Investigation on Microstructure and Fracture Behavior of β-SiC Monofilament

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Abstract: In this study, the ultimate tensile strength of W-core/β-SiC monofilament was measured in the as-produced condition at room temperature. Fractographic analysis and interfacial reaction zone was characterized by using field emission scanning electron microscopy (FE-SEM) and transmission electron microscopy. Nano-indentation tests across the section of a monofilament embedded in a Ti-17 metal matrix. The Fracture mechanism was show that the key microstructural parameters which dominate damage initiation, and fracture behavior of the SiC monofilament were explained in detail.

1. Introduction

Nowadays, ceramic monofilaments are widely used in metal matrix composite materials (MMCs). Monofilaments provide stiffness and the matrix ductility in the case of metal matrix composites, which as structural materials for great potential applications in aircraft engine and hypersonic flight vehicles with light-weight high-temperature properties [1-4]. Continuous silicon carbide (SiC) monofilament produced by chemical vapor deposition (CVD) method with high strength, stiffness and creep resistance at both room and elevated temperatures [5], it can be used as prior reinforcement for metal matrix composites, especially for titanium alloy matrix composites [6]. In fact, the microstructure of SiC monofilaments has great related to fabrication process parameter, e.g, the reactor configurations and deposition conditions and so on. In order to understand high-performance SiC monofilament as reinforcement and predict the proper mechanical behavior in MMCs, it is necessary to study the relationship between microstructure and fracture behavior in detailed.

SiC fibers are deposited on the tungsten core by CVD method. The high fabricated temperature leads to an interfacial reaction layer between SiC and W-core. Because a layer of pyrolytic carbon is deposited on the outermost layer of the monofilament as a protective coating, the defects on the surface of the fiber are eliminated, and the fracture mechanism of the single fiber under axial tensile load is mainly dominated by the internal defects of the fiber and the interfacial reaction layer [7-9]. SiC fibers grow radially from the surface of the tungsten core in the form of columnar crystals. Due to the process stability and other reasons, the crystal morphology of SiC is not completely columnar, and the growth direction of the crystal is not strictly in accordance with the < 111 > crystal direction. It is worthy noted that the exploration on the detailed microstructure of SiC fibers along radius and correlated the mechanical behavior to further understand fracture mechanisms, which can provide technical support for material design and aircraft part development.

In this work, tensile strength of 50 specimens were obtained and divided into three types according to mean tensile strengths, Type A monofilaments ~2.6GPa, Type B monofilaments ~3.2GPa, Type C monofilaments ~3.8GPa, and then the fractographic were analyzed under SEM. The detailed microstructure of three types monofilaments embedded in Ti-17 matrix along radius were studied by FE-SEM and HRTEM, nano-indentation is explored as a tool for measuring Young's modulus of monofilaments.

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2. Materials and Experimental

A two-stage CVD reactor with a cold wall was used to make the SiC monafilament coated with surface C coating at atmospheric pressure. The tungsten wire (~15 μm in diameter) enters the reaction chamber through mercury seals which permit electric heating of the substrate. In the first stage, prior to SiC deposition, the tungsten filament was firstly cleaned via heating in a hydrogen atmosphere. Subsequently, the cleaned W filament entered a 100-cm-length CVD reactor for SiC deposition.

Tensile tests have been performed on the model filaments with the help of an electro-mechanical tensile testing machine [5]. The filamentary specimens were prepared following a standardized procedure [6]. Fractographic analysis was performed using a scanning electron microscopy with backscatter electron (SEM, Zeiss Sigma 300, GER), and scanning electron microscopy with Inlens detector(SEM, Zeiss Ultra55, GER), the fractured specimen was further grinded and polished along fiber direction(Longitudinal direction) and vertical fiber direction(Transverse cross section). In order to study the microstructure of samples, focused-ion beam (FIB, FEI/Thermo Fisher Scientific Quanta 3D FEG Dual Beam, Hillsboro, USA) was used to prepare specimen of SiC fiber for transmission electron microscopy (TEM, FEI/Thermo Fisher Scientific Titan G2 ChemiSTEM, Hillsboro, USA) investigation. Nano-indenter (HYSITRON 950, Thermo-fisher-science, USA) was used to examine fiber and matrix micromechanical property.

3. Results and Discussion

3.1 Tensile properties and Fractography

The ultimate tensile strength (UTS) of 50 monofilaments are measured and three types monofilament are classified by tensile test results, Type A monofilaments′ UTS ~2.6GPa, Type B monofilaments′ UTS ~3.2GPa, Type C monofilaments′ UTS ~3.8GPa, tensile strength scattering diagram is shown in Fig.1.

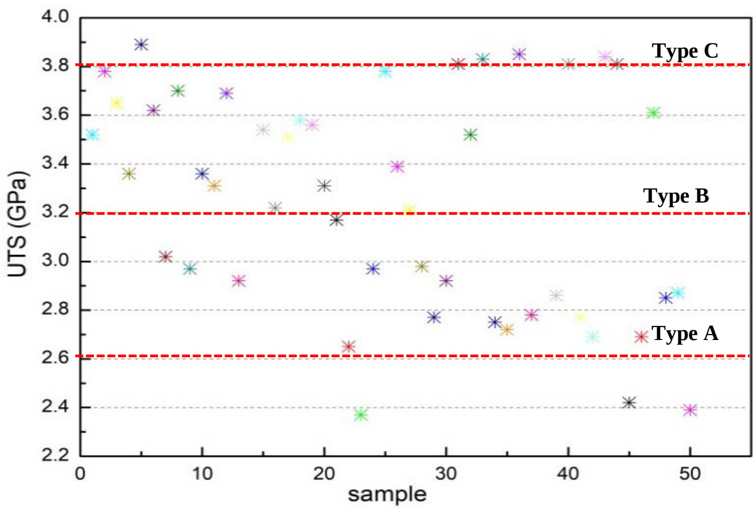

Fig.1 Tensile strength distribution diagram

Because of SiC monofilament surface were protected by outmost carbon layer, surface defect dominated fracture mode have not occur during tensile test. In the case of W-core SiC monofilament, the interfacial reaction between W-core and SiC bulk exists in each monofilament, which would be provided interfacial bonding strength improved monofilament stability, however, like a coin has two faces, cracks initiated at interfacial reaction layer due to its brittle fracture [11]. From various observations of fractography of three tpyes monofilament, it is asserted that fracture initiates at the interface between tungsten core and fibre bulk SiC for type A and type B, except these, cracks initiates at inside defects in SiC of type A monofilament, as shown in Fig.2a~Fig.2d. Initially, cracks propagated in a fan shape along radial through the SiC. At the same time the cleavage facet and intergranular is observed at the tungsten core near the interfacial reaction layer. More than one crack initiation sites are observed in type A and type B monofilament, crack propagated along hoop direction at interfacial reaction layer surrouding the W-core, at same time, carcks propagated outwards (through the SiC) and inwards (through the core) until the new fracture surface created. Finally, inward cracks converged to a point in W-core, beside that, outward cracks through the SiC bulk and carbon coating created the flat plane as cross-section of monofilament. This characteristic fracture surface was also reported by Gambone and Gundel [1] in a similar fibre (Trimarc 1) , M.G. Leiva et al. have proved tensile fracture initiates at the W/SiC interface in Sigma SM1140+ fiber [7]. Luo et al. [10] and Faucon et al. [8] further studied the W/SiC interface evolutions induced by high-temperature heat exposure experiments and their effect on tensile strengths.

The fracture mode of type C is different from that of type A and type B, cracks initiated at local area in W-core near the interfacial reaction layer, as shown in Fig.2e and Fig.2f. From this point, crack propagated inward through the W-core and outward created intergranular cracks in W-core near the interface like as other two type monofilament. At the same time, cracks process along the interface around W-core at first, and then new fracture surfaces are created outwards through the SiC bulk, due to the time difference between crack initiation and propagation at interface, there are unflatter steps plane developed on the SiC bulk surface. High-magnification SEM images are presented in Fig. 2, in the interface of W/SiC, ~0.35 μm-thickness interfacial reaction layer were measured for type A monofilament, ~0.32μm-thickness for type B and ~0.29 μm-thickness for type C monofilament, thickness of type B and type C monofilament have closing value that approaches the Zhang's result of 0.32 μm and 0.30 μm in the W-core SiC fiber that represent UTS ~3.2Gpa and UTS ~3.8Gpa [19]. The relationship between the thickness of interfacial reaction layer and UTS is shown in Figure 3. With the increase of UTS, the thickness of interfacial reaction layer decreases linearly.

It is obvious that the degree of interfacial reaction has a direct effect on the fracture mechanism of the fiber, because the outer surface of the fiber is coated with 1.5 μm. The interface-dominated has play more important role in fracture mechanisms of SiC monofilament under tensile load. Base on the fracture mode for three type monofilaments, the thickness of W/SiC interface should be a crucial parameter determining their tensile strength except the inside defects of SiC monofilament. Furthermore, another phenomenon should draw attention that is crack initiated at inside of W-core near the interfacial layer not in or at interfacial layer, which was found in type C monofilament. According to the report, a critical thickness of W/SiC interfacial layer exists in SiC monofilament, the critical thickness of W/SiC interface calculated by Liu ect. is ∼0.55 μm in similar W-core SiC fibers [12], above which the tensile strength of SiC fibers would degrade. In this case, almost same thickness (∼0.32 − ∼0.35 μm) of the W/SiC interfacial layer existing for type A monofilament and type B monofilament is yet below the critical thickness, which should not dominate the observed large difference in tensile strength [13-14,19].

Notably, in contrast to smooth fractograph of type A and type B monofilaments, fracture cleavages through different crystallographic grains and planes make more steps at different heights on the surface of SiC bulk for type C monofilament, which mean crack propagating along radial direction is not straight through the SiC bulk but crack deflections dominated the process. Accorrding to fracture energy, longer path and cracks deflections mean it need more energy to go through the cross-section than other two type monofilament, this may be responsible for higher UTS in type C monofilaments than those. Besides, the different UTS in type A monofilament and type B monofilament should primally come from the discrepant microstructure of SiC bulk due to fabrication process. In addition to the W/SiC interfacial layer, microstructure of W-core, SiC bulk and C-coating all have key effects on the UTS of monofilament.

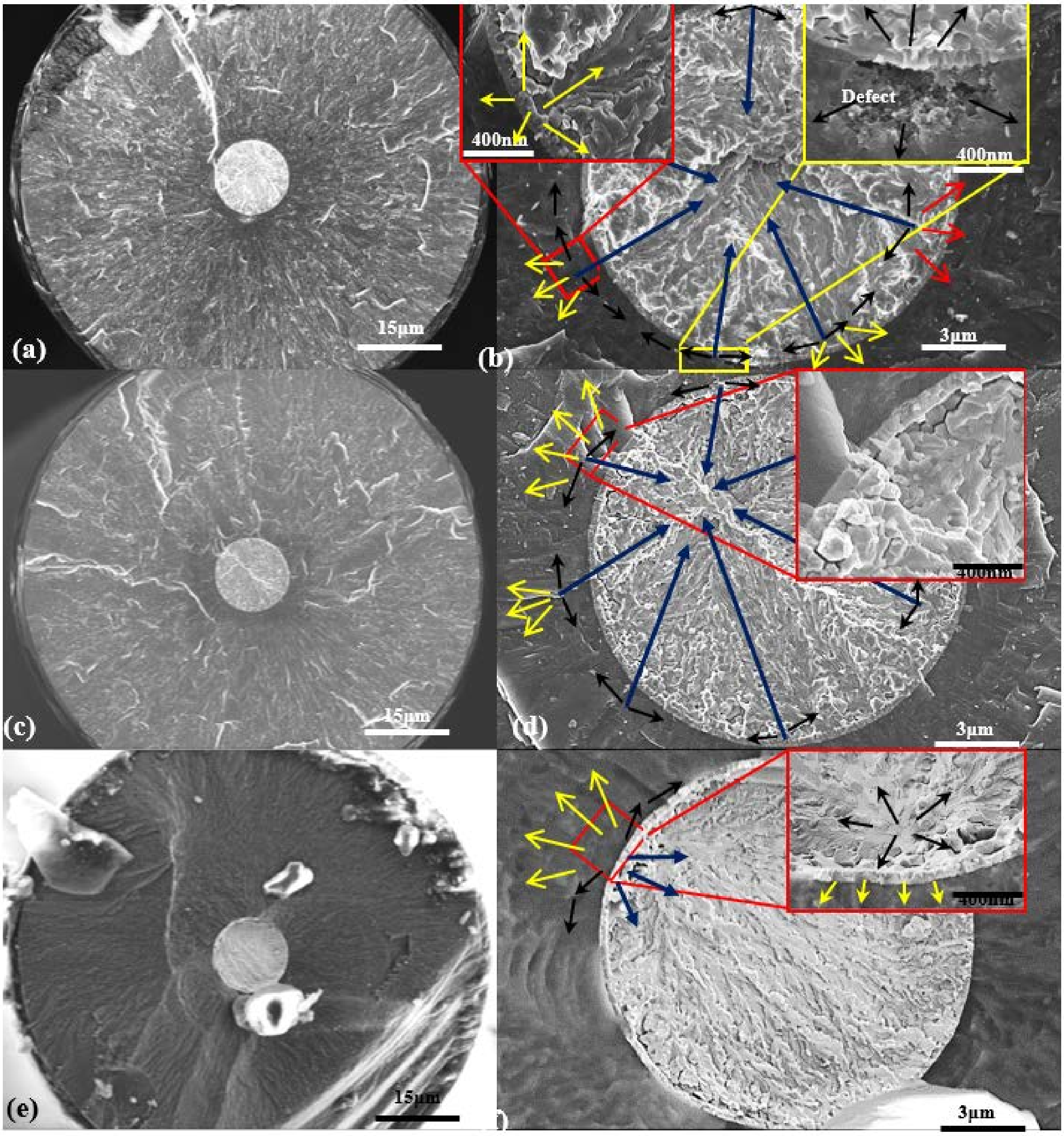

Fig. 2 The fractography of SiC monofilament under tensile load: a) b) The fracture morphology of Type A; c) d) The fracture morphology of Type B; e) f) The fracture morphology of Type C.

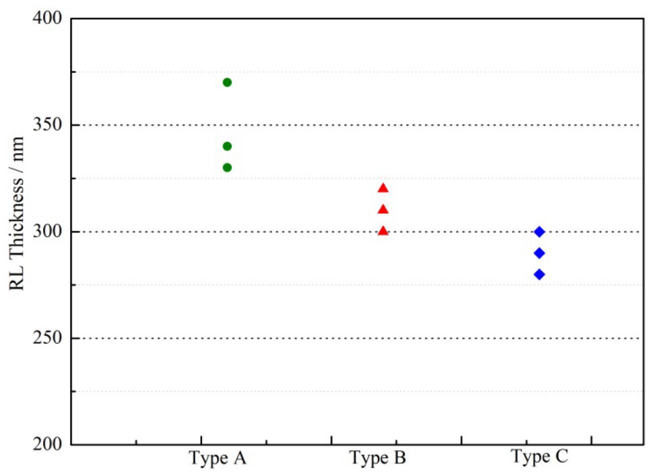

Fig.3 Reaction layer thickness measurement of three type monofilaments

3.2 Microstructure and micromechanical behavior of SiC monofilament.

Focusing on SiC bulk of three type monofilament, after cracks progresses along radial direction, typical columnar growth from inner to outer zones can be observed in all monofilaments. Generally, coarse columnar appear in the inner zones of SiC along radial direction, and grains are significantly refined and transit into fine columnar grains in the outer zones. Obviously, the SiC grains size and crystalline morphology have great related to UTS of SiC monofialment. Accordingly, the more detailed structure information would be further explored to clarify their structure discrepancy contributing to the different tensile strength [15-16].

Three type SiC monofilaments were embedded in Ti-17, and cross-section of specimen were prepared. FE-SEM with In-len SE2 detector was used to observe the microstructure of SiC monofilament. Type B and type C have similar microstructure, columnar grain appear in the inner zone along radius direction grew to outer zone, and then coarse grain transform to fine columnar grain as shown in Fig.4a. The ring pattern was found on cross-section of type A monofilament by contrast the uniform and continuous microstructure of type B and typ C as shown in Fig.4b. The microstructure of SiC fiber was used TEM to examine with SADP mode shown that a colony small size grain have different growth direction with mainly growth direction of columnar grain [18].

As further revealed by the nanoindentation results on the cross section of two kinds microstructure of monofilament (Fig.5), higher modulus and stability appears in type C monofilament across the radius than type A monofilament. The micromechanical behavior results show that the ring pattern has a great influence on performance stability of SiC monofilament, which should mainly come from the high-quality SiC columnar grains. The high-crystallinity SiC columnar grains across the radius for type C should be contributed to its higher UTS via forming rougher stepped fracture morphology than type A monofilament.

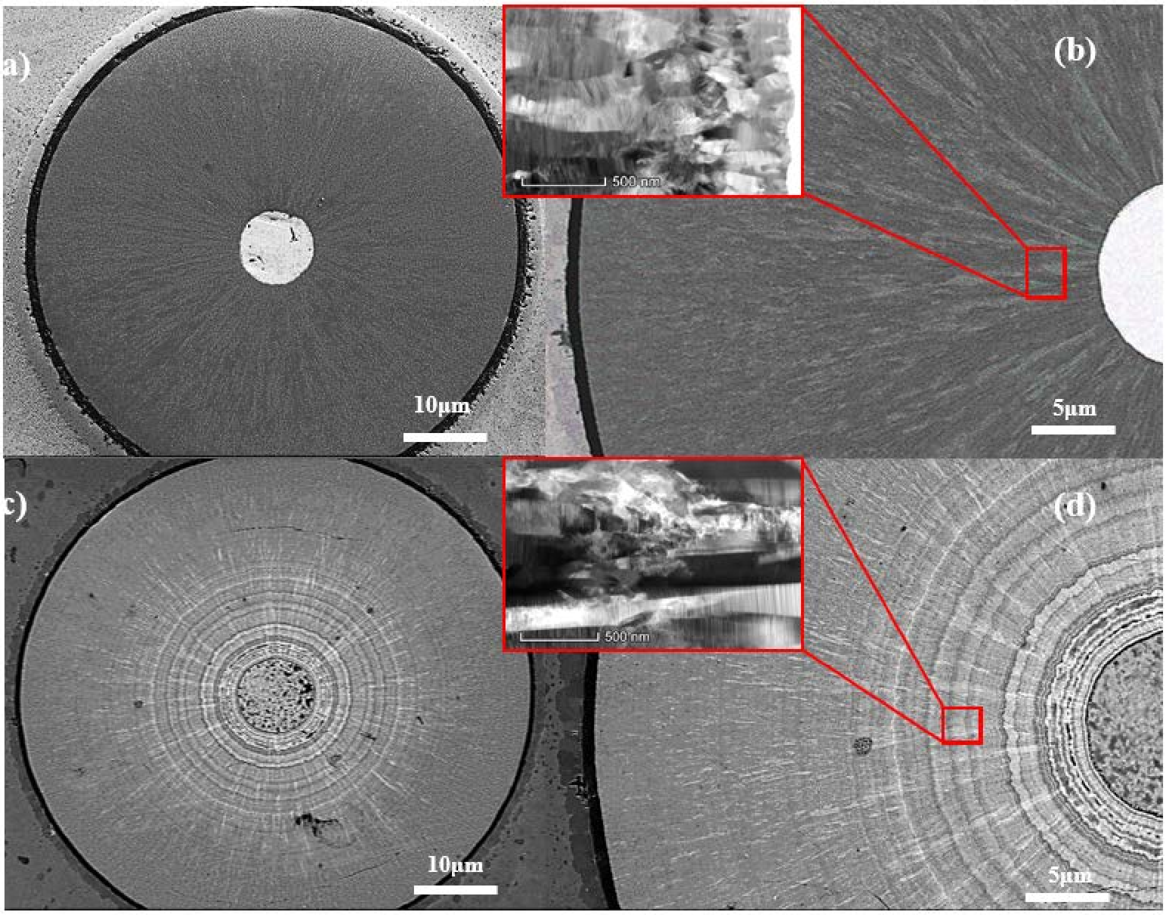

Fig. 4 The microstructure of SiC monofilament: a) b) Cross-section of Type C embedded in Ti-17 matrix; c) d) Cross-section of Type A embedded in Ti-17 matrix.

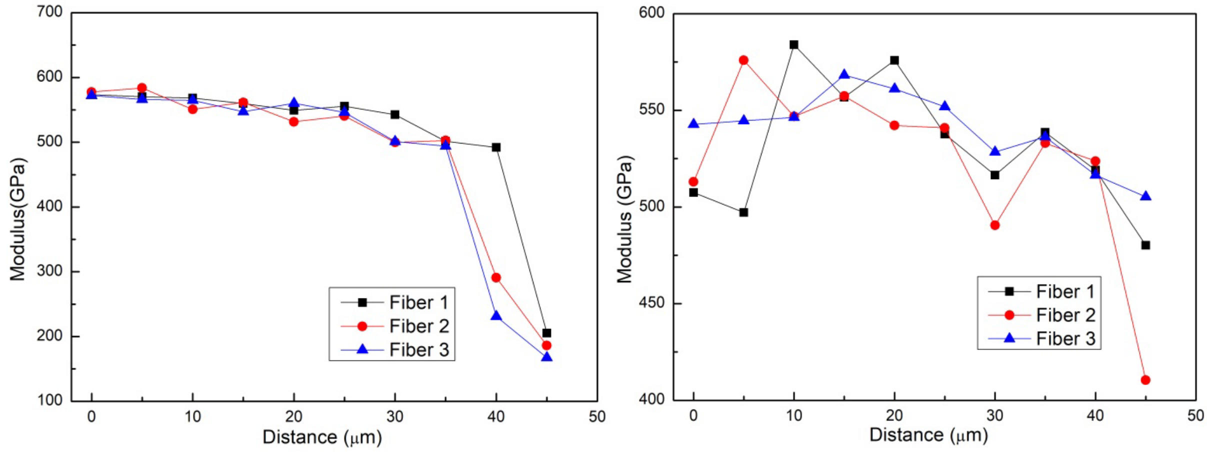

Fig. 5 The radial modulus curve of different type monofilaments

4. Conclusions

(1) As for both Type B and Type C monofilaments, the high-crystallinity columnar β-SiC grains grew along radius direction with their long axes, Type A monofilaments appear ring patterns around W-core from inner zone to outer zone which is a large number of fine SiC grains with different grew direction, thereby the growth of columnar grains is blocked and the continuity of grains is destroyed, but yet retaining some surviving columnar grains that even extend to SiC surface.

(2) The different fracture mode appear in different type monofilaments, interface-dominated fracture mode appear in type A and type B monofilament with lower UTS, which have more than one initiating sites at the interfacial zone between W core and SiC bulk took place at the same time. Type C monofilament with higher UTS initiated at inside of W-core near interfacial layer, which only have one crack setup site. After crack propagation, a cleavage facet appears in the W core and a fan-shaped feature occurs in the SiC bulk. The high-crystallinity SiC columnar grains across the radius for type C monofilament introduced much rougher stepped fracture morphology than other two type monofilaments, which is responsible for its higher tensile strength.

References

[1] Gambone ML, Gundel DB. The effect of W-core/SiC reaction on the strength of SiC fibres in SiC/Ti-alloy composites. Key Eng Mater 1997;127–131:1251–8.

[2] M. V. Rix, M. Baker, M. J. Whiting, et al. An Improved Silicon Carbide Mono filament for the Reinforcement of Metal Matrix Composites[J]. The Minerals, Metals & Materials Series-2017, Proceedings of the 3rd Pan American Materials Congress, edited by M.A. Meyers et al. The Minerals, Metals & Materials Society, 2017, pp. 317–324.

[3] Shatwell RA, Dyos KL, Prentice C, Ward Y, Young RJ: Microstructural analysis of silicon carbide mono filaments[J]. J Microsc, 2001, 201:179–188.

[4] Shatwell RA: Adhesion of SM1140+ coatings to silicon carbide substrate in Sigma monofilament, Mater Sci Technol, 10 (1994) 552-557.

[5] Y Wang, G Zhang, X Zhang, Q Yang, et al. Advances in SiC fiber reinforced titanium matrix composites, Acta Metall Sin, 2016, 52(10):1153–1170.

[6] RJ Zhang, YQ Yang, WT Shen, et al. Microstructure of SiC fiber fabricated by two- stage chemical vapor deposition on tungsten filament[J]. J Cryst Growth, 2010a, 313(1):56–61.

[7] García-Leiva MC, Ocaňa I, Martín-Meizoso A, Martínez-Esnaola JM: Fracture mechanics of Sigma SM1140+ fibre[J]. Engineering Fracture Mechanics, 69 (2002) 1007–1013.

[8] A. Faucon , T. Lorriot , E. Martin, et al. Fracture behaviour of model silicon carbide filaments[J]. Composites Science and Technology, 61 (2001) 347–354.

[9] S. Guo, Y. Kagawa, Y. Tanaka, C. Masuda, Microstructure and role of outermost coating for tensile strength of SiC fiber[J]. Acta Mater. 46 (14) (1998) 4941–4954.

[10] X. Luo, P. Guo, Y. Yang, N. Jin, S. Liu, Z. Kou, S. Wu, Microstructure, tensile strength and thermostability of W-core SiC fibers with or without carbon coating, Mater. Sci. Eng. A 647 (2015) 265–276.

[11] S. R. Nutt, F. Wawner, Silicon carbide filaments: microstructure, J. Mater. Sci. 20 (6)(1985) 1953–1960.

[12] S. Liu, Y. Yang, X. Luo, B. Huang, et al. Microstructure and mechanical property of high growth rate SiC via continuous hot - wire CVD[J]. J. Am. Ceram. Soc.102 (9) (2019) 5656–5667.

[13] T. Cheng, I. Jones, R. Shatwell, et al. The microstructure of sigma 1140+ SiC fibres[J]. Mater. Sci. Eng. A 260 (1–2) (1999) 139–145.

[14] X. Ning, P. Pirouz. The microstructure of SCS-6 SiC fiber[J]. J. Mater. Res. 6 (10)(1991) 2234–2248.

[15] S. Guo, Y. Kagawa, Y. Tanaka, C. Masuda, Microstructure and role of outermost coating for tensile strength of SiC fiber[J]. Acta Mater. 46 (14) (1998) 4941–4954.

[16] F.W. Wawner, A.Y. Teng, S.R. Nutt. Microstructural characterization of SiC (SCS) filaments[J]. Sampe Quarterly, 14 (3) (1983) 39–45.

[17] D. Gundel, F. Wawner, Experimental and theoretical assessment of the longitudinal tensile strength of unidirectional SiC-fiber/titanium-matrix composites[J]. Compos.Sci. Technol. 57 (4) (1997) 471–481.

[18] WX Zhao, Octav. Ciuca, XG Yang, et al. Characterization on microstructure of interface and failure analysis of SiC fiber reinforced Ti-17 composites under tension load[J]. IOP Conference Series: Earth and Environmental Science, 2021.

[19] Y. Zhang, M. Wen, G.X. Sun, et al. Insight into microstructural architectures contributing to the tensile strength of continuous W-core SiC fiber[J]. Journal of the European Ceramic Society 40(2020)5147-5161.