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Fabrication of SiC fibres from yttrium-containing polycarbosilane

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Abstract: The yttrium as a sintering aid was introduced into polycarbosilane (PCS) to prepare yttrium-containing PCS (PYCS). Two types of yttrium-containing SiC fibres, the SiC(OY) fibres and the SiC(Y) fibres, were fabricated with PYCS. The structural evolution and the associated properties on changing from SiC(OY) to SiC(Y) fibres during the sintering process were studied. The chemical composition of the SiC(OY) fibres is SiC_{1.53}O_{0.22}Y_{0.005} with an amorphous structure. The composition of SiC(Y) fibres is SiC_{1.23}O_{0.05}Y_{0.005}. The fibres are composed of a large number of β -SiC crystallites with a size of 50 nm and a small amount of α -SiC crystalline. The tensile strength and fracture toughness of the SiC(OY) fibres are 2.25 GPa and 2.37 MPa·m^{1/2}, respectively, and 1.61 GPa, 1.91 MPa·m^{1/2}, respectively for SiC(Y) fibres. The SiC(Y) fibres have a higher thermal stability than the SiC(OY) fibres. **Key words:** polycarbosilane; yttrium; silicon carbide; fibre

1 Introduction

The pyrolysis of polycarbosilane (PCS) used for the preparation of silicon carbide (SiC) fibres has been extensively investigated [1-6]. Since the pioneering work of YAJIMA et al [1], three generations of SiC fibres have been produced from PCS, according to the spinning/curing/pyrolysis route. The first generation fibres with very high percentage of oxygen and excess carbon, such as Nicalon [1] and Tyranno Lox M [7] from PCS and polytitanocarbosilane produced respectively by the thermal oxidation curing method, were limited to the maximum temperature of around 1000 °C above which the fibres crept and ultimately decomposed. The later generation fibres were produced by cross-linking the precursors with electron irradiation to avoid the introduction of oxygen. These fibres are known as Hi-Nicalon [8], which contains 0.5% oxygen and Tyranno Lox E [9] which approximately contains 5% oxygen. The Hi-Nicalon fibres have a much higher thermal stability than the Nicalon fibres. However, the Hi-Nicalon fibres not only consist of SiC nanocrystals but also contain a large excess of turbostratic carbon which affects the oxidation and the creep resistance of the Hi-Nicalon fibres [10-12]. To reduce the free carbon content and eventually to improve the high temperature properties of the fibres, much effort has been devoted to developing near-stoichimetric $(n(C)/n(Si)\approx 1)$ SiC fibres. Therefore, the third generation fibres were nearstoichiometric fibres. The most successful examples include Hi-Nicalon-S from Nippon Carbon Company of Japan [13], Sylramic from ATK COI Ceramics of U.S.A. [4], and Tyranno-SA from Ube Industries Company of Japan [14–19]. The creep and strength retention at high temperatures of the third generation fibres approach those of bulk SiC.

Indeed, the modification of the PCS with metal (Ti, Zr or Al) complex compound or alkoxide offers a convenient route for improving high temperature

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resistance of SiC fibres [14]. This route allows the introduction of trace sintering aids homogeneously distributed in SiC fibres. For example, the Tyranno Lox M SiC fibres were achieved by polytitanocarbosilane, which was prepared by the crosslinking of PCS with titanium tetraalkoxide [7]. The commercial Tyranno ZM SiC fibres were produced by polyzirconocarbosilane, which was synthesized by the condensation reaction of PCS with zirconium acetylacetonate [20]. The addition of aluminium acetylacetonate to PCS gave rise to the polyaluminocarbosilane, which was pyrolyzed in an inert atmosphere to lead to the formation of the Si–Al–O–C fibres [16].

Yttrium as a sintering aid had been extensively explored in the research of sintering SiC ceramics [21]. However, to the best of our knowledge, there is no report on yttrium-containing SiC fibres. In this study, yttrium acetylacetonate (Y(AcAc)₃) was used to introduce traces of yttrium as a sintering aid for preparation of SiC fibres by the modification of PCS. The two types of yttrium-containing SiC fibres named SiC(OY) and SiC(Y) fibres were fabricated by the use of yttrium-containing PCS (PYCS). The microstructure and chemical composition of the two kinds of fibres were investigated by chemical element analysis, X-ray diffraction (XRD), scanning electron microscopy (SEM), Raman spectroscopy and high resolution transmission electron microscopy (HRTEM).

2 Experimental

2.1 Materials

Y(AcAc)₃ was obtained from Aldrich (reagent grade of 99%). The PCS was prepared according to the process in Ref. [1] and its structure can be ideally represented by [SiHCH₃CH₂]_n. The PCS has a softening point of 200 °C and relative molecular mass of 1500 with a dispersion coefficient of 1.8. All samples described in this investigation were manipulated in a purified nitrogen or argon atmosphere.

2.2 Preparation of fibres

The PYCS was synthesized by the reaction of 100 g PCS with 6 g Y(AcAc)₃ at 300–320 °C for 4 h. During the process of synthesis, the yttrium element was introduced into the PYCS by the cross-linking reaction. The obtained PYCS was continuously melt-spun at 300 °C and then cured by chemical vapour curing (CVC) process up to 410 °C for 5 h. The cured PYCS fibres were heated up to 1000 °C with a heating rate of 2 °C/min and held for 30 min to produce the SiC(OY) fibres. Then, the SiC(OY) fibres were converted into sintered SiC(Y) fibres by heating in argon up to 1800 °C for 1 h. The typical properties of SiC(OY) and SiC(Y) fibres are listed in Table 1, compared with Tyranno SA

fibres [5].

To investigate the effect of the heat treatment on the tensile strengths, the SiC(OY) and SiC(Y) fibres were thermally exposed to various heat treatment temperatures (1000, 1200, 1400, 1600, 1800, and 1900 °C) in argon for 1 h. In order to evaluate their oxidation resistance, the fibres were exposed in air at 1000–1500 °C for 1 h.

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Fibre	Density/	Diameter/	Strength/	Fracture toughness/	
1 1010	$(g \cdot cm^{-3})$	μm	GPa	$(MPa \cdot m^{1/2})$	
Tyranno SA [9]	3.1	7.5–10	2.8	2.70	
SiC(OY)	2.25	10-12	2.25	2.37	
SiC(Y)	2.95	8.0-10	1.61	1.91	

2.3 Characterization

The fibres were characterized by the following ways. The tensile strength of the samples was measured using a tensile testing machine (Model-YG-002, Taicang, China) with a gauge length of 25 mm at room temperature. The mean value for 25 measurements was taken as the average tensile strength of each fibre. Fibre diameter was determined by a microscope. The densities of ceramic fibers were measured at room temperature by the sink-float method. The fracture toughness of the fibres was determined by introducing an artificial notch directly into the fibre test specimens with a focused Ga⁺-ion beam according to Ref. [22]. Determination of silicon, carbon, oxygen and yttrium concentrations in the fibres was carried out. For the measurement of silicon content, a ceramic fibre sample was attacked by a fused alkali salt followed by neutralization with hydrochloric acid. Silicon content was determined by gravimetry of SiO₂ obtained from above solution. In order to measure yttrium content, a ceramic fibre sample was decomposed by means of fused sodium hydroxide and then neutralized with an acid for spectrophotometry. Carbon and oxygen content were determined by means of a combustion volumetric method with a carbon/sulfur analyzer (HORIBA EMIA-320V) and oxygen/nitrogen analyzer (HORIBA EMGA-620V), respectively. Raman spectroscopy was recorded between 500 and 3000 cm⁻¹ on a RM 1000 spectrometer (Renishaw) with a 1 µmmicroprobe (514 nm argon laser). The morphology of the fibres was examined on a scanning electron microscope (SEM, JEOL JSM-5600LV, Japan), operated at 20 kV. The crystal phase of the fibres and size of β -SiC crystallites were determined by X-ray diffraction (XRD, Cu K_{α} radiation, Siemens D500, Germany). The microstructure of the monofilaments was studied at the nanometer scale on a high resolution transmission electron microscope (HRTEM, Tecnai F30, Netherlands), operated at 300 kV.

3 Results and discussion

3.1 Compositions and structural evolution from SiC(OY) to SiC(Y) fibres

The average bulk compositions of SiC(OY) and SiC(Y) fibres measured by chemical element analysis are listed in Table 2. The SiC(OY) fibres are rich in carbon (C/Si in mole ratio, 1.53), containing 6.93% oxygen and 0.91% yttrium. The formula of SiC(OY) fibres is SiC_{1.53}O_{0.22}Y_{0.005}. After sintering at 1800 °C, the oxygen content and the ratio of C to Si decrease obviously, the oxygen content decreases by 5.16% and corresponding carbon was taken away with the pyrolytic decomposition of SiC_xO_v . The yttrium content is slightly increased due to the removal of some oxygen and free carbon. For SiC(Y) fibres, composition was confirmed as chemical composition of SiC_{1.23}O_{0.05} $Y_{0.005}$. The *n*(C):*n*(Si)=1.23 of the SiC(Y) fibres shows that the SiC(Y) fibres are still rich in carbon, which is consistent with the following Raman spectrum result.

Table 2 Chemical analysis of SiC fibres

Sample	w(Si)/ %	w(C)/ %	w(O)/ %	w(Y)/ %	<i>n</i> (C)∕ <i>n</i> (Si)	Formula
SiC(OY)	55.63	36.45	6.93	0.91	1.53	SiC _{1.53} O _{0.22} Y _{0.005}
SiC(Y)	63.17	33.31	1.77	1.04	1.23	SiC _{1.23} O _{0.05} Y _{0.005}

Raman spectroscopy is one of the most sensitive methods for the characterization of the different modifications of carbon [20]. Typical Raman spectra obtained from the surface of the SiC(OY) and SiC(Y) fibres are displayed in Fig. 1. Two peaks at 1350 and 1580 cm⁻¹ assigned to the typical vibration frequencies of free carbon were observed. The peak at 1350 cm⁻¹ is due to the "unorganized" carbon involving lattice defects, while the peak at 1580 cm⁻¹ is due to the E_{2g} symmetric mode vibration in graphite [23]. The free carbon vibration bands of the spectrum of the SiC(Y) fibres are sharper than those for the SiC(OY) fibres, due to the SiC(OY) fibres, the Raman lines characteristics of crystalline SiC are almost unobservable. On the contrary, two sharp lines occur at about 790 and 967 cm⁻¹ attributed to β -SiC for the SiC(Y) fibres, due to a better crystallization state.

Figure 2 shows SEM images of the surface and cross section of the SiC(OY) and SiC(Y) fibres. The



Fig. 1 Raman spectra of surface of SiC(OY) and SiC(Y) fibres



Fig. 2 SEM images of surface and cross section of SiC(OY) and SiC(Y) fibres: (a) Surface of SiC(OY) fibre; (b) Section of SiC(OY) fibre; (c) Surface of SiC(Y) fibre; (d) Section of SiC(Y) fibre

SiC(OY) fibres have a very smooth surface without any observable flaws and their fracture surface is a typical glassy appearance (Figs. 2(a) and (b)). After sintering the SiC(OY) fibres at 1800 °C, the SiC(Y) fibres still show a smooth surface and densified structure (Fig. 2(c)). On the fracture surface of the SiC(Y) fibres (Fig. 2(d)), polycrystalline structure and transgranular fracture behaviour are observed. Under a higher magnification view, some particles of about 50 nm in size, presumably crystalline SiC, can be seen to be connected tightly with each other. Furthermore, after sintering at 1800 °C, the SiC(OY) fibres shrink by a remarkable 20% from 10 to 8 µm in average diameter. The high shrinkage of fibres indicates a high level of densification, and suggests that pores and defects formed by the removal of oxygen and excess carbon might be efficiently healed by calcination in the presence of yttrium element. This is also consistent with other reports which describe the use of Y₂O₃ as a sintering aid [24].

The microstructure evolution of the SiC(OY) fibres prepared at various sintering temperatures under Ar for 1 h was characterized by XRD. Figure 3 displays the XRD patterns. At 1000 °C, the SiC(OY) fibres exhibit a broad scattering hump (at an angle of 36.5°), which shows that the SiC(OY) fibres should be in amorphous structure. Above 1000 °C, the XRD patterns become sharp with increase of the sintering temperature, so that crystal growth must occur. At 1200 °C, three peaks at 2θ =36.5°, 60°, and 73° attributing to the (111), (220) and (311) plane of β -SiC, respectively, are still broad. The broad XRD patterns show that the SiC(OY) fibres prepared at 1200 °C still have the amorphous structure, but contain β -SiC microcrystallinity. At 1400 °C, the diffraction peaks (220) and (311) of β -SiC are separated and are narrower, indicating that crystallization of β -SiC is shown. At 1600 °C, new peaks corresponding to α -SiC appear and are sharpen at 1800 °C, revealing a wellcrystallized material. On the basis of above results, it is



Fig. 3 XRD patterns for SiC(OY) fibres prepared at various sintering temperatures under Ar for 1 h

concluded that the crystallization process of the SiC(OY) fibres have been clearly presented in the XRD patterns, the SiC(Y) fibres have high crystalline structure, and the transformation temperature of amorphous state into crystallization state is 1400 °C.

The variation of the mean grain size of β -SiC, as a function of the sintering temperature, is given in Table 3. The mean grain size of the fibres is determined by Scherrer line width method with the (111) reflection of XRD patterns. The mean β -SiC grain size grows up with increasing the sintering temperature. The crystallinity of the fibres is closely related to the sintering temperature and the composition of fibres. Below 1400 °C, the content of oxygen and carbon can prevent the nucleation and growth of SiC phase in fibres. A slight increase in the grain size is observed. From 1400 to 1600 °C, the oxygen and free carbon are released, and there is a sharp grain growth. This is also why the tensile strength degraded during this temperature stage. At 1800 °C, the grain size of β -SiC reached 47.5 nm.

Table 3 Mean grain size of β -SiC crystalline of SiC(OY) fibres prepared at various sintering temperatures under Ar for 1 h

Temperature/°C	Average grain size/nm
1000	0
1200	1.7
1400	2.8
1600	18.9
1800	47.5

Figure 4(a) shows the TEM images of the SiC(Y) fibres. The TEM clearly reveals a microcrystalline structure. The β -SiC grains sintered together with the grain size of around 50 nm, which is in good agreement with the XRD results. A clean grain boundary with about 1 nm in thickness was found by HRTEM observation as well (Fig. 4(b)). It is difficult to conclude if an amorphous phase exists at the grain boundary from this micrograph. However, two grains can be tightly bonded together by this boundary. A fibre with such boundary phase is expected to have good thermal stability and environmental resistance, which is verified by the following mechanical properties. The typical stacking faulted β -SiC can also be observed in the SiC(Y) fibres (Fig. 4(c)), which was confirmed by the corresponding Fourier transformation (Fig. 4(d)).

3.2 Mechanical properties evolution from SiC(OY) fibres to SiC(Y) fibres

The tensile strength of the SiC(OY) fibres prepared at various sintering temperatures in argon is shown in Fig. 5. The change of the tensile strength is divided into



Fig. 4 HRTEM images of SiC(Y) fibres (a), gain boundary between two β -SiC grains (b), stacking fault β -SiC grain (c) and corresponding Fourier transformations (d)



Fig. 5 Tensile strength of SiC(OY) fibres prepared at various sintering temperatures

four stages. Firstly, the tensile strength increases with increasing temperature, and the SiC(OY) fibre reaches the highest tensile strength at 1300 °C. Secondly, the tensile strength begins to decrease at a temperature above 1300 °C. It has the lowest tensile strength at 1600 °C. It is found that the reason for the decrease in tensile

strength is due to the formation of porosity and the volatile of CO and SiO during the pyrolysis process. Thirdly, the tensile strength increases again when the temperature is higher than 1600 °C. However, when the heat treatment temperature exceeds 1800 °C, the tensile strength of the SiC(OY) fibre decreases again. The tensile strength change of the SiC(OY) fibre with pyrolysis temperatures looks like saddle shape, which is similar with that of Tyranno-AM fibre [5].

For safety design for practical use of ceramic materials, the fracture toughness is one of the most important parameters. To determine the fracture toughness, the focused-ion beam micromachining method is applied to introducing an artificial notch directly into a small diameter fibre. Figure 6 shows the appearance of notched SiC(OY) fibre and SiC(Y) fibre. The notch tip radius is 25 nm in average. From the measured fracture strength and notch depth and the correction factor calculated by a finite element method according to Ref. [23], the fracture toughness of the SiC(OY) fibres and SiC(Y) fibres is determined to be



Fig. 6 Appearance of side surface of notched fibres: (a) SiC(OY) fibre; (b) SiC(Y) fibre

2.37 and 1.91 MPa·m^{1/2}, respectively.

Figure 7 shows the room temperature tensile strength of the SiC(Y) fibres after thermal exposure at various temperatures in argon for 1 h, compared with Hi-Nicalon and Tyranno SA fibres. The tensile strength of SiC(Y) is maintained up to 1500 °C. Above 1500 °C, the tensile strength of the SiC(Y) fibres reduces slowly, after thermal exposure at 1800 °C in argon, 73.2% of its initial strength is preserved. On the other hand, the tensile strength of a representative commercial Hi-Nicalon fibre is reduced to 65% and 40% of the initial strength by heating in argon for 1 h at 1550 °C and 1800 °C [5]. The higher tensile strength retention of SiC(Y) fibres after sintering at 1800 °C is presumably attributed to two reasons. The first is associated with the incorporated yttrium, which may play a role as a sintering aid in densifying the fibres at high temperatures. Secondly, PYCS green fibres are consolidated by the combination of brief air-curing and further CVC curing in an inert atmosphere to minimize the oxygen content of the ceramic fibres. The reduction in the SiC(Y) fibre tensile strength observed after sintering at 1800 °C is likely caused by crystal coarseness. In the case of the



Fig. 7 Tensile strength changes of SiC(Y) fibres after heat treatment in Ar for 1 h, compared with Hi-Nicalon and Tyranno SA fibres

Tyranno SA fibre, the tensile strength is almost retained up to 1800 °C [5]. The highest observed tensile strength retention of the Tyranno SA fibre is believed to be due to its oxygen-free composition and its compact structure.

By comparing with carbon fibres, the most distinguishing feature of SiC fibres is high temperature oxidation resistance. Figure 8 shows the room tensile strength changes of the SiC(OY) and the SiC(Y) fibres after heat treatment in air at different temperatures for 1 h. The SiC(Y) fibres present good high temperature oxidation resistance. With the exposure temperature increasing in air, the room tensile strength of the SiC(Y) fibres decreases very slowly. When the temperature is up to 1500 °C, the strength retention is 83.9%. However, the SiC(OY) fibres can only stand up to 1200 °C in air. When the temperature exceeds 1200 °C, there is a sharp decrease of the tensile strength. Up to 1500 °C, the strength retention is 26.5%.



Fig. 8 Tensile strength changes of SiC(OY) and SiC(Y) fibres after heat treatment in air at different temperatures for 1 h

4 Conclusions

1) The yttrium as a sintering aid is introduced into PCS to prepare ceramic precursor PYCS. Ceramic fibres with the SiC(OY) composition are manufactured by melt-spinning PYCS, air and chemical vapour curing the resultant fibres, followed by pyrolysis at 1000 °C. Then, the SiC(OY) fibres are converted into sintered SiC(Y) fibres by heating them in argon up to 1800 °C for 1 h.

2) The chemical composition of the SiC(OY) fibres is SiC_{1.53}O_{0.22}Y_{0.005} with an amorphous structure. The composition of SiC(Y) fibres is SiC_{1.23}O_{0.05}Y_{0.005}, which is composed of a large number of β -SiC crystallites with a size of about 50 nm and a small amount of α -SiC crystalline. The yttrium in the SiC(Y) fibres exists between two SiC grains.

3) The tensile strength and fracture toughness of the SiC(OY) fibres are 2.25 GPa and 2.37 MPa \cdot m^{1/2}, respectively, and 1.61 GPa and 1.91 MPa \cdot m^{1/2}, respectively for the SiC(Y) fibres. The SiC(Y) fibres have a higher thermal stability than the SiC(OY) fibres.

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含钇聚碳硅烷制备碳化硅纤维

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摘 要:将钇元素作为烧结助剂引入到聚碳硅烷(PCS)中制备含钇 PCS。由含钇 PCS 制成 SiC(OY)和 SiC(Y) 2 种 碳化硅纤维。对 SiC(OY)纤维转变成 SiC(Y)纤维的结构与相关特性的变化进行研究。SiC(OY)纤维的化学组成为 SiC_{1.23}O_{0.05}Y_{0.005},纤维是无定型结构;SiC(Y)纤维含有大量的直径为 50 nm 的 β-SiC 晶粒和少量的 α-SiC 晶粒, 钇元素存在于 SiC 晶粒之间。SiC(OY)的拉伸强度为 2.25 GPa,断裂韧性为 2.37 MPa·m^{1/2};SiC(Y)的拉伸强度为 1.61 GPa,断裂韧性为 1.91 MPa·m^{1/2};SiC(Y)纤维比 SiC(OY)纤维具有更好的热稳定性。 关键词:聚碳硅烷;钇;碳化硅;纤维

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