

Effect of Heat Treatment on the Thermal Expansion of 1D C/SiC Composites from Room Temperature to 1400°C

Laifei Cheng*, Yongdong Xu, Litong Zhang and Dong Wang

*State Key Laboratory of Solidification Processing
Northwestern Polytechnical University, Xi'an Shaanxi 710072, P.R. China*

ABSTRACT

1D C/PyC and 1D SiC/C/SiC composites as well as a bulk CVD SiC material were prepared. The effects of heat treatment on the thermal expansion behavior of the composites from room temperature to 1400°C were investigated. The thermal expansion behavior of 1D C/PyC and 1D SiC/C/SiC composites depended on the thermal stress and the transition temperature of thermal stress from compressive to tensile in both the fibers and the matrix. The heat treatment affected the expansion behavior by changing the thermal stress and the transition temperature. The treatment increased the transition temperature of axial stress in 1D C/PyC from its deposition temperature to 1300°C and then increased its longitudinal CTE above this temperature, but it had no obvious effect below this temperature. The treatment did not increase the transition temperature of axial stress in 1D SiC/C/SiC, but led to fracture of the fibers from the deposition temperature to 1300°C. As a result, the treatment increased its longitudinal CTE below 1300°C, but had nearly no effect on it above 1300°C. The treatment increased the transition temperature of radial stress in 1D C/PyC from its deposition temperature up to 1300°C, and then the transverse CTE of the composite below 1300°C depended on that of the SiC matrix.

Keywords: Carbon fiber composites, heat treatment, thermal expansion

1. INTRODUCTION

Carbon fiber reinforced silicon carbide composites (C/SiC) have been developed and tested for structural applications, such as components of turbine engines, the reentry thermal protection system of spacecraft, ultralightweight mirrors and so on [1,2]. The coefficient of thermal expansion (CTE) is one of the most relevant properties for all these applications. Thermal expansion of C/SiC composites has been reported and some data can be obtained [3], but it has not been systematically investigated.

The fibers, matrix and interlayer in C/SiC composites have different CTEs and, consequently, thermal stress will be produced in these three microstructure constituents. Clearly, the expansion behavior of the C/SiC composites depends upon the thermal stress [4]. It is necessary to relate the expansion behavior to the thermal stress in order to reveal expansion mechanisms of the composites. When used at high temperatures, the thermal stress conditions will change and then the composites will exhibit a different expansion behavior. Therefore, it is very important to clarify the effect of heat treatment on the expansion behavior for applications of C/SiC composites. It is more convenient to relate the expansion behavior to the thermal stress by using 1D C/PyC because the thermal stress is here relatively simple.

In this article, 1D C/PyC and 1D SiC/C/SiC composites as well as a bulk CVD SiC material were

* Corresponding author. Tel.: +86-29-849-4616; Fax: +86-29-4620.
E-mail address: chenglf@nwpu.edu.cn (L.F. Cheng)

prepared and the effects of heat treatment on the thermal expansion behavior of the composites from room temperature to 1400°C were investigated.

2. EXPERIMENTAL PROCEDURE

One-dimensional preforms (1D) were prepared by putting 3K T-300 carbon fibers in order. The preforms were deposited with pyrolysis carbon (PyC) to fabricate a 1D C/PyC composite and with both PyC and SiC to fabricate a 1D C/SiC composite using butane and methyltrichlorosilane (MTS). The deposition conditions of the PyC interlayer were as follows: temperature 960°C, pressure 5 KPa, time 20 hours, Ar flow 200 ml·min⁻¹, butane flow 15 ml·min⁻¹. The deposition conditions of the SiC matrix were as follows: temperature 1000°C, pressure 5KPa, time 120 hours, H₂ flow 350 ml·min⁻¹, Ar flow 350 ml·min⁻¹, and the molar ratio of H₂ to MTS 10. Specimens were machined from the fabricated materials and the 1D C/SiC specimens were further deposited under the same conditions to prepare a SiC coating and to densify the composite. The PyC matrix in C/PyC was deposited under the same conditions as the PyC interlayer for 300 hours.

The deposition conditions of the bulk SiC material were as follows: temperature 1100°C, time 200 hours, H₂ flow 400 ml·min⁻¹, Ar flow 400 ml·min⁻¹, and the molar ratio of H₂ to MTS 3-5. Although the deposition conditions of the bulk SiC material were different from those of the SiC matrix and the SiC coating, measuring its CTE is necessary for investigation of the expansion behavior of C/SiC composites.

2.2. Measurements of CTE

A DIL 402C dilatometer made by Netzsch Company was employed for measurements of CTE. The required specimen size was 3.5 × 3.5 × 20 mm³. All measurements were conducted in a nitrogen atmosphere from room temperature to 1400°C. The SiC/C/SiC specimens were measured twice and the C/PyC specimens three times. The first measurement acted on the second one as a heat treatment, so did the second on the third.

3. RESULTS AND DISCUSSION

3.1. Effect of heat treatment on the CTE of 1D C/PyC

Fig. 1 shows the relation of CTE to the temperature of the C/PyC specimen obtained in the first, second and third measurements. Below the deposition temperature of PyC, which was 960°C, the curves of CTE vs. temperature from the three measurements coincided completely with each other. This indicates that heat treatment has no effect on the CTE of 1D C/PyC. Above the deposition temperature, the CTE of 1D C/PyC from the second measurement was much higher than that from the first measurement. The CTE of 1D C/PyC from the third measurement was nearly coincident with that from the second measurement below 1300°C. The former was a little higher than the latter above 1300°C. It can be seen that the heat treatment has a strong effect on the CTE of 1D C/PyC, but further treatment has no obvious effect. Below 900°C, the CTE of 1D C/PyC increased linearly with increasing temperature. Above 900°C, the CTE of the prepared 1D C/PyC decreased rapidly and increased again after reaching its minimum value at about 1200°C. However, the CTE of the treated 1D C/PyC always decreased above 900°C.

The thermal expansion behavior of C/PyC can be related to thermal stress change with increasing temperature in the fibers and the PyC matrix (Fig. 2). Since the CTE of the fibers was higher than that of the

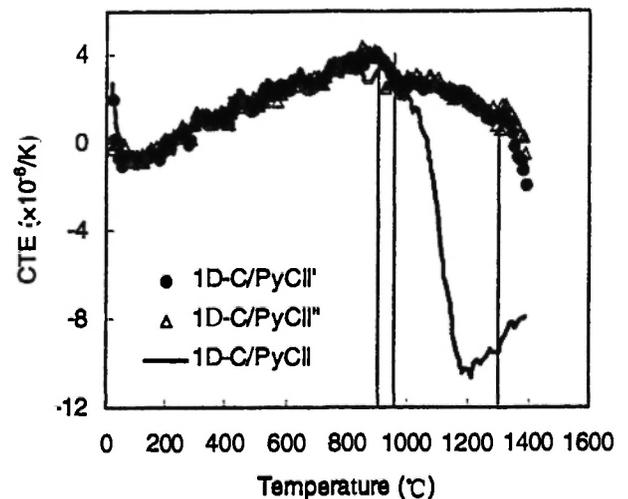


Fig. 1: Effect of heat treatment on the relation of longitudinal CTE to temperature in 1D C/PyC.

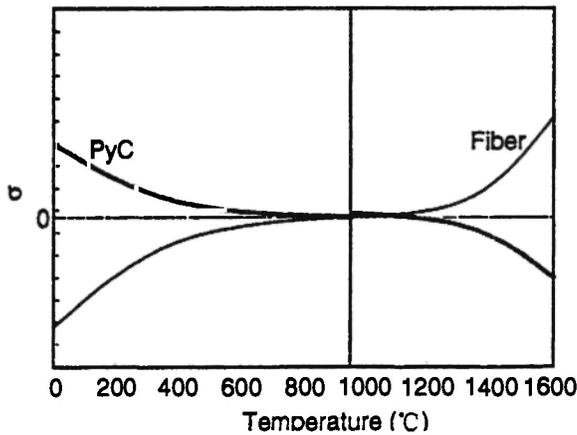


Fig. 2: Schematic stress change with temperature in fibers and PyC of C/PyC.

PyC matrix, a tensile stress was produced above the deposition temperature and a compressive stress below this temperature in the fibers. Correspondingly, a compressive stress was produced above the deposition temperature and a tensile stress below this temperature in the PyC matrix. In the first measurement, the CTE of 1D C/PyC above the deposition temperature was mainly controlled by contraction of the fibers and then decreased rapidly with increasing temperature. Below the deposition temperature, it was mainly controlled by expansion of the PyC matrix and then increased linearly with increasing temperature. Above 1200°C, the interfacial shear stress was so high that yielding took place in the PyC matrix with a low modulus, and then the CTE of 1D C/PyC increased with increasing temperature due to expansion of the PyC matrix. The transition temperature of thermal stress from tensile to compressive in the fibers was increased from 960°C to 1300°C by the yielding. The higher transition temperature led to a higher compressive stress in the fiber at room temperature. This can be confirmed by shortening of the specimen. After the first measurement, the length change was -0.26%.

In the second measurement, the CTE of 1D C/PyC was mainly controlled by contraction of the fibers above the transition temperature and by expansion of the PyC matrix below the transition temperature. As a result, the CTE of 1D C/PyC from the second measurement coincided completely with that from the first measurement and increased greatly above the deposition temperature.

After the second measurement, the transition temperature was not significantly increased. Therefore, the relation of CTE to temperature from the third measurement was the same as that from the second measurement.

3.2. Effect of heat treatment on the CTE of 1D SiC/C/SiC

Fig. 3 shows the effect of heat treatment on the relation of longitudinal CTE to temperature in 1D SiC/C/SiC. The longitudinal CTE of 1D SiC/C/SiC was obviously decreased by treatment in the temperature range from 100°C to 1300°C. It reached its maximum value at about 900°C and changed linearly from 100°C to 900°C both before and after the treatment. The relations of the transverse CTE of 1D SiC/C/SiC before and after the treatment are shown and compared with that of the bulk CVD SiC in Fig. 4. The transverse CTE increased irregularly in the full temperature range before the treatment and decreased with increasing temperature above 800°C after the treatment. The treatment had no obvious effect on the transverse CTE of 1D SiC/C/SiC below 800°C. It should be noted that there were three deep valleys on the CTE vs. temperature curve after the treatment.

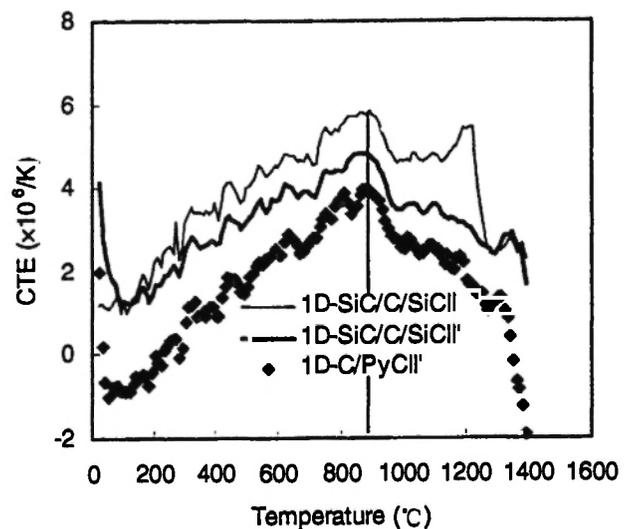


Fig. 3: Effect of heat treatment on the relation of longitudinal CTE to temperature in 3D SiC/C/SiC.

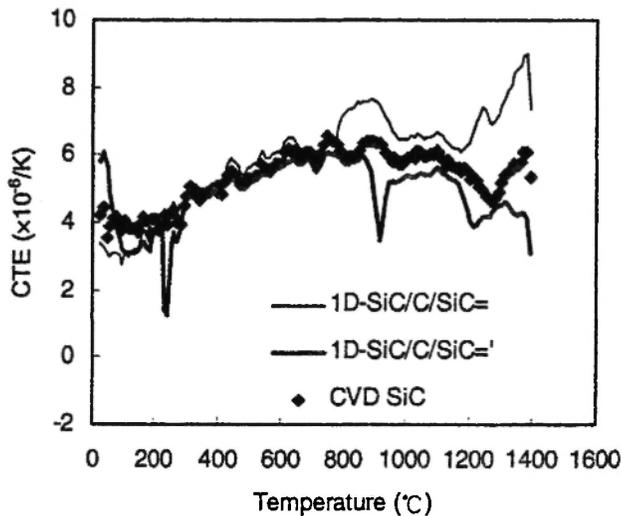


Fig. 4: Effect of heat treatment on the relation of transverse CTE to temperature in 1D SiC/C/SiC.

The effect of heat treatment on the longitudinal CTE of 1D SiC/C/SiC composite can be explained by the thermal expansion behavior of 1D C/PyC. Although the CTE of 1D SiC/C/SiC before the treatment was higher than that of 1D C/PyC in the full temperature range, they increased linearly with a similar slope below 900°C and reached their maximum values at the same temperature. Because the SiC matrix had a larger expansion mismatch with the fibers than the PyC matrix, it was cracked by the longitudinal tensile stress. As a result, the longitudinal CTE increase with temperature of 1D SiC/C/SiC was controlled by that of the fibers, although the CTE of 1D SiC/C/SiC was increased by the SiC matrix due to its higher expansion. Above 900°C, the tensile stress in the fibers of 1D SiC/C/SiC was much higher than that of 1D C/PyC owing to the high modulus of the SiC matrix. After the treatment, fibers were fractured by the tensile stress. For this reason, the matrix cracks played a more important role in the relation of longitudinal CTE to temperature in 1D SiC/C/SiC and the transition temperature from compressive stress to tensile stress did not change. Therefore, the longitudinal CTE of 1D SiC/C/SiC increased linearly with a lower slope below 900°C after the treatment and reached its maximum value at the same temperature. Because the length of the fibers was still greater than the critical value after fracture, no

obvious strength loss of 1D SiC/C/SiC corresponded to the CTE decrease after the treatment.

The radical CTE of the fibers was much lower than that of the SiC matrix, and then no stress was produced in the fibers or the matrix below 900°C. This indicates that the relation of transverse CTE to temperature in 1D SiC/C/SiC completely depended on and nearly coincided with that of the SiC matrix. The radical stress was produced in both the fibers and the matrix above 900°C. Consequently, the transverse CTE was controlled by the radical CTE of the fibers, increasing then with increasing temperature. After the treatment, the radical compressive stress led to yielding of the SiC matrix with a high porosity and then the transition temperature of radical stress was increased from 900°C to 1300°C. The transverse CTE after treatment was mainly controlled by that of the SiC matrix below 1300°C and by the radical CTE of the fibers above 1300°C. Since yielding of the matrix further lowered the transverse modulus of 1D SiC/C/SiC, deformation of the specimen took place in the second measurement at different temperatures. As a result, three deep valleys appeared on the transverse CTE vs. temperature curve after the treatment.

4. CONCLUSIONS

1. The thermal expansion behavior of 1D C/PyC and 1D SiC/C/SiC composites depended on the thermal stress and the transition temperature of thermal stress from compressive to tensile in both the fibers and the matrix. The heat treatment affected the thermal expansion behavior by changing the thermal stress and the transition temperature.
2. The treatment increased the transition temperature of axial stress in 1D C/PyC from its deposition temperature to 1300°C and then increased its longitudinal CTE in the full temperature range, but it had no obvious effect below the deposition temperature due to yielding and the low modulus of the PyC matrix.
3. The treatment did not increase the transition temperature of axial stress in 1D SiC/C/SiC, but led to fracture of the fibers from the deposition temperature to 1300°C owing to the high modulus of

the SiC matrix. As a result, the treatment increased the longitudinal CTE below 1300°C, but had nearly no effect above 1300°C.

4. The treatment increased the transition temperature of residual stress in ID C/PyC from its deposition temperature to 1300°C because of residual yielding of the porous SiC matrix, and then the transverse CTE of the composite below 1300°C depended on that of the SiC matrix.

ACKNOWLEDGEMENTS

The authors acknowledge the support of the Chinese National Foundation for Natural Sciences under Contract No. 59772023 and the Chinese Defense Foundation for Sciences under Contract No. 99J12.5.2.

REFERENCES

1. J.M. Jamet, P.J. Lemicq and G.H. Schiroky. Composite thermo-structures: an overview of the French experience, in: R. Naslain (Ed.), *High Temperature Ceramic Matrix Composites*, Bordeaux: Woodhead Publications, 1993; pp. 215-229.
2. T. Laux, T. Ullmann, M. Auweter-Kurtz, H. Hald and A. Kurz. Investigation of thermal protection materials along an X-38 re-entry trajectory by plasma wind tunnel simulations, in: *2nd International Symposium on Atmospheric Reentry Vehicles and Systems*, Arcahon, France, 2001; pp. 1-9.
3. P. Schanz and W. Krenkel. Description of the mechanical and thermal behavior of liquid siliconized C/C, in: R. Naslain (Ed.), *High Temperature Ceramic Matrix Composites*, Bordeaux: Woodhead Publications, 1993; pp. 715-724.
4. W. Cheng, S. Zhao, Z. Liu, Z. Feng, C. Yao and Y. Yu. Thermal expansion in 3-D braided fiber composites. Experimental and numerical results, in: Y. Zhang (Ed.), *13th International Conference on Composite Materials*, Beijing: Science and Technical Documents Publishing House, 2001; p. 440.

