ELSEVIER

Ceramics International 33 (2007) 141–146

CERAMICS
INTERNATIONAL

www.elsevier.com/locate/ceramint

Effects of heat treatment on the microstructure and flexural properties of CVI–Tyranno-SA/SiC composite

Wen Yang [a,b,*], Hiroshi Araki [b], Akira Kohyama [c], Qifa Yang [a], Tetsuji Noda [b]

[a] *China Institute of Atomic Energy, P.O. Box 275-51, Beijing 102413, China*
[b] *National Institute for Materials Science, Tsukuba 305-0047, Japan*
[c] *Institute of Advanced Energy, Kyoto University, Uji 611-0011, Japan*

Received 12 July 2005; received in revised form 19 July 2005; accepted 16 August 2005

Abstract

Effects of heat treatment in vacuum on the microstructure and strength of a chemical vapor infiltration (CVI)–Tyranno-SA/SiC composite were investigated. Very thin (20 nm) carbon layer was deposited on the fibers as the fiber/matrix interlayer to modify the interfacial bonding strength. The room temperature ultimate flexural strength showed no degradation at heat treatment temperature up to near 2073 K, beyond witch the strength decreased with increasing the heat treatment temperature. About 58% of as-fabricated strength maintained even after heat treatment at 2273 K. Damages to the specimen surfaces and the inner walls of relatively large open pores in the specimens that might be caused by the sublimation of the CVI–SiC matrix during the heat treatment, are believed to be the main reason causing the decrease of the strength of the composite. The present CVI–Tyranno-SA/SiC composite showed much improved RT strength and structure thermal stability than the CVI–SiC/SiC composites reinforced with old-generation Nicalon^{TM} or Hi-Nicalon^{TM} fibers, and the Tyranno-SA/SiC composites fabricated by PIP process.

Keywords: D. SiC; Chemical vapor infiltration; Heat treatment; SiC/SiC composite; Tyranno-SA fiber; Flexural strength

1. Introduction

Ceramic-matrix composites with continuous fiber reinforcements (CFCC), such as SiC fibers reinforced SiC matrix composites (SiC/SiC), generally exhibit attractive properties such as low densities, high elastic moduli, markedly improved fracture toughness over the bulk ceramics, and high strength at elevated temperature [1–5]. Owing to these advantages, ceramic composites are considered as materials with a high potential in various fields of applications including hot engines, advanced energy conversion systems, and spacecraft [2–8].

Since they are supposed to be in use in various environments under elevated temperature conditions, the high temperature thermal stability of the microstructure and mechanical performances of CFCC is a very important issue, and quite an amount of studies has addressed this issue [9–16]. Guo and Kagawa [13] studied the tensile fracture behaviors of plain-woven SiC fabric (carbon-coated Nicalon^{TM} and BN-coated Hi-Nicalon^{TM})-reinforced SiC/SiC composites at elevated temperature in air. It was found that the composites reinforced with carbon-coated Nicalon^{TM} fiber showed significant decrease of strength when the test temperature was above 800 K, from 140 MPa at 800 K to 41 MPa at 1200 K. The BN-coated Hi-Nicalon^{TM}/SiC composites showed better performance. The strength started to decrease at 1200 K. Similar phenomena were also observed with single tow Nicalon^{TM} and/or Hi-Nicalon^{TM} reinforced SiC/SiC minicomposites [14,15]. The strengths of Hi-Nicalon^{TM}/C/SiC minicomposites at 973 and 1223 K were about 63 and 36% of as-produced strength, respectively. Oxidation-induced degradation of the interphase (carbon or BN), and hence, the mechanical properties of interfaces, was one of the main reasons causing the degradations of the mechanical properties of above-mentioned composites. The effects of heat treatment at temperature from 1273 to 2273 K in vacuum on the room temperature (RT) strength of Nicalon^{TM}/SiC and Hi-Nicalon^{TM}/SiC composites were investigated by Araki et al. [16] Weight loss and deoxidation, to different extent, occurred in the two kinds of composites when heat treatment temperature was over 1673 K, resulting in accordingly the decrease of the

---

* Corresponding author. Fax: +81 298 59 2701.
E-mail address: YANG.Wen@nims.go.jp (W. Yang).

0272-8842/$32.00 © 2005 Published by Elsevier Ltd and Techna Group S.r.l.
doi:10.1016/j.ceramint.2005.08.011

RT strengths of both composites with increasing the heat treatment temperature. The weight loss and deoxidation of the composites were attributed to the sublimation of SiO resulting from the thermal decompositions of the Nicalon^{TM} and Hi-Nicalon^{TM} fibers. Hi-Nicalon^{TM} fiber contains much less oxygen and shows better thermal stability than the Nicalon^{TM} fibers [17]. Therefore, in above-mentioned studies, Hi-Nicalon^{TM} fibers reinforced composites showed slower degradation than the Nicalon^{TM} reinforced ones against temperature both in air and in vacuum.

Recently, a highly crystalline near stoicheometric SiC fiber, Tyranno-SA, has been developed [18]. The fiber possesses excellent mechanical properties and much improved thermal stability over the Nicalon^{TM} and Hi-Nicalon^{TM} fibers. The RT strength of the Tyranno-SA fibers showed no degradation when annealed at temperature up to 2173 K in argon atmosphere [18]. Very recently, Nozawa et al. [19] reported the effects of heat treatment on the flexural strength of polymer impregnation and pyrolysis (PIP)–Tyranno-SA/SiC composites. The composites showed stable RT strength against heat treatment temperature (in flowing commercial argon atmosphere) up to 1773 K. However, after heat treatment at 1973 K for 1 h, the composites showed a RT strength loss of about 30%. Rather than the advanced Tyranno-SA fibers and the interphase, the degradation of the composites was attributed to the extensive crystallization of the partially amorphous PIP-matrix during the heat treatment at 1973 K [19]. Chemical vapor infiltration (CVI) is a widely used process to produce high purity and highly crystalline β-phase SiC matrix in SiC/SiC composites [4,20]. The thermal resistance of CVI–SiC matrix should be much better than that from PIP because of its pure and high crystallinity. However, up to now, it remains unclear the thermal stability of SiC/SiC composites reinforced with Tyranno-SA fibers and densified with CVI–SiC matrix. This study investigates the RT flexural properties and fracture behaviors of a CVI–Tyranno-SA/SiC composite after high temperature heat treatment in vacuum. The main interests are the effects of the heat treatment on the microstructure and RT strength of the composite. Twenty nanometers CVI–carbon was deposited on the fibers in the composite as the interfacial layer.

2. Experimental

2.1. Composite fabrication

The composite preform was prepared by stacking 18 sheets of plain-woven Tyranno-SA fiber cloths in 0/90°. The fibers were provided by Ube Industry Ltd., Japan. The chemical composition, thermal-mechanical properties of the fibers can be found elsewhere [17]. The preform was compressed with a set of graphite fixtures to keep a fiber volume fraction of ~30%, resulting in a dimension of the preform 120 mm in diameter and ~3 mm in thickness. The preform was densified with SiC-matrix using the thermal decompositions of methyltrichlorosilane (MTS). MTS was carried by hydrogen with volume ratio about 0.5 and a total hydrogen flow rate 2.0 l/min. The CVI densification process was carried out at 1273 K for about 80 h at reduced pressure of 14.4 kPa. Prior to the CVI matrix densification, the preform was pre-coated with 20 nm pyrolytic carbon as fiber–matrix interlayer from methane (CH_4) using the CVI process, to modify the interfacial bonding strength.

2.2. Heat treatment and mechanical tests

Upon the completion of the fabrication, 35 specimens with a surface area of 25 mm × 4.0 mm were cut parallel to one of the fiber bundle directions of the fabric cloths. Both surfaces of the specimens were carefully ground to remove the surface chemical vapour deposition (CVD)–SiC layer that was formed at the end of the CVI, and to guarantee a same surface condition of the specimens for the following heat treatment and bending tests. The resulting specimen size was 25 mm in length, 4.0 mm in width and 2.0 mm in thickness. The average density of the composite was derived from the mass and volume of each specimen. Heat treatments in vacuum of $\sim 3 \times 10^{-4}$ Pa for one hour at temperature ranging from 1273 to 2273 K were carried out with five specimens for each temperature. After the heat treatment, the flexural strength and fracture behaviors were investigated at RT by three-point bending tests with support span 16 mm and crosshead speed 0.0083 mm/s.

2.3. Microstructure characterization

The crystallinity of the specimens before and after heat treatment was examined with X-ray diffraction (XRD). The microstructures, carbon interlayers, and the fracture surfaces were examined by means of field emission scanning electron microscopy (SEM, JEOL JSM-6700F).

3. Results and discussion

3.1. Fracture behaviors and flexural strength

Fig. 1 shows representative RT load–displacement curves of the specimens. The average density of the composite is 2.70 ± 0.14 Mg/m^3, corresponding to an overall porosity of

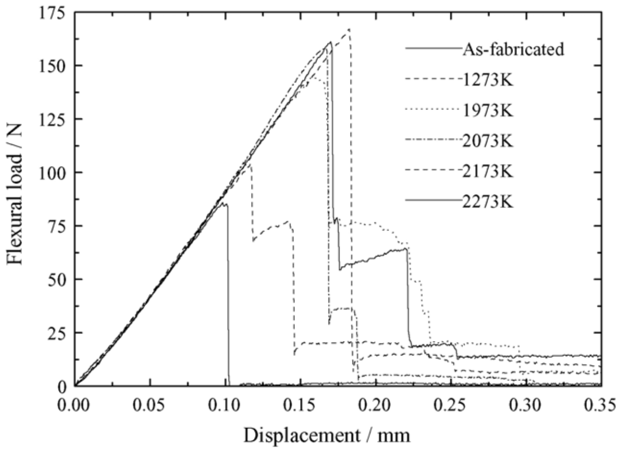

Fig. 1. Representative RT load–displacement curves before and after heat treatment.

~12%. The load–displacement curve of as-fabricated specimen displayed typical fracture behaviors for Tyranno-SA/SiC composite [21]: An initial linear increasing of the load up to near to the load maximum, reflecting the elastic response of the composites. Deviation of the curves from the linearity occurred at certain loads, followed by a nonlinear domain of deformation till the load maximum, due mainly to the matrix cracking, interfacial debonding and fiber sliding, and individual fibers failures. A quick drop of the load beyond the load maximum indicates the failure of a significant fraction of the fibers. The level of the remained load after the first quick drop of the load depends on the amount of the intact fibers in the specimen. No significant difference of the fracture behaviors of the specimens after heat treatment up to 2073 K was noticed. The specimens after heat treatment at 2173 and 2273 K showed much lower load maximums, respectively about 65 and 58% of that of the as-fabricated one.

The ultimate flexural strengths (UFS) were derived from the RT load–displacement curves according to ASTM C 1341-97 [22]. The average UFS of the as-fabricated composite is 380 ± 110 MPa, which is lower that those previously reported 2D CVI–Tyranno-SA/SiC composites with carbon interlayers [21]. The strengths of the Tyranno-SA/SiC composites showed high sensitivity to the thickness of carbon interlayers, especially when the carbon layer is thin (less than 100 nm). The UFS of the composites increased from 410 to 606 MPa when the carbon interlayer increased from 50 to 100 nm. The present composite possesses thinner carbon interlayer (20 nm) and resulting in lower strength. The lower RT UFS of present composite is in accordance with the short fiber pullouts fracture behaviour as will be discussed later.

Fig. 2 shows the normalized RT UFS against the heat treatment temperature. Although relatively large error bars appeared indicating the quite scattering of the strengths of the five specimens for each temperature, Fig. 2 shows a clear trend of the RT strength via heat treatment temperature. No obvious decrease of the RT UFS was noticed up to as high as 2073 K, beyond which the RT strength decreased quickly from 380 ± 110 MPa (as-fabricated) to 250 ± 80 MPa (2173 K) and 220 ± 90 MPa (2273 K), respectively. The composite exhibited an excellent strength thermal stability compared with the PIP–Tyranno-SA/SiC [19] and the Hi-Nicalon^{TM}/SiC composites (the data for the Hi-Nicalon^{TM} composites are also shown in the figure) [16], of which the ambient temperature strength start to decrease after heat treatment at 1773 and 1673 K, respectively.

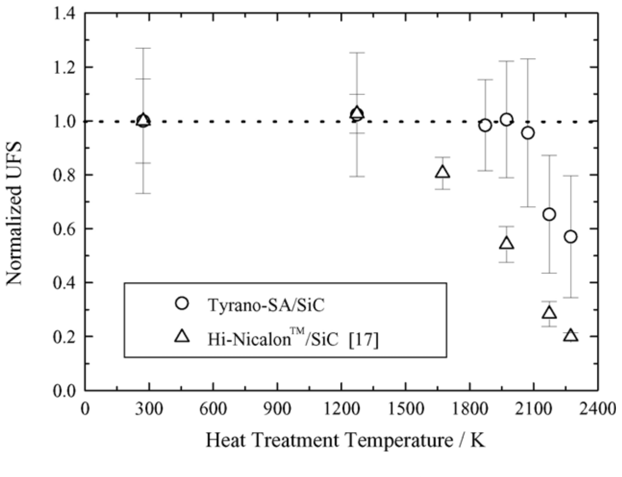

Fig. 2. Heat treatment temperature dependence of normalized RT UFSs of CVI–Tyranno-SA/SiC and Hi-Nicalon^{TM}/SiC composites.

3.2. Microstructure examination and discussion

There are several possible reasons causing the RT strength decrease of SiC/SiC composites after heat treatment at elevated temperature. Oxidation induced degradation of the carbon interlayer is the most common case [10,11,14,15,23], in which the carbon layer would be burnt away and a gap between the fiber and the matrix left. This phenomenon is usually observed at medium temperature, say 773–973 K, when the carbon layer is thick enough (over 100 nm) [6,11], while the present composite showed very stable RT strength after heat treatment in this temperature range. SEM microstructure and fracture surface examinations showed well-bonded interface and no obvious damage/degradation of the carbon layer was observed in all the specimens even after heat treatment at 2273 K. Therefore, oxidation-induced degradation of the carbon interlayer is not likely the situation for the present composite. Fig. 3 shows a typical fracture surface image taken at the tension region (near to the tension side upon bending load) of one of the specimens after heat treatment at 2273 K. The image revealed a matrix cracking, interfacial debonding and fiber pullouts fracture behavior of the specimen. The length of the pullout fibers is relatively short (the mean pullout length is ~7 μm). This is not because of the degradation of the interlayer during the heat treatment because similar short fiber pullouts fracture occurred for all the specimens including the as-fabricated ones. The short fiber pullouts resulted from a very strong bonding strength between the fiber and matrix due to the very thin carbon interlayer (20 nm) and quite rough surface of the Tyranno-SA fiber. According to previous empirical study [24] on the carbon interlayer thickness dependence of the interfacial bonding strength in CVI–Tyranno-SA/SiC composites, 20 nm carbon interlayer would produce a interfacial bonding strength as high as up to 500–600 MPa. It is difficult

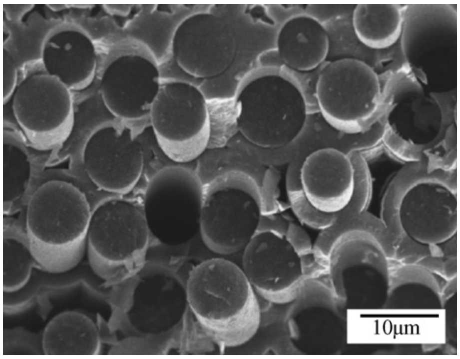

Fig. 3. Fracture surface of specimen after heat treatment at 2273 K, showing matrix cracking, interfacial debonding and short fiber pullouts fracture behaviors.

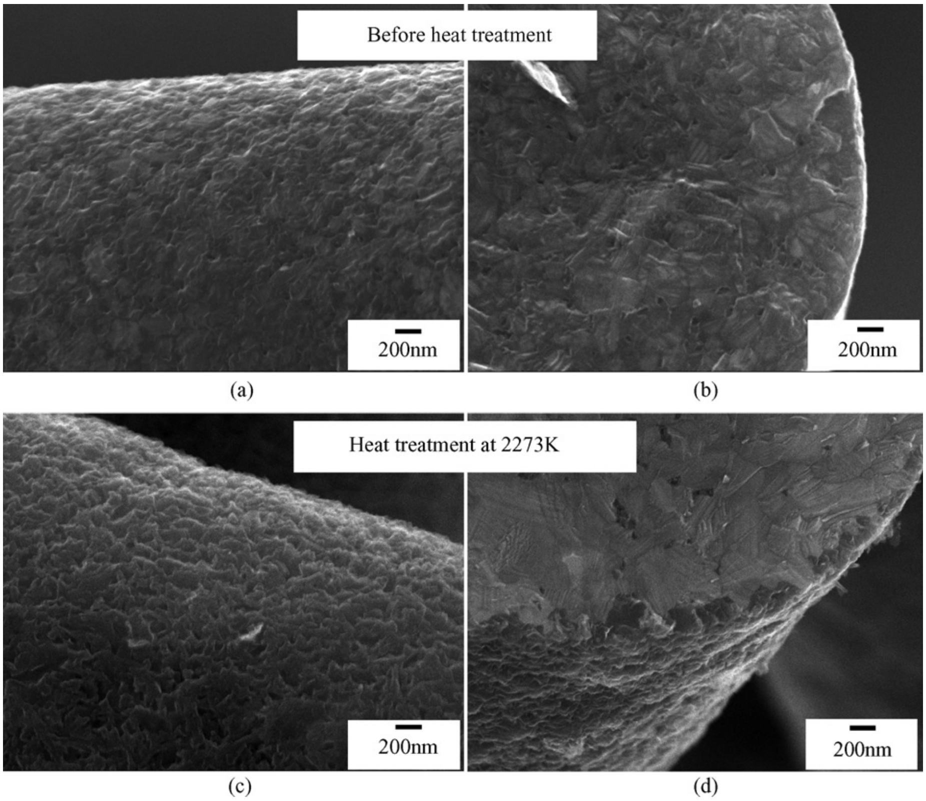

Fig. 4. Surfaces and cross sections of Tyranno-SA fibers before and after heat treatment at 2273 K, showing damages occurred at the surface of the fibers at 2273 K.

for the composite with such high interfacial bonding strength to fail with more elegant fiber pullout fracture. The strength is therefore lower than previous reported ones in which either thicker or multiple SiC–carbon interlayers were applied [21].

The degradation of the reinforcement fibers, as frequently exhibited by the old-generation Nicalon™ and Hi-Nicalon™ reinforced SiC/SiC composites [13–16], is believed not to be the main reason either to cause the significant decrease of the RT strength of the composite upon heat treatment at temperature higher than 2073 K because the Tyranno-SA fiber is a near stoicheometric highly crystalline SiC fiber. The RT strength of the Tyranno-SA fibers showed no degradation at annealing temperature up to 2173 K in argon atmosphere [18]. During the heat treatment, several bundles of free-standing Tyranno-SA fibers were also put in the furnace together with the specimens at each temperature for a comparative study. Fig. 4 are SEM images showing the surfaces and cross sections of Tyranno-SA fibers before and after heat treatment at 2273 K. Slight changes/damages at the surfaces of the fibers after heat treatment at 2273 K are evident. The thickness of this damaged layer is 200–300 nm, likely due to the sublimation of the SiC as will be discussed later. However, such damages to the free-standing fibers were not observed at the surfaces of the fibers (at the fracture surfaces) that were buried in CVI–SiC matrix.

Further fracture surfaces examinations found that significant damages occurred at the regions near to the specimens surfaces for the specimens after heat treatment at 2273 K, as shown in Fig. 5. This damage to the specimens is attributed to the heat treatment because no such phenomenon was observed at the fracture surfaces of as-fabricated specimens. The depth of this damaged layer is 3–5 μm. After the fracture surface examinations, the whole surfaces of the specimens were also examined. Fig. 6 is a typical image of the specimen surface after heat treatment at 2273 K. As mentioned before, the specimen surface had been carefully polished with diamond slurry (grain size of 9 μm). After the heat treatment, the whole surface of the specimen became very rough, similar as the situation of the Tyranno-SA fiber (Fig. 4c) but more serious. XRD analysis (Fig. 7) of this surface showed pure β-phase SiC crystal structure, as that of the as-fabricated one. Therefore, no phase transformation from β to α took place during the heat treatment. Oxidation of SiC usually starts to happen at

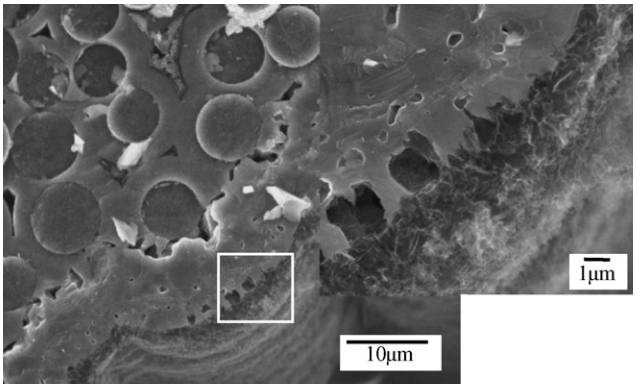

Fig. 5. Fracture surface at the region near to the surface of specimen after heat treatment at 2273 K, showing damage occurred at the specimen surface.

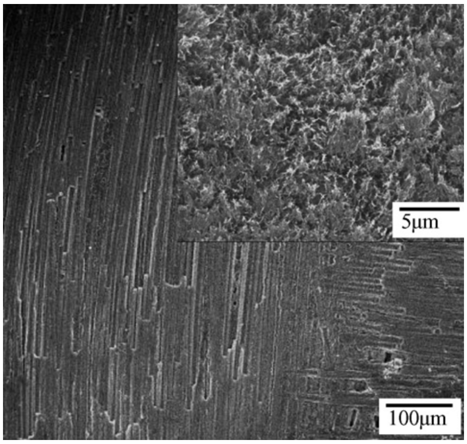

Fig. 6. Surface of specimen after heat treatment at 2273 K, showing significant damages occurred at the specimen surface.

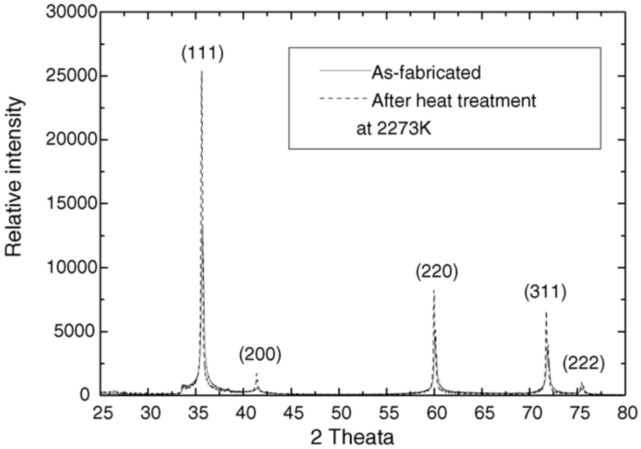

Fig. 7. XRD patterns of as-fabricated specimen and that after heat treatment at 2273 K.

temperature lower than 1373 K to form silicon oxide layer while neither silicon oxide nor oxygen was detected in the heat-treated specimens by the XRD and FE-SEM EDS analysis. Therefore, this damage to the specimen surface was unlikely to be caused by a passive oxidation of SiC either. In addition, although similar damage was found at the surfaces of the specimens after heat treatment at 2173 K but not found at 2073 K. The depth of the damaged layer at 2173 K is less than 1 μm.

It is considered that the damages to the specimens after heat treatment at 2173 and 2273 K might be due to the sublimation of the CVI–SiC matrix during the heat treatment [16]:

$$\mathrm{SiC} \rightarrow \mathrm{SiC}\,(\text{sublimation})$$

At ambient pressure, sublimation of stoicheometric β-SiC occurs at temperature higher than 2273 K. While in vacuum chamber, this might occur at lower temperature with the CVI–SiC matrix that might be slightly deviated from stoicheometric SiC depending on the CVI process conditions when MTS + H_2 were used as the source gases [25]. However, under present heat treatment conditions, there might be other possibilities: (1) a surface decomposition through:

$$\mathrm{SiC_{(s)}} \rightarrow \mathrm{Si_{(g)}} + \mathrm{C_{(s)}}$$

Or (2) an active oxidation of SiC due to possibly trace oxygen in the high temperature heat treatment furnace:

$$\mathrm{SiC_{(s)}} + \mathrm{O_{2(g)}} \rightarrow \mathrm{SiO_{(g)}} + \mathrm{CO_{(g)}}$$

Further experiment observations are necessary to make clear this issue. After bending tests, some of the specimens were cut and polished to inspect the microstructure in the specimens. It was found that significant changes had occurred at the inner walls of relatively large open pores in the specimens after heat treatment at 2273 K. Fig. 8 is a typical image of such changes. Instead of dense and smooth surface as in typical CVI–SiC/SiC composites, a layer of particles with size of several to ten micrometers was formed at the surfaces of inner wall of a large pore. This is different to that observed at the surface of the specimen. These particles are still β-phase SiC with Si–C chemistry. Although it remains unclear the formation mechanism of this particle layer, it is believed that such damages to the specimen surfaces and inner walls of relatively large open pores in the specimens are the main reasons causing the decrease of composite RT strength. The strength of the layer of those particles is essentially zero because they are basically isolated. The much rough surface decreased the critical stress to form main matrix crack in the specimen upon bending load, and resulted in decreased RT ultimate flexural strength. The damages to the specimens after heat treatment at 2173 K are less than those at 2273 K, and almost no damage at 2073 K,

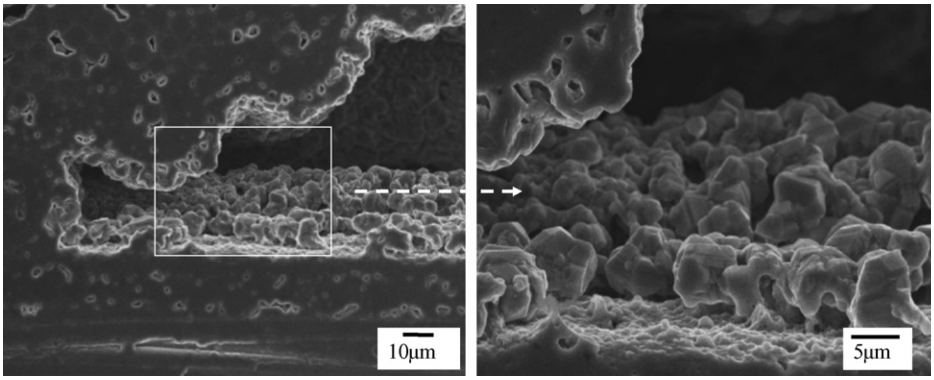

Fig. 8. SEM images from specimen after heat treatment at 2273 K, showing the formation of a layer of particles at the inner walls of a large open pore in the specimen.

resulting in the accordingly decreased strength of the composite as shown in Fig. 2. Nevertheless, the present CVI–Tyranno-SA/SiC composite demonstrated excellent thermal stability. Both the microstructure and the RT ultimate flexural strength showed almost no degradation against heat treatment temperature up to near 2073 K, and about 65 and 58% of as-fabricated strength maintained after heat treatment at 2173 and 2273 K, respectively.

4. Conclusion

A Tyranno-SA/SiC composite was fabricated by CVI method. The average ultimate flexural strength of the as-fabricated composite is 380 ± 110 MPa, which is lower than that of previously reported ones. The lower strength of the current composite was attributed to strong interfacial bonding strength owing to the very thin carbon interlayer.

The ambient temperature ultimate flexural strength showed no degradation against heat treatment temperature up to near 2073 K, beyond which the strength decreased with increasing the heat treatment temperature. About 58% of as-fabricated strength maintained even after heat treatment at 2273 K in vacuum.

Microstructure examinations revealed excellent thermal stability of the composite. No obvious change/degradation occurs to the fibers, the 20 nm carbon interlayer, and the matrix of the composite after heat treatment at temperature up to 2073 K. Significant damages to the specimen surfaces and the inner walls of relatively large open pores in the specimens were observed after heat treatment at 2273 K. These damages that might be caused by the sublimation of the CVI–SiC matrix during the heat treatment, are believed to be the main reason causing the decrease of the RT strength of the composite.

This study demonstrated that with the highly crystalline near stoicheometric Tyranno-SA fibers, CVI is an appropriate process to produce SiC/SiC composites with excellent thermal stability. The composite has shown great advantages over the Nicalon^{TM}/SiC and Hi-Nicalon^{TM}/SiC composites and the PIP-Tyranno-SA/SiC composites on both the microstructure and the RT strength upon heat treatment.

Acknowledgement

This study was financially supported by the Budget for Nuclear Research of the Ministry of Education, Culture, Sports, Science and Technology, based on the screening and counselling by the Atomic Energy Commission.

Reference

[1] B. Wilshire, F. Carreno, Deformation and failure processes during tensile creep of fibre and whisker reinforced SiC/Al_2O_3 composites, Mater. Sci. Eng. A272 (1999) 38–44.

[2] T. Noda, A. Kohyama, Y. Katoh, Recent progress of SiC-fibers and SiC/SiC-composites for fusion applications, Phys. Scripta T91 (2001) 124–129.

[3] D. Brewer, HSR/EPM combustor materials development program, Mater. Sci. Eng. A261 (1999) 284–291.

[4] A. Kohyama, M. Seki, K. Abe, T. Muroga, H. Matsui, S. Jitsukawa, S. Matsuda, Interactions between fusion materials R&D and other technologies, J. Nucl. Mater. 283–287 (2000) 20–27.

[5] A.G. Evans, Perspective on the development of high-toughness ceramics, J. Am. Ceram. Soc. 73 (1990) 187–206.

[6] R. Naslain, in: A.G. Evans, R. Naslain (Eds.), Ceramic Transactions, High-Temprature Ceramic–Matrix Composite. II. Manufacturing and Materials Development, vol. 58, America Ceramic Society, Westerville, OH, 1995, p. 23.

[7] T.M. Besmann, D.P. Stinton, E.R. Kupp, S. Shanmugham, P.K. Liaw, Fiber-matrix interfaces in ceramic composites, J. Mater. Res. Soc. Symp. Proc. 458 (1997) 147–159.

[8] L.L. Snead, O.J. Schwarz, Advanced SiC composites for fusion applications, J. Nucl. Mater. 219 (1995) 3–14.

[9] S. Guo, Y. Kagawa, Temperature dependence of tensile strength for a woven boron-nitride-coated Hi-Nicalon^{TM} SiC fiber-reinforced silicon carbide–matrix composite, J. Am. Ceram. Soc. 84 (9) (2001) 2079–2085.

[10] G.N. Morscher, Tensile stress rupture of SiC_f/SiC minicomposites with carbon and boron nitride interphases at elevated temperautres in air, J. Am. Ceram. Soc. 80 (8) (1997) 2029–2042.

[11] L. Filipuzzi, R. Naslain, Oxidation mechanisms and kinetics of 1 D-SiC/SiC composite materials. II. Modeling, J. Am. Ceram. Soc. 77 (2) (1994) 467–480.

[12] S. Guo, Y. Kagawa, Temperature dependence of in situ constituent properties of polymer-infitration-pyrolysis-processed Nicalon^{TM} SiC fiber-reinforced SiC matrix composite, J. Mater. Res. 15 (4) (2000) 951–960.

[13] S. Guo, Y. Kagawa, Tensile fracture behavior of continuous SiC fiber-reinforced SiC matrix composites at elevated temperatures and correlation to in situ constituent properties, J. Eur. Ceram. Soc. 22 (2002) 2349–2356.

[14] J. Martinez-Fernandez, G.N. Morscher, Room and elevated temperature tensile properties of single tow Hi-Nicalon, carbon interphase, CVI–SiC matrix minicomposites, J. Eur. Ceram. Soc. 20 (2000) 2627–2636.

[15] L. Filipuzzi, G. Camus, R. Naslain, Oxidation mechanisms and kinetics of 1 D-SiC/SiC composite materials. I. An experimental approach, J. Am. Ceram. Soc. 77 (2) (1994) 459–466.

[16] H. Araki, H. Suzuku, W. Yang, S. Sato, T. Noda, Effect of heat treatment in vacuum on microstructure and bending properites of SiC_f/SiC composites prepared by CVI, J. Nucl. Mater. 258–263 (1998) 1540–1545.

[17] W. Yang, A. Kohyama, Y. Katoh, H. Araki, J. Yu, T. Noda, Effect of carbon and silico carbide/carbon interlayers on the mechanical behavior of Tyranno-SA-fiber-reinforced silicon carbide–matrix composites, J. Am. Ceram. Soc. 86 (5) (2003) 851–856.

[18] T. Ishikawa, Y. Kohtoku, K. Kumagawa, T. Yamamura, T. Nagasawa, High-strength alkali-resistance sintered SiC fiber stable to 2200 °C, Nature 391 (1998) 773–775.

[19] T. Nozawa, K. Ozawa, Y. Katoh, A. Kohyama, Effect of heat treatment on microstructure and mechanical properties of stoichiometric SiC/SiC composites, Mater. Trans. 45 (2) (2004) 307–310.

[20] T.M. Besmann, B.W. Sheldon, R.A. Lowdon, D.P. Stinton, Vapor-phase fabrication and properties of continuous-filament ceramic composites, Science 253 (1991) 1104–1108.

[21] W. Yang, T. Noda, H. Araki, J. Yu, A. Kohyama, Mechanical properties of several advanced Tyranno-SA fiber-reinforced CVI–SiC/SiC composites, Mater. Sci. Eng. A345 (2003) 28–35.

[22] ASTM C 1341-97, Standard test method for flexural properties of continuous fiber-reinforced, Adv. Ceram. Comp., 2000, 509–526.

[23] N. Frety, M. Boussuge, Relationship between high temperature development of fiber–matrix interfaces and the mechanical behavior of SiC/SiC composites, Comp. Sci. Technol. 37 (1990) 177–189.

[24] W. Yang, A. Kohyama, T. Noda, Y. Katoh, T. Hinoki, H. Araki, J. Yu, Interfacial characterization of CVI–SiC/SiC composites, J. Nucl. Mater. 307–311 (2002) 1088–1092.

[25] J.J. Brennan, Interfacial studies of chemical-vapor-infiltrated ceramix matrix composites, Mater. Sci. Eng. A126 (1990) 203–223.