Tensile strength degradation of a 2.5D-C/SiC composite under thermal cycles in air

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Abstract

The present work investigated the effects of the thermal cycles in air on the tensile properties of a 2.5 dimensional carbon fiber reinforced silicon carbide composite (2.5D-C/SiC) in weft and warp direction, prepared by low-pressure chemical vapor infiltration. The composite was exposed to different thermal cycles between 900 °C and 300 °C in air. The fracture morphologies of the failed composite were examined by a scanning electron microscope to investigate the underlying damage mechanisms. It is found that the composite can retain its tensile strength within 40 thermal cycles. Comparatively, the modulus of 2.5D-C/SiC decreases with increasing thermal cycles. Extensive pullout of fibers on the fractured surface and interface debonding suggest that the damage caused by the thermal cycles involves weakening of the bonding strength of coating/composite and fiber/matrix. The above damage supply the channel for the oxidation of PyC and carbon fibers in the composites. In addition, the degradation in warp direction is faster than that in weft direction.

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1 Introduction

Carbon fiber reinforced silicon carbide composites (C/SiC) overcome the brittleness and low reliability of monolithic ceramics, and therefore, widely used as structural materials in different fields including advanced engines, gas turbines for power/steam co-generation, heat exchangers, as well as future nuclear reactors [1-4]. Components made of C/SiC are expected to experience the thermal cycles (sudden changes in temperature) during the above mentioned applications. The thermal cycles can lead to damage accumulation and eventually failure. Hence, it is essential to clearly understand the effects of the thermal cycles on the mechanical properties of C/SiC.

Continuous fiber reinforced ceramic composites display much more complex behaviors than monolithic ceramics under thermal transient conditions [5]. The presence of fibers appears to improve the resistance to the thermal cycles by raising the predicted critical temperature somewhat [6]. The fiber reinforcement might significantly influences the crack initiation sites and propagation paths in the composites under thermal shock [7]. Meanwhile, additional thermal stresses can be introduced because of the thermal expansion mismatch between fibers and matrix. Moreover, many factors, such as the type of fiber/matrix, fiber architecture, interfacial characteristics, and matrix-forming processes, also result in the mechanical property degradation during the thermal cycles [8-11]. For example, it is indicated by Udayakumar *et al* that a thinner BN interface could improve resistance to the thermal cycles of SiC/SiC composite [12]. Therefore, the resistance to thermal cycles of continuous fibers reinforced ceramic composites cannot be explicitly evaluated in simple ways used for monolithic ceramics [13]. The abilities to withstand

thermal cycles were always evaluated by measuring the residual strength or modulus, or observing the damage within the composites.

The thermal cycles lead to kinds of damages in the composites, including matrix cracks, interface bonding and fibers oxidation. It was found by Yin et al that there is a critical temperature difference ΔT_c for a 3D-C/SiC. The matrix cracks close to the surface appeared even if the temperature difference than ΔT_c . However, the surface cracks does not propagate and cannot damage the strength. Above ΔT_c , the surface crack propagate inwards and lead to degradation in the strength [14]. Furthermore, the cyclic stresses could lead to cracking in coating, debonding of fiber/matrix interface, and interface sliding. In addition, the oxygen in the atmosphere could aggravate the damage by alternate oxidation between internal and external fibers in the composites. In comparison with one dimensional composite, additional matrix damage is observed in woven materials, particularly around the crossover points of the tow weaves. Matrix cracking intensifies at the crossover point, and result in debonding and delamination between the longitudinal and transverse tows [15-17]. Moreover, the fiber bundles may be split internally, often due to the coalescence of shear cracks emanating from adjacent transverse bundles [18, 19]. Previous thermal cycle processes were always cooled either by an environment (air or water) or a controlled atmosphere. The components, made of C/SiC, always touched or connected with metal parts. The conduction of heat in C/SiC through the metal parts played a more important role in the temperature change than the heat exchange by convection or radiation. There should be a different responses to the thermal cycles when the composites is cooled by the metals. Therefore, the resistance to the thermal cycles of a 2.5 dimensional carbon fiber reinforced silicon carbide composite (2.5D-C/SiC) was investigated by cooling the composite a steel plate.

In present works, the degradation in the tensile properties of a 2.5D-C/SiC composite were investigated after the composites experienced different thermal cycles between 300 °C and 900 °C in air, during which the composites was cooled by a steel plate. The effect of the thermal cycles on the mechanical properties of the 2.5D-C/SiC was characterized by measuring residual tensile strength and modulus. In addition, the fracture morphology of the specimens was examined to reveal the damage caused by the thermal cycles. Moreover, the tensile properties of the composite annealed for 10 h at 900°C were examined to further reveal how repeated heating-cooling cycles introduce additional and typical damage mechanisms.

2 Experimental Procedures

2.1 Materials

The 2.5D-C/SiC composite was reinforced by a 2.5 dimensional woven T-300 carbon fiber (1K, Toray Co, Japan) preform, shown in Fig. 1. The preform of the 2.5D-C/SiC composite is composed of layers of straight weft yarns and a set of sinusoidal warp yarns. The interlaminar strength of the composite is improved significantly due to interlaced warp yarns in the adjacent layers [20]. Each yarn contained 6000 fibers. The densities of the fiber in warp and weft direction are 10 and 3 ends/cm, respectively. The reinforcement contained 20 layers of weft carbon fiber yarns. A low-pressure chemical vapor infiltration (CVI) was used to prepare the composite. The pyrolytic carbon (P_yC) interphase with thickness of 0.2 μm was deposited on carbon fibers with C₃H₆ at 1173 K and 5 kPa. Methyltrichlorosilane (MTS, CH₃SiCl₃) was used for depositing the SiC matrix. The applied temperature, the total pressure and the molar ratio of H₂ to MTS were 1000 °C, 5 kPa and 10, respectively.

After SiC deposition, the contoured, edge-loaded test specimens, shown in Fig. 2, were

machined from the fabricated plates with 40 vol% fiber. Then, the specimens were further coated with a SiC coating of about 50 μm in thickness. The porosity is 17%, measured by mercury intrusion porosimetry on a CE Instruments Pascal 140 porosity analyzer.

Fig. 1. Schematic of reinforcement in 2.5D-C/SiC.

Fig.2. Dimension of the specimen used for the experiments (in mm).

2.2 Procedures for the thermal cycles

A tube furnace was employed to carry out the thermal cycles experiments. It consists of a cylindrical alumina tube surrounded by heating coils, which are made of Nichrome 80/20 and embedded in a thermally insulating matrix. Temperature was controlled via feedback from a Type K thermocouple. The experiments were conducted by alternating the specimens quickly between 900 $^{\circ}\text{C}$ ~300 $^{\circ}\text{C}$ in static air. Each specimen held in an alumina crucible was heated in the furnace, which was preheated to 900 $^{\circ}\text{C}$, for 10 minutes. During the heating, the specimens were positioned in the furnace center, so that the thermal gradients were minimized. No relative

movement between the specimens and the crucible took place in order to prevent any mechanical damage caused by the operation. After then, the specimens were carefully withdrawn from the furnace within 3 s and laid on an iron sheet of 100 cm×100 cm ×2 mm. After then, the specimens were cooled to 300 °C within 15 s by an iron plate. The temperature of the specimen is monitored by a Type K thermocouple during the cooling. The accuracy of the thermocouple is ± 3.6 °C. The average cooling rate was about 40 °C/s. After cooled to 300 °C, the specimen was immediately sent back to the 900 °C furnace for next heating-cooling cycle. In order to reveal the effect of the thermal cycles on the tensile properties of the composite, at least 3 specimens were investigated at predetermined cycles (20, 40 and 60).

In order to separate the effects of the thermal cycles from the oxidation on the mechanical properties of the composite, additional 3 warp specimens and 3 weft specimens were annealed at 900 °C for 10 hours in air. The annealing period is equal to the accumulated holding time at 900 °C for 60 thermal cycles.

2.3 Tests of the tensile properties and mass variation

The tensile properties were measured using a mechanical testing machine (Model: CSS-280S-100, Changchun, China) at room temperature (RT). The procedure for measuring the residual tensile properties was in accordance to ASTM 1275. The tensile experiments were conducted when the specimens, shown in Fig. 1, were pulled into the wedge of the grip. To guarantee the specimen alignment, lateral centering of the test specimen within the grip attachments was accomplished by wedge type inserts machined to fit within the grip cavity. The applied displacement rate was 0.5 mm·min⁻¹. The displacement was recorded by an extensometer (Epsilon 3542, USA) with a gauge length of 25 mm. The ultimate tensile strength (*UTS*) was

calculated by the failure force divided by the cross section of the gauge part and modulus (E) of the composite was determined from the slope ($\Delta\sigma/\Delta\varepsilon$) of the initial linear region in the stress-strain curves. The mechanical properties of the as-prepared material were determined with 5 specimens, 3 specimens for both exposed to thermal cycles and annealed.

The mass of the specimens was measured by a balance (AG204, Mettler Toledo, Switzerland) with a precision of 0.1 mg. The mass variation (Δm) was calculated according to the equation $\Delta m = (m_0 - m_I)/m_0$, where m_I stands for the mass of the specimens with 0, 1, 6, 12, 20, 40 or 60 thermal cycles and m_0 represents the mass of the as-prepared specimens.

2.4 Microstructural examination

The fractured morphologies and microstructure of the composite were examined by a scanning electron microscope (SEM, S2700, Hitachi, Japan) operated at 20 kV in backscattered electron mode. The specimens used for examining microstructure were cut from the gauge section after the specimens experienced different thermal cycles. The composition was determined by an energy dispersive X-ray spectroscope (EDS, Oxford Instruments, ISIS300, England) attached in the SEM.

3 Results

3.1 Residual Tensile Properties

Fig. 3 shows the representative tensile stress-strain curves for the warp and weft 2.5D-C/SiC specimens with 0 and 60 thermal cycles, as well as the annealed ones. It can be seen that the curves exhibit a linear response near the origin, followed by nonlinear response up to fracture. The transition from the linear to the nonlinear response is caused by the initiation of matrix cracks [21]. The nonlinear responses of stress versus strain are responsible for the damage accumulation

including matrix cracking and interfacial debonding or sliding [20]. It can be found that the strength and the modulus decreased for the 2.5D-C/SiC specimens with 60 thermal cycles in both directions.

(a)

(b)

Fig. 3. Representative tensile stress-strain curves of the warp (a) and weft (b) 2.5D-C/SiC specimens with as-received, exposed to 60 thermal cycles and annealed state.

Table 1 Tensile properties of the 2.5D-C/SiC specimen with thermal cycles and annealed specimen.

		Thermal cycle number				Annealed
		0	20	40	60	
UTS/MPa	Warp	211.5±23.1	209.5±30.6	195.4±22.3	168.2±14.7	188.5±15.3
	Weft	150.0±6.7	149.0±10.1	148.7±15.7	115.3±6.6	127.2±8.6
E/GPa	Warp	122.1±14.0	86.7±4.6	76.4±6.0	72.7±9.2	81.3±7.7
	Weft	54.7±5.3	44.0±8.2	42.9±4.1	40.7±9.4	43.8±3.6

(a)

(b)

Fig. 4. Retained tensile properties of the 2.5D-C/SiC composites with different thermal cycles.

(a) Strength, (b) Modulus

Table 1 summarizes the tensile strength and modulus of 2.5D-C/SiC with different thermal

cycles, as well as those of the annealed composite. Fig. 4 presents the variation of the tensile properties of 2.5D-C/SiC with increasing thermal cycles. It is revealed that the warp tensile strength and modulus are higher than those in weft direction. The difference is simply a direct result of more warp fibers aligned than weft fibers.

As shown in Table 1 and Fig. 4, the strength can be retained within 40 thermal cycles, and decreases significantly for more than 40 cycles. The warp strength presents somewhat different responses to the thermal cycles. It begins to degrade after about 40 thermal cycles. However, the variation of the modulus decreases with increasing thermal cycles, regardless of the directions. The retained warp and weft modulus of the 2.5D-C/SiC with 60 thermal cycles are about 63% and 82% of those for the as-prepared material, respectively. In addition, the warp modulus decreases more quickly in the first 20 thermal cycles. The result might be caused by the effect of the fiber weaving on damage initiation and delamination. If the composite is loaded in the warp direction, there are more delamination sites and potential matrix cracking for warp bundles. More the interfaces are subjected to the applied stress (i.e. there are more warp fibers in the loading direction). This, in effect, contributes to the relative rapid degradation in the warp modulus.

According to the variation of the strength and modulus, shown in Table 1 and Fig. 4, it is found that the modulus might be more sensitive to the damage caused by the first 20 thermal cycles.

3.2 Mass variations

Fig. 5 shows the mass variation of the 2.5D-C/SiC with increasing thermal cycles. A slight mass loss take place within 20 thermal cycles, and followed by a great loss after 20 thermal cycles. The mass loss is about 6.5 % for the specimen with 60 thermal cycles.

Fig. 5. Mass loss of the 2.5D-C/SiC composite determined at different thermal cycles.

3.3 Fracture Morphology

Fig. 6 displays typical tensile fracture morphologies of the weft specimens with different thermal cycles. The broken weft fiber bundles and the bare warp fibers can be found. The fracture morphology suggests that the fracture takes place at the crossover points of warp/weft bundles [21]. Similar morphologies are present on the fracture surfaces exposed to different thermal cycles, apart from the appearance of some products (bright matter), preferentially around the pores. Such product cannot be found on the fracture surface for the specimen with 20 thermal cycles in Fig. 6(a). However, it can be observed on the fractured surface that the amounts of the products increase with increasing thermal cycles, seen in Fig. 6(b) and (c). Fig. 7(a) is a magnified image of the bright region in Fig. 6(b). The dome-like columnar grains, typical form of CVIed SiC, cannot be found. The EDS result in Fig. 7(b) of the bright matter indicates that the matter is mainly composed of Si and O, and can be considered as SiO₂.

Fig. 6. Macroscopic view of the weft specimen with (a) 20 cycles, (b) 40 cycles, (c) 60 cycles.

Fig. 7. An enlarged image (a) and EDS result (b) of the oxide region of Fig. 6(b).

Fig. 8 shows the microscopic view of the fracture surfaces for the weft specimens with different thermal cycles. The pullout length of the weft fibers increases with increasing thermal cycles. Moreover, the bare warp fibers suggest that the tunneling matrix cracking is generated in the warp bundles, oriented 90° to the loading direction.

Fig. 8. SEM images of the fractured morphology of weft 2.5D-C/SiC specimen with various thermal cycles. (a) 20 cycles, (b) 40 cycles, (c) 60 cycles.

The fracture morphology of the warp composite mainly presents the failed warp bundles, and almost intact weft bundles, shown in Fig. 9(a). Similar to the weft specimens, more extensive pullout of warp fibers can be seen in the warp specimens with more thermal cycles, seen in Fig. 9(b). However, some warp fibers become thinner and are characteristic of cone-like ends in the composite with more than 40 thermal cycles.

Fig. 9. Fracture morphology of warp 2.5D-C/SiC specimen with 60 thermal cycles.

(a) Fractured warp fibers and extensive pullout of warp fiber, (b) A magnified image of (a).

4 Discussion

The above results show that the main damage mechanisms include matrix cracking, debonding of the fiber/matrix or coating/composite interface, as well as the oxidation of the carbon fibers after the 2.5D-C/SiC composite composite experienced different thermal cycles.

The more sensitivity of the modulus than that of the strength, displayed in Table 1 and Fig. 4, result from the different responses of both properties to the damage accumulation with different thermal cycles. It is known that the early damage mainly involves matrix microcracks at the composite surface [22]. The subtle development of these microcracks was undetectable by the conventional strength tests, such as flexural strength [23] and tensile strength in the present study, whereas, it could be sensitively detected by modulus [24, 25].

The surface of the 2.5D-C/SiC composite was cooled to 300 °C from 900 °C within ~15 s, whereas the interior still remained at higher temperature. The stress gradient, generated cyclically during the tests, can result in cracking and debonding of the coating, seen in Fig. 10 (a). Also, the mismatch of coefficient of thermal expansion (CTE) between the SiC matrix and carbon fibers is

responsible for such damage. SiC matrix has a greater CTE ($4.6 \times 10^{-6} / \text{K}$) than the longitudinal CTE of the carbon fiber ($0.5 \times 10^{-6} / \text{K}$). Tensile stress can be generated in SiC matrix by the rapid temperature change, and accumulated with increasing thermal cycles, and consequently create matrix cracks [23, 37, 38]. In addition, the radial CTE of the fiber ($7.0 \times 10^{-6} / \text{K}$) is greater than that of the matrix. Tensile stress was also produced on the fiber (bundles)/matrix interface during the thermal cycles. Consequently The repeated tensile stresses in interfaces lead to the delamination and debonding of fiber/matrix interface, seen in Fig. 10(b).

Fig.10. Debonding of the coating/composite interface (a) and fiber/matrix interface (b) caused by the thermal cycles.

The interfacial shear strength is inverse proportional to the pullout length of fibers[26]. Longer pullout length of fiber suggests weaker bonding between the fiber and matrix. Therefore, the weakened interface strength between the fiber and matrix could promote longer fiber pullout, shown in Fig. 8 and 9.

Moreover, the generated matrix cracks could further propagate with increasing thermal cycle [27]. The matrix cracking leads to the degradation of the tensile properties, shown in Fig. 4. In addition, the matrix cracks and debonding of the coating provide channels for oxygen in air to

diffuse into the composite. The temperature difference investigated in present works was 300~900 °C. SiC can be oxidized at about 600 °C by the possible reactions: $\text{SiC(s)} + 2\text{O}_2\text{(g)} = \text{SiO}_2\text{(s)} + \text{CO}_2\text{(g)}$. According to the thermochemical data [28], the Gibbs free energy for the reaction is -1028 kJ/mol at 900 °C, which indicates that above oxidation take place spontaneously when the specimens were heated to 900 °C during the thermal cycles in air. However, previous works indicate that the oxidation of SiC is too slow at the intermediate temperature [29, 30], that is, 700-900 °C, and silica can be generated rapidly above 900°C [31, 32]. Carbon are susceptible to the oxidation when the temperature was higher than 400°C [33]. Therefore, the interface and the carbon fibers of the SiC matrix composites can be quickly consumed through matrix cracking during the thermal cycles with the maximum temperature of 900°C. The burn-out of the carbon phases (the interface and the fibers) is faster than SiC oxidation, and result in weight loss, shown in Fig. 5.

If the coating is intact, only superficial oxidation takes place and the oxygen could not attack the carbon fiber or the interface. Therefore, the tensile strength can be retained. More thermal cycles can result in serious damage, including matrix cracking and debonding of fiber/matrix and coating/composite interfaces.

The heavily cracked coating and matrix no longer protect the carbon fibers and PyC interface from oxidization. The oxygen in the environment can diffuse into the composite through these channels and consume the carbon fibers and PyC interface, as can be evidenced by the cone-like fiber end in Fig. 9. The presence of the cone-like fiber end indicates that the carbon fibers are consumed by a manner of oxidation [34]. Oxidation occurs preferentially at weak bonding zones i.e. fiber-matrix interfacial zones. This is in line with mismatch of CTE of the fiber and matrix which resulted in interfacial debonding. At the beginning of oxidation, oxygen penetrates the weak

bonding zones, preferentially enlarges them by oxidising the fibre and the matrix. [35]. Moreover, the interior of fibers is more oxidation resistant than the edges because of a high preferred orientation of crystallites parallel to the fiber axes. As a result, the carbon fibers become thin and their ends become sharp with continued oxidation [36]. Therefore, the enhanced consumption of the warp carbon fibers means that more cracks and (or) larger crack might appear in the warp bundles when the composite was exposed to more thermal cycles. The oxidation of PyC interphase lead to the degradation in the interfacial shear strength and matrix cracking stress [8], and accordingly promote the matrix cracking at lower stress. Moreover, the oxidation degrades the strength of fibers and the interfacial shear strength according to Marshall's equation [26]. In addition, its effective loaded area reduces because of the consumption of the fibers. Therefore, the oxidization of the carbon fibers further degrades the strength of the composite greatly. As mentioned above, the fiber weaving might have apparent effect on damage initiation and delamination. More delamination sites and potential matrix cracking appear for warp bundles, if the composite is loaded in the warp direction. Therefore, the warp fibers should be consumed faster than weft fibers during the thermal cycles, as evidenced in the Fig. 8 and 9.

The product of the oxidation of carbon phases in the composite should be CO_2 or CO gases. These gases escape outwards and responsible for the mass loss for the composite with more than 20 thermal cycles, shown in Fig. 5. According to Fig. 6, the carbon fibers are almost intact within 20 cycles. Therefore, no significant mass loss in Fig. 5 can be found for the composite with 20 thermal cycles. The coating and matrix with accumulated cracks could not prevent the oxygen from penetrating into the composite with increasing thermal cycles. As a result, the carbon fibers were gradually oxidized. Accompanied by the escapement of the gases, broken bubbles or pores

were left on the surface, shown in Fig. 11. Similar morphology was also found on 2D-C/SiC [22] and SiC/SiC [39]. The oxidation of the PyC interface and/or carbon fibers directly lead to the degradation in the mechanical properties. Therefore, an appropriate means of protecting the carbon fibers from oxidation are of critical importance to improve the resistance to thermal cycles. By comparing the tensile properties (Table 1) of the specimens with different thermal cycles and those of the annealed specimens, it can be found that the retained tensile properties of the annealed specimen is larger than those of the specimen with 60 thermal cycles, although the annealing period is equal to the accumulated holding time at 900 °C for 60 thermal cycles. Therefore, the thermal cycles introduce progressively more damage in the 2.5D-C/SiC specimens than the annealing, and in this way degrade their tensile properties. Similar result can also be found for 2D-C/SiC [40].

Fig. 11. Broken bubbles caused by the oxidation of the carbon fibers in the warp specimen with 60 thermal cycles.

5. Conclusions

The resistance to the thermal cycles was investigated for a 2.5D-C/SiC composite in warp and weft direction. A series of thermal cycle experiments were performed between 900 °C and 300 °C in air. The retained tensile properties were employed to characterize the resistance to the

thermal cycles. The damage mechanisms were investigated by observing the fractured morphology. Several main conclusions were made as following.

- 1) The 2.5D-C/SiC composite retains its tensile strength within 40 thermal cycles. The residual modulus in both directions decrease with increasing thermal cycles. The different responses for both directions to the thermal cycles result from the damage mechanisms, related to the woven fibers in both directions.
- 2) The degradation in the mechanical properties is directly caused by the damage accumulation during the thermal cycles. The damage mechanisms include matrix cracking, debonding of the fiber/matrix or coating/composite interface, as well as the oxidation of the carbon fibers.
- 3) More delamination and potential matrix cracking contribute to the earlier degradation in the warp direction than that in the weft direction that when the composite is loaded in the warp direction. This allows oxidation of the reinforcements to occur more readily.

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