Springer

KSME

Journal of Mechanical Science and Technology 31 (8) (2017) 3647~3651
www.springerlink.com/content/1738-494x(Print)/1976-3824(Online)
DOI 10.1007/s12206-017-0702-3

Fracture behavior of C/SiC composites at elevated temperature †

Dong Hyun Yoon[1], Jeong Won Lee[1], Jae Hoon Kim[1,*], Ihn Cheol Sihn[2] and Byung Joo Lim[2]

[1]*Departtment of Mechanical Engineering, Chungnam National University, Daejeon 34134, Korea*
[2]*Dai-Yang Industries Co., Icheon-si, Gyeonggi-do 17383, Korea*

(Manuscript Received February 1, 2017; Revised March 27, 2017; Accepted April 11, 2017)

Abstract

The fracture behavior of carbon fiber-reinforced silicon carbide (C/SiC) composites used in rocket nozzles has been investigated under tension, compression, and fracture conditions at room temperature, 773 K and 1173 K. The C/SiC composites used in this study were manufactured by liquid silicon infiltration process at ~1723 K. All experiments were conducted using two types of specimens, considering fiber direction and oxidation condition. Experimental results show that temperature, fiber direction, and oxidation condition affect the behavior of C/SiC composites. Oxidation was found to be the main factor that changes the strength of C/SiC composites. By applying an anti-oxidation coating, the tensile and compressive strengths of the C/SiC composites increased with temperature. The fracture toughness of the C/SiC composites also increased with increase temperature. A fractography analysis of the fractured specimens was conducted using a scanning electron microscope.

Keywords: Carbon fiber-reinforced silicon carbide (C/SiC) composite; Fracture toughness; Liquid silicon infiltration (LSI); Oxidation; Residual thermal stress; Scanning electron microscope (SEM); Uniaxial compression; Uniaxial tension

1. Introduction

In a solid propulsion system, the rocket nozzle is exposed to high-temperature combustion gases. Therefore, selecting an appropriate material that can help yield high performance under high temperatures is important. In view of structural integrity, several studies have been conducted on the application of carbon fiber-reinforced silicon carbide (C/SiC) composites to rocket nozzles. High-temperature properties of C/SiC composites have been recently investigated. Tao et al. conducted quasi-static and dynamic uniaxial compressive tests on C/SiC composites under temperatures ranging from 293 K to 1273 K. The results showed that temperature and strain rate affected the compressive behavior of 2D C/SiC composites to a great extent [1]. Mao et al. applied a high-temperature digital image correlation technique to an in situ fracture experiment of a C/SiC composite. They evaluated fracture toughness changes at room temperature (R.T.) and at 1600 °C [2]. Although the properties of C/SiC composites have been widely investigated, the number of literature focusing on the directional properties and oxidation effects on the strength of C/SiC composites is limited. These points cannot be overlooked when considering its applications to actual structures. In this study, the fracture characteristics of C/SiC composites were investigated under tension, compression, and cracking conditions at various temperatures, considering fiber direction and oxidation conditions. A fractography analysis of the fracture surface was conducted using a Scanning electron microscope (SEM).

2. Experimental procedures

2.1 Preparation of composite

C/SiC composites were manufactured through a Liquid silicon infiltration (LSI) process by Dai-Yang Industries Co. Liquid silicon melted from silicon granules infiltrated into the open pores and cracks of the C/C preform and reacted with the carbon fiber surface and carbon matrix to form a SiC matrix. Coal-tar pitch was used to densify the C/C preforms fabricated by needle punching. During LSI, the processing temperature reached 1723 K, at which the silicon melted. Compressive and tensile specimens were prepared according to ASTM standards [3, 4]. Compact specimens were designed to evaluate the fracture toughness of C/SiC composites [5]. A pre-crack was formed at the notch tip of a compact tension specimen using a saw to introduce natural cracks. Tensile, compressive, and compact specimens are shown in Fig. 1. The specimens used to verify the directional properties of C/SiC composites were in the Horizontal direction (HD) and Vertical direction (VD) with fiber directions perpendicular and parallel to the test load direction, respectively.

*Corresponding author. Tel.: +82 42 821 6645, Fax.: +82 42 821 8894
E-mail address: kimjhoon@cnu.ac.kr
† This paper was presented at the ACEM 2016, Ramada Plaza Jeju Hotel, Jeju, Korea, November 13-16, 2016. Recommended by Guest Editor Chongdu Cho.

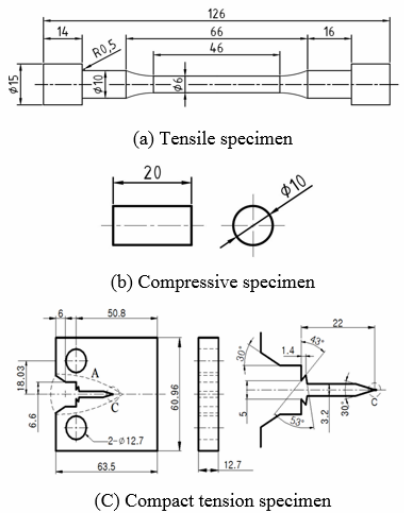


Fig. 1. Tensile, compressive, and compact tension specimens.

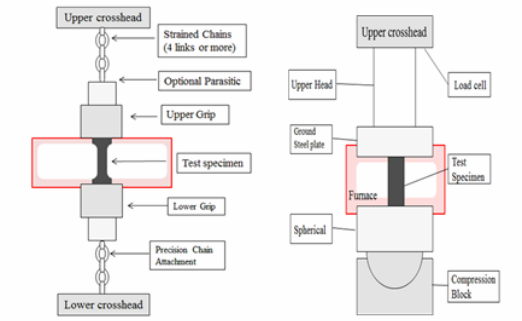


Fig. 2. Schematics of load systems for the uniaxial test.

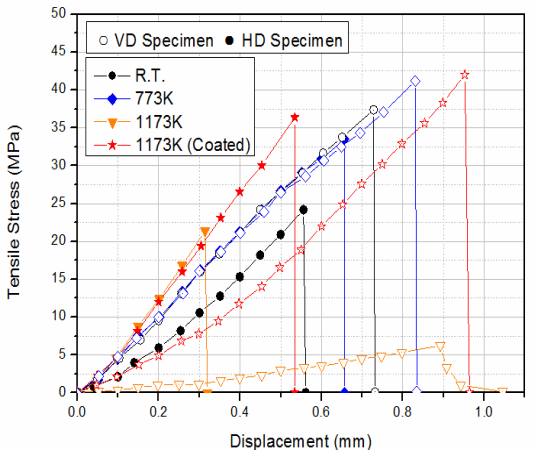


Fig. 3. Stress–displacement curves for C/SiC composites subjected to tensile loads at various temperatures.

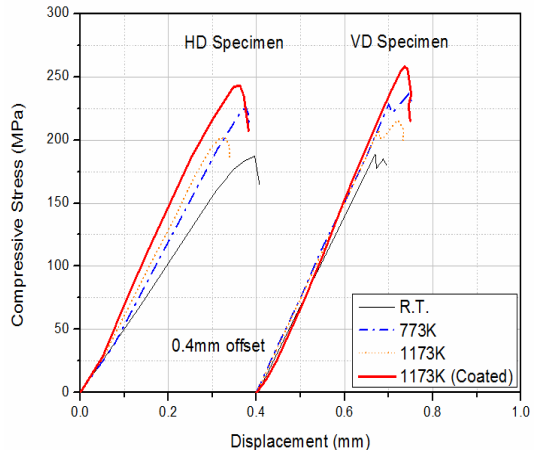


Fig. 4. Stress–displacement curves for C/SiC composites subjected to compressive loads at various temperatures.

2.2 Experimental procedure

Tests were conducted according to ASTM standards using an MTS 810 universal testing machine. For the uniaxial condition, compressive and tensile tests were conducted with a spherical block and chain grip, respectively. Fig. 2 shows the schematics of the test setups. Mode I fracture toughness tests were performed according to ASTM E399. The test rate was maintained at 0.1 mm/min, and temperatures were maintained at 298, 773 and 1173 K. After the specimen was heated to the test temperature, it was held for an additional 15 min to achieve uniform temperature distribution. Certain specimens were coated with oxidation-resistant coating to verify the effects of oxidation on the strength of C/SiC composites.

3. Results and discussion

3.1 Tensile behavior under temperature change

Fig. 3 shows a tensile stress–displacement curve of the C/SiC composites at various temperatures. The curves show a linear behavior until the failure of the specimen. The strength of the C/SiC composite increased with the increase of temperature from R.T. to 773 K. However, when the temperature increased above 773 K and reached 1173 K, the stress in the materials decreased because of oxidation effects. Thick oxide layers were found on the specimens that were tested at 1173 K, which was a major contributing factor to the diminished tensile strength. Carbon fibers could not play a role in strengthening C/SiC composites because of oxidation. In view of fiber direction, displacement of the HD specimen was smaller (approximately 35 %) than that of the VD specimen. The failure of the HD specimens at an interface reveals a relatively small displacement between the carbon fibers and SiC matrix.

3.2 Compressive behavior under temperature change

Fig. 4 shows the compressive stress–displacement curves. The results of the VD compressive test specimen have been moved 0.4 mm to the right to avoid confusion with those of the HD specimen. The curves exhibit linear behavior before yielding or failure and exhibit nonlinear behavior after initial

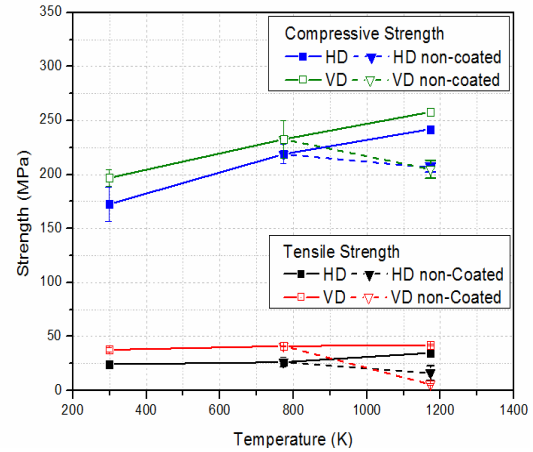


Fig. 5. Compressive and tensile strengths at various temperatures.

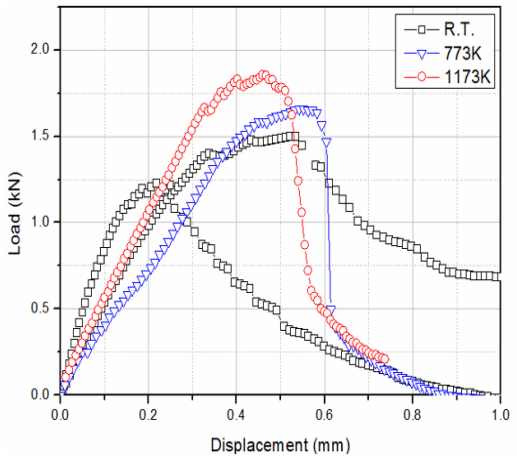


Fig. 6. Load–displacement curves for VD fiber CT specimens of C/SiC composites at various temperatures.

fracture. The strength of C/SiC composites increased with the increase of temperature from R.T. to 773 K. However, when the temperature increased up to 1173 K, the composites were oxidized, and their strength decreased. Oxide layers were found on the specimens tested at 1173 K, which is similar to the observations in tensile tests conducted at high temperatures. The fiber direction did not have a pronounced effect on the compressive strength of the C/SiC composites. The initiation of failure led to a fluctuation in the strength of the VD specimens, because the matrix interrupted the crack propagation during failure.

3.3 Oxidation effects on C/SiC composite

The results of tensile and compressive tests showed the decrease of strengths due to oxidation at 1173 K. Certain tests were conducted with oxidation-resistant coatings at 1173 K to verify the effects of oxidation on strength. In comparison with uncoated specimens, the tests with coated specimens showed higher tensile and compressive stress in the C/SiC composites (Figs. 3 and 4). In addition, their strength was higher than that at 773 K. Fig. 5 shows the tensile and compressive strengths at various temperatures. Without oxidation, the tensile and compressive strengths of the C/SiC composites would have increased with temperature.

3.4 Fracture toughness of C/SiC composites

Fracture toughness tests were conducted at various temperatures. In the high-temperature tests, oxidation-resistant coatings were applied to the specimens. Fig. 6 shows the load–displacement curves of compact tension specimens. In fracture toughness analysis, the fiber direction was not considered because initial cracks on HD specimens easily and randomly propagated along the fiber axis without any resistance [6]. The fracture loads increased with temperature. An increase in strength was observed in previous tensile and compressive tests. This trend was also observed in the toughness test. Mode

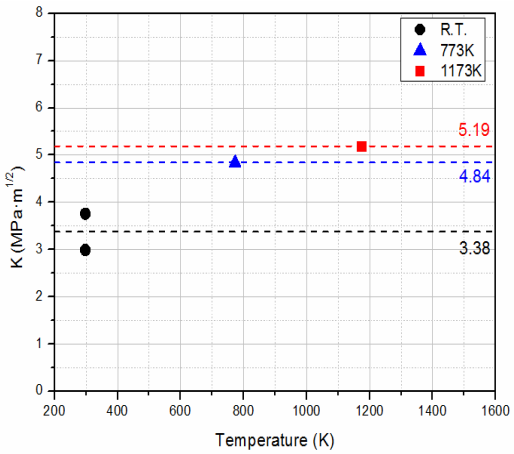
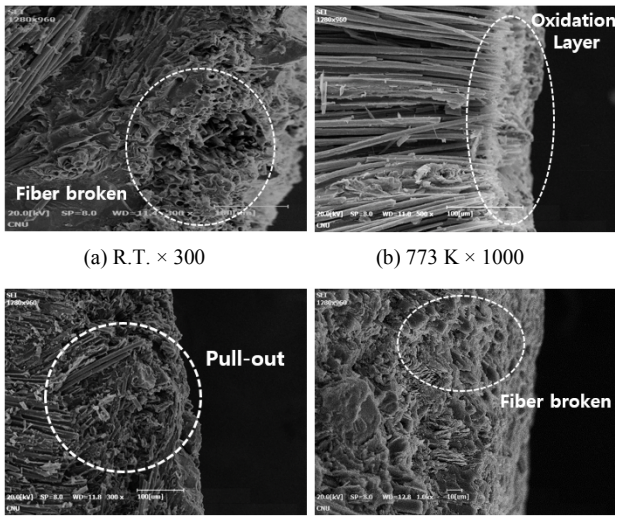


Fig. 7. Fracture toughness K values at various temperatures.

I fracture toughness value K was calculated according to the ASTM E399 standard using the fracture load value. The K values calculated from the fracture load also increased with temperature. Fig. 7 shows the changes in the value of the fracture toughness K with temperature variations.

3.5 Effects of temperature on strength

The C/SiC composite was manufactured by the LSI at a high temperature (1723 K) and cooled to R.T. During cooling, a differential coefficient of thermal expansion between the carbon fiber and SiC matrix generated Thermal residual stress (TRS). Some studies performed measurements and calculations of the TRS generated in the C/SiC composite [7]. From the previous study, the tensile TRS on the interface of the composite was calculated from the decrease in temperature. Microcracks in the SiC matrix could be opened by tensile TRS. Additionally, the TRS weakened the bonding of the interface between the fiber and matrix. Consequently, the TRS gener-



(a) R.T. × 300 (b) 773 K × 1000

(c) 1173 K × 300 (d) 1173 K × 500

Fig. 8. SEM images of fracture surfaces tested at R.T. (a); 773 K (b); and 1173 K (c, d) with various magnifications.

ated in the composite by the temperature decrease is a key factor contributing to the decreased strength at a low temperature. The TRS generated in the composite decreases with the increase in temperature. Therefore, the strength of the C/SiC composites also increases with the temperature because of the decreased TRS. In particular, the tensile test of HD specimens shows a greater increase in strength with the increase in temperature compared with that of VD specimens, because the HD tensile test directly reflects the reinforced interfacial strength due to the decrease in the TRS. The effects of TRS on the interface bonding can be observed experimentally through the difference in magnitudes of strength between the HD and VD tensile tests.

3.6 Microstructural observations by SEM analysis

Fig. 8 shows the fracture surfaces of the tensile specimens tested at various temperatures. The fracture surfaces of the tested HD specimens show only the layers of the chopped fibers. Fig. 8(a) shows a long carbon fiber pulled out from a composite that was tested at R.T. However, short or chopped fibers can be found on the fracture surfaces tested at elevated temperatures (Figs. 8(b) and (c)), which explains the increase of interfacial bonding strength with temperature and supports the ideas of the analysis conducted in Sec. 3.5. Oxidation layers can be observed in Fig. 8(d). Oxidation results in sharp and fine carbon fibers and causes strength reduction in C/SiC composites.

4. Conclusions

The fracture behavior of C/SiC composites used in rocket nozzles of a solid propulsion system was investigated in this study. Temperature, oxidation condition, and direction of the fiber were considered as variables in the tensile, compressive, and fracture toughness tests. The conclusions drawn from this study are as follows.

(1) The tensile and compressive strengths of the composites increased with temperature until 773 K; however, oxidation at 1173 K reduced the strengths.

(2) Without the oxidation effect, the tensile, compressive, and fracture strengths of the C/SiC composites continuously increased with temperature, and mode I fracture toughness K also increased.

(3) Under the processing temperature, the strength increased with temperature because of the TRS generated from the cooling of composites between the carbon fiber and SiC matrix.

(4) With regard to the direction of the fiber, HD specimens showed smaller displacement and strength than VD specimens under tensile conditions. However, changing the fiber direction had a negligible effect on displacement and strength in the compressive condition.

(5) Fractography analysis proved the variations in interfacial bonding strength with a change in temperature, which is evident in the different lengths of carbon fibers pulled at R.T. and other elevated temperatures.

Acknowledgment

This study was supported by Dai-Yang Industries Co., Korea.

References

[1] T. Suo, X. Fan, G. Hu, Y. Li, Z. Tang and P. Xue, Compressive behavior of C/SiC composites over a wide range of strain rates and temperatures, *Carbon Letters*, 62 (2013) 239-249.

[2] W. G. Mao, J. Chen, M. S. Si, R. F. Zhang, Q. S. Ma, D. N. Fang and X. Chen, High temperature digital image correlation evaluation of in-situ failure mechanism: An experimental framework with application to C/SiC composite, *Material Science and Engineering: A*, 665 (2016) 26-34.

[3] *ASTM Standard C695-15*, Standard test method for compressive strength of carbon and graphite, ASTM International (2015).

[4] *ASTM Standard C749-15*, Standard test method for tensile strength of carbon and graphite, ASTM International (2015).

[5] *ASTM Standard E399-12*, Standard test method for linear-elastic plane-strain fracture toughness KIC of metallic materials, ASTM International (2012).

[6] R. Inoue, J. M. Yang, H. Kakisawa and Y. Kagawa, Mode I fracture toughness of short carbon fiber-dispersed SiC matrix composite fabricated by melt infiltration process, *Ceramic International*, 39 (2013) 8341-8346.

[7] H. Mei, Measurement and calculation of thermal residual stress in fiber reinforced ceramic matrix composite, *Composites Science and Technology*, 68 (15-16) (2008) 3285-3292.

Dong Hyun Yoon received his B.S. degree in Mechanical Design Engineering from Chungnam National University, Korea in 2016. His research interests are in the area of fracture mechanics, fatigue behaviors, and composite materials.

Jae Hoon Kim received his B.S. degree in Precision Mechanical Engineering from Chungnam National University, Korea in 1980. He received his master's and Ph.D. degrees in Mechanical Engineering from Chungnam National University, Korea in 1982 and 1989, respectively. He is currently a Professor at Chungnam National University, Korea. Prof. Kim's research interests are in the area of fracture mechanics, fatigue behaviors, and composite materials.