress is applied. Generally, the growth rate of the cracks in Fig. 3(a) obey the Paris equation which describes a stable and essentially linear process of crack formation and can be modeled by power law equations. Fig. 3(b) shows the typical optical microstructures of C/C–SiC after a fatigue load of 105 cycles with a stress level of 83%. The cracks fully bridge adjacent 901 orientated fiber bundles in the fatigued sample.

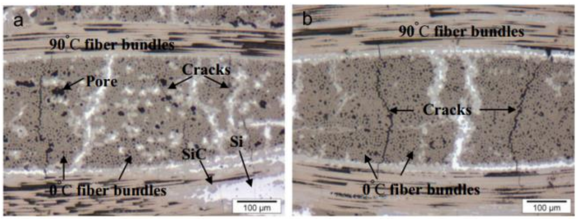

Fig. 3. Typical optical microstructures of C/C–SiC before/after fatigue, (a) Sample before fatigue; (b) Sample after fatigue with stress level 83% after 105 cycles.

The typical tensile stress–strain curves of C/C–SiC composites are plotted in Fig. 4. The starting point of the fatigued sample is artificially shifted to a strain of 0.1% to enhance the readability of the figure.

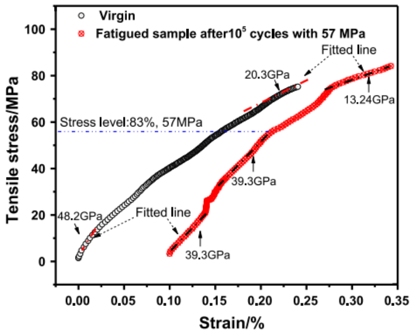

Fig. 4. Typical tensile stress–strain curves of C/C–SiC composites before and after 105 cyclic loading with stress level of 83%.

The elastic modulus of static tensile test samples decreases with the increasing of the previous applied fatigue stress. In general, the lower modulus after fatigue is a result of the higher amount of cracks within the composites. Fig. 5 shows the cracks distribution in C/C–SiC before and after the fatigue test.

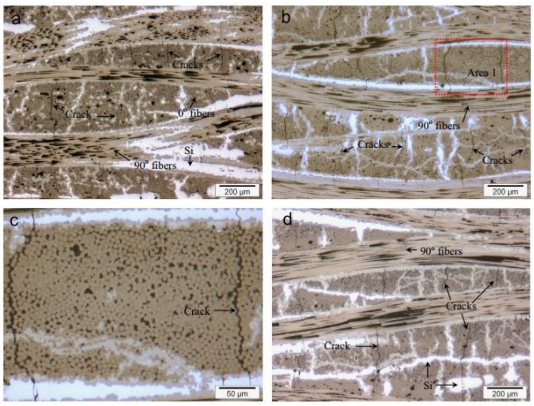

Fig. 5. Crack density and distribution in the C/C–SiC composites, (a) Microstructure before fatigue; (b)microstructure after fatigue with a stress level of 83%;(c) microstructure after fatigue with a stress level of 90%.

Based on the comparison of cracks in Fig. 3 and Fig. 5, Fig.6 displays a sketch of possible TRS in C/C–SiC before/after fatigue and the influences of the TRS on the crack propagation.As shown in Fig. 6(a), the TRS caused by the mismatch of CTE, which results in tensile stresses within the matrix and

Compression stresses within the fiber bundles. In general,fatigue loads could activate the initiation and extension of multiple cracks. Therefore, TRS in fiber bundles and matrix as illustrated in Fig. 6(b) were released. The removal of the TRS in composites after the fatigue tests enables more fiber bundles to carry the applied load uniformly and simultaneously. Hence,the increased number and width of cracks (see Fig. 5) is one of the main reasons for the increase of the tensile strength.

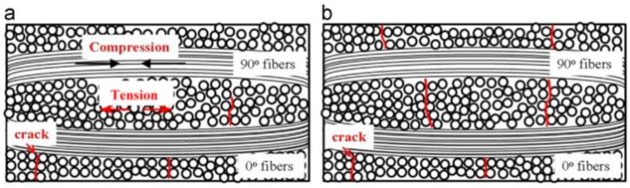

Fig. 6. Schematic sketch of the cracks caused by thermal residual stresses in C/C–SiC composites before/after fatigue, (a) C/C–SiC composites without fatigue load;(b) C/C–SiC composites after fatigue load. The black arrows in Fig. 6(a) indicate the compressive stresses, and the red arrows indicate the tensile stresses.

Fig. 7 presents typical fractured surfaces of the virgin and the fatigued C/C–SiC composites after the tensile tests. As shown in Fig. 7(a) and (b), the tensile damage mode including the cracking in 01 fiber bundles and pull-out of 901 fiber bundle segment could be observed in the fractured surface of the virgin sample. Moreover, as shown in Fig. 7(b), there are compact fiber bundles with few single fiber pull-outs. Furthermore, only few interfacial detaching between fiber/matrix can be observed in the pull-out fiber bundle segment. Compared with the fracture of the virgin sample, the debonding in 01 fiber bundles and the pull-out of 901 fiber segments as shown in Fig.7(c) and (d), can be observed in the fracture surface of prefatigue sample as well. However, the fracture surface as shown in Fig. 7(d) is obviously different from the virgin sample.Definitely, many single fibers and interfacial debonding can be observed in the pulled-out fiber bundle segments as shown in Fig. 7(d). Generally, more pull-out of single fiber, indicates that higher interfacial friction energy is required to break the interfacial bonding between fiber/matrix, which leads to a higher tensile strength.

Fig. 8 shows the typical interfaces between carbon fiber and matrix in a virgin and a pre-fatigued sample. As shown in Fig. 8(a),the bonding between fiber/matrix is still intact in the virgin sample.Fig. 8(b) exhibits the interface status of the pre-fatigue sample. The interfacial debonding, which is mainly caused by fatigue loading,can be observed between the fiber and matrix. Additionally, the interfacial debonding could affect the enhancement of the retained strength. When the tensile stress is applied, there are two major phenomena for a further crack extension: crack deflection and crack propagation. Both of them are involved with the state of the stress concentration and the interfacial bonding in the composites.

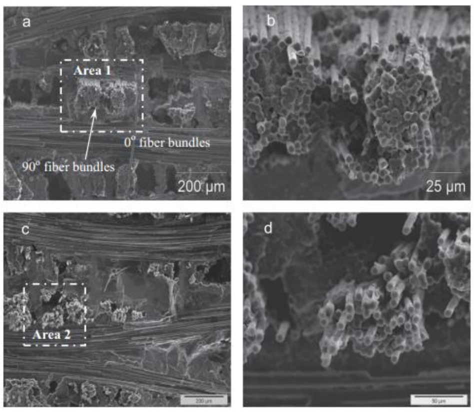

Fig. 7. The fracture surface of C/C–SiC composites (SEM images, topography contrast), (a) The virgin sample; (b) The magnification of Area 1; (c) The fatigued sample after 105 cycles with 83% stress level; (d) The magnification of Area 2.

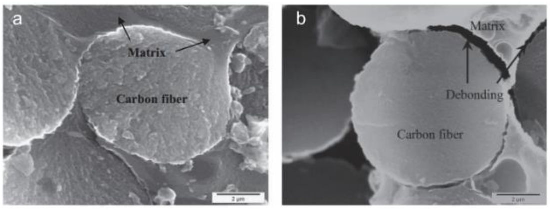

Fig. 8. SEM image of the interface between carbon fiber and matrix in virgin and pre-fatigued sample, (a) Intact interface in the virgin sample; (b) Interfacial debonding in the pre-fatigued sample.

When a virgin C/C–SiC sample suffers tensile load, high stress concentrations in the crack tip probably lead to the crack penetration from the integrated interface into the fibers as illustrated in Fig. 9(a). However, when the virgin C/C–SiC sample suffers a previous fatigue load, the interfacial debonding occurs, as shown in Fig. 8(b). The interfacial debonding decreases the stress concentration to a certain extent. After applying a static load, a new crack coalesces with the already existed interfacial crack, as illustrated in Fig. 9(b). Therefore, the interfacial debonding ascribed to fatigue

Load can result in more single fibers pull-out (see Fig. 7(d)). Thus,the fibers can carry higher loads. Furthermore, the interfacial debonding is another reason for the increase of the measured tensile strength after the fatigue test.

4. Conclution

The tensile fatigue behavior of plain-weave Cf/C–SiC composites prepared by liquid silicon infiltration was studied at room temperature. The S–N curve was acquired by a series of fatigue tests with various stress levels, regarding to the static stress strength. The fatigue damage was studied, based on the observation of microstructures, fractured surfaces and the residual static tensile strength.

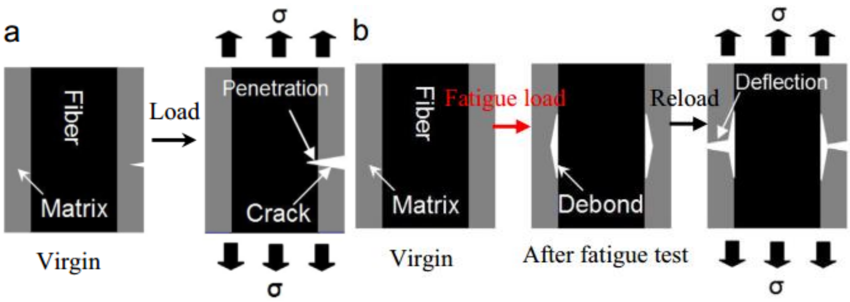

Fig. 9. Crack propagation schematic in basic cell of the C/C–SiC composites, (a) Crack penetration in a virgin sample; (b) Crack deflection in a pre-fatigued sample.

Acknowledgements

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References

[1] R. Naslain, Design, preparation and properties of non-oxide CMCs for application in engines and nuclear reactors: an overview, Compos. Sci.Technol. 64 (2) (2004) 155–170.
[2] W. Krenkel, Carbon fiber reinforced CMC for high-performance structures, Int. J. Appl. Ceram. Technol. 1 (2) (2004) 188–200.
[3] W. Krenkel, F. Berndt, C/C–SiC composites for space applications and advanced friction systems, Mater. Sci. Eng.: A 412 (1–2) (2005) 177–181.
[4] Z. Li, P. Xiao, X. Xiong, B.-y Huang, Preparation and tribological properties of C fibre reinforced C/SiC dual matrix composites fabrication by liquid silicon infiltration, Solid State Sci. 16 (2013) 6–12.

[5] Z. Li, P. Xiao, B.-g Zhang, Y. Li, Y.-h Lu, Preparation and tribological properties of C/C–SiC brake composites modified by in situ grown carbon nanofibers, Ceram. Int. 41 (9, Part B) (2015) 11733–11740.

[6] W. Krenkel, Carbon fibre reinforced silicon carbide composites (C/SiC,C/C–SiC), in: N. Bansal (Ed.), Handbook of Ceramic Composites,Springer, US, 2005, pp. 117–148.

[7] H. Abu El-Hija, W. Krenkel, S. Hugel, Development of C/C–SiC brake pads for high-performance elevators, Int. J. Appl. Ceram. Technol. 2 (2) (2005) 105–113.

[8] K.G. Dassios, D.G. Aggelis, E.Z. Kordatos, T.E. Matikas, Cyclic loading of a SiC-fiber reinforced ceramic matrix composite reveals damage mechanisms and thermal residual stress state, Compos. Part A: Appl.Sci. Manuf. 44 (2013) 105–113.

[9] H. Mei, L. Cheng, Stress-dependence and time-dependence of the postfatigue tensile behavior of carbon fiber reinforced SiC matrix composites,Compos. Sci. Technol. 71 (11) (2011) 1404–1409.

[10] C. Liu, L. Cheng, X. Luan, B. Li, J. Zhou, Damage evolution and realtime non-destructive evaluation of 2D carbon-fiber/SiC-matrix composites under fatigue loading, Mater. Lett. 62 (24) (2008) 3922–3924.

[11] A. Morales-Rodríguez, M. Moevus, P. Reynaud, G. Fantozzi, Strength enhancement of 2D-SiCf/SiC composites after static fatigue at room temperature, J. Eur. Ceram. Soc. 27 (11) (2007) 3301–3305.

[12] C. Zhang, X. Wang, Y. Liu, B. Wang, D. Han, S. Qiao, Y. Guo, Tensile fatigue of a 2.5D C/SiC composite at room temperature and 900 1C,Mater. Des. 49 (2013) 814–819.

[13] P. Reynaud, Cyclic fatigue of ceramic-matrix composites at ambient and elevated temperatures, Compos. Sci. Technol. 56 (7) (1996) 809–814.

[14] A.G. Evans, F.W. Zok, R.M. McMeeking, Fatigue of ceramic matrix composites, Acta Met. Et. Mater. 43 (3) (1995) 859–875.

[15] G. Fantozzi, P. Reynaud, Mechanical hysteresis in ceramic matrix composites, Mater. Sci. Eng.: A 521-522 (2009) 18–23.

[16] C. Liu, L. Cheng, X. Luan, W. Zhang, C. Wang, Real-time damage evaluation of a SiC coated carbon/carbon composite under cyclic fatigue at high temperature in an oxidizing atmosphere, Mater. Sci. Eng.: A 524 (1–2) (2009) 98–101.

[17] L. Longbiao, Fatigue hysteresis behavior of unidirectional C/SiC ceramic–matrix composite at room and elevated temperatures, Mater. Sci.Eng.: A 625 (2015) 1–18.