Synthesis of β-SiAlON whiskers: dependence of uniform morphology upon preparation conditions

X. M. Hou[1,2], Z. Y. Yu[1], K. C. Chou*[1] and B. Z. Zhao[2]

β-SiAlON whiskers with uniform morphology were prepared using reaction sintering method under different conditions. The effect of preparing conditions on the morphology of β-SiAlON whiskers was systematically studied by SEM, XRD, TEM and HRTEM. The results showed that single crystalline β-SiAlON whiskers with uniform morphology were successfully fabricated at 1773 K for 6 h under flowing nitrogen atmosphere. The well synthesised whiskers were of several hundreds of nanometres in diameter and a few hundreds of micrometres in length. Although the morphology and its size distribution are mainly determined by the reaction temperature and holding time, they can also be tailored by controlling the reaction atmosphere. The ratio of staring materials has no significant influence on the morphology of β-SiAlON whiskers. The growth of β-SiAlON whiskers follows a vapour–solid mechanism, and the formation of the belt-like whiskers is attributed to an anisotropic growth at the early nucleation/growth stage.

Keywords: Reaction sintering, β-SiAlON whiskers, Uniform morphology, Synthesis parameters

Introduction

In recent years, there has been increasing interest in the synthesis of ceramic whiskers because of its offering of advantages such as high melting points, low densities and high moduli. It has already been reported that ceramic whiskers could improve the mechanical properties of both ceramic matrix composites and metal matrix composites.[1,2] In addition, ceramic whiskers as filters have a potential to greatly improve the performance of high temperature membrane with engineering capabilities to filter exhaust gases from coal fired power stations with the highest separation efficiency and the highest possible heat recovery owing to their chemical and thermal stability.[3–8] The use of ceramic whiskers to fabricate ceramic membrane filters is a new direction in developing high performance ceramic membranes.[9–11]

Compared to oxide whiskers investigated in the literature, nitride whiskers show higher potential application as membrane filters due to their excellent properties, such as high strength, fracture toughness, thermal shock resistance and chemical resistance.[12] Nitride whiskers possess much higher porosity (up to 70%) resulting in higher permeability in filtration than oxide whiskers.[12] Among the nitride family, SiAlONs have attracted increasing attention as an engineering ceramic material due to their many excellent mechanical properties. Since Jack's pioneering work,[13] many efforts have been put on the investigation of their fabrication and property. The most commonly known phase of SiAlONs is β-SiAlON with a chemical formula of $Si_{6-z}Al_zO_zN_{8-z}$ (z=0–4·2), and its hexagonal crystalline structure is derived from β-Si_3N_4. With a hexagonal lattice structure, β-SiAlON crystals usually develop into elongated prisms. In fact, the unique properties of β-SiAlON, such as high strength, good thermal stability and low coefficient of thermal expansion, make it an ideal candidate for high temperature ceramic membrane filters.[1,2,13,14]

Some works have been carried out to synthesise elongated β-SiAlON material by combustion synthesis, reaction sintering, etc.[15–21] Among these methods, reaction sintering of the Al, Si and SiO_2 mixture is the most efficient method to manipulate the morphology of β-SiAlON whiskers.[19,21] In our previous work, β-SiAlON (z=1·1) nanowhiskers with uniform morphology have been synthesised using reaction sintering method.[21] However, the effect of the main synthesis parameters such as temperature, partial pressure and flowrate of gas phases etc. on the morphology of β-SiAlON whiskers has not yet been systematically investigated. It still remains a vital challenge to produce β-SiAlON whiskers with uniform morphology. Considering a uniform morphology is very important for the application of β-SiAlON whiskers, it is necessary to explore systematically the effect of synthesis parameters on its morphology.

In this study, the effects of reaction temperature, holding time, reaction atmosphere and ratio of raw materials on the morphology of β-SiAlON whiskers were systematically studied. The studies on the above mentioned points will promote new and comprehensive

[1]State Key Laboratory of Advanced Metallurgy, University of Science and Technology Beijing, Beijing 100083, China
[2]School of Chemical Engineering, The University of Queensland, Brisbane, Qld 4072, Australia

*Corresponding author, email houxinmei01@126.com

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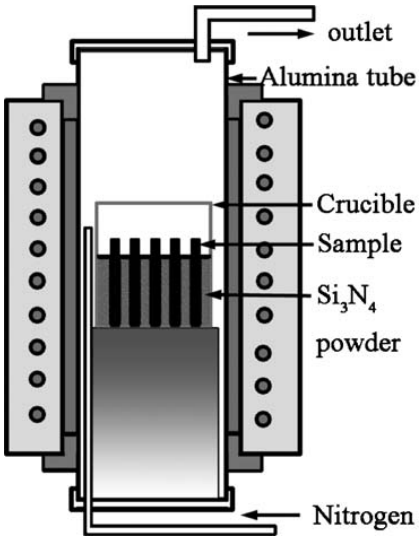
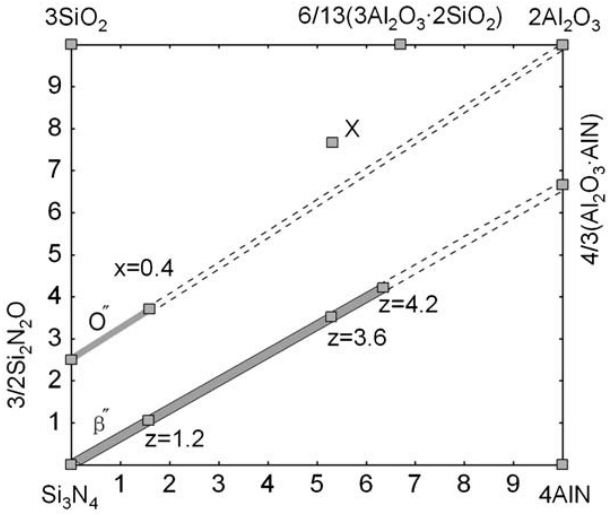


1 Schematic diagram of vertical atmosphere controlled furnace



2 Phase diagram of Si_3N_4–AlN–SiO_2–Al_2O_3 system (X, O″ and β' represent X phase, O′-SiAlON and β-SiAlON respectively)

understanding of the synthesis of uniform β-SiAlON whiskers and pave a way for its practical application as high performance ceramic membrane filters.

Experimental

Si ($\geq$99·0 mass-%, 7·4 μm), Al ($\geq$99·5 mass-%, 7·4 μm) and Al_2O_3 ($\geq$99·5 mass-%, 5 μm) were used as raw materials and mixed by mesh with stoichiometry according to the formula $Si_{6-z}Al_zO_zN_{8-z}$ ($0<z\leq4{\cdot}2$), i.e.

$$(6-z)\mathrm{Si}+\frac{z}{3}\mathrm{Al}+\frac{z}{3}\mathrm{Al_2O_3}+\frac{8-z}{2}\mathrm{N_2(g)}\rightarrow\mathrm{Si_{6-z}Al_zO_zN_{8-z}} \quad (1)$$

In the experiment, the nominal z values were set as 1·0, 1·6 and 2·5 respectively. The starting materials were milled for 10 h using absolute ethyl alcohol as the medium, and the obtained slurry was dried and sieved to <212 μm for use.

β-SiAlON whiskers were synthesised using the reaction sintering method under flowing high purity nitrogen ($\geq$99·999%) at a pressure of 0·1 MPa. The typical synthesis procedure is described as follows. The powder obtained from above experimental procedure was pressed into pellets as the substrate with a die and a hydraulic press. The samples were contained in a high purity alumina crucible covered with β-Si_3N_4 powder and placed into a vertical furnace (Fig. 1). To obtain uniform morphology, different preparation conditions were investigated:

(i) reaction temperature
(ii) holding time
(iii) reaction atmosphere
(iv) ratio of raw materials.

The reaction temperatures adopted in this work were 1723, 1773 and 1823 K respectively. The sintering time was 3, 6 and 9 h respectively. Three different conditions, i.e. nitrogen atmosphere, weak reductive atmosphere by using β-Si_3N_4 powder in flowing nitrogen and strong reductive atmosphere by using carbon in flowing nitrogen, were employed respectively during the synthesis process. The ratio of raw materials with stoichiometry according to z values of 1·0, 1·6 and 2·5 was used to obtain β-SiAlON whiskers.

The phases present in the products were identified by X-ray diffraction (XRD; M21XVHF22, MAC Science, Yokohama, Japan) using Cu K_α radiation in the angular 10–80°. The morphology of the synthesised products was investigated using thermal field emission scanning electron microscopy (FE-SEM; Zeiss SUPRA 55, Germany) and transmission electron microscopy (TEM; Hitachi H8100, Hitachi, Japan), high resolution transmission electron microscopy (HRTEM; JEM 2010, Joel Ltd Japan) operating at 200 kV.

Theoretical calculation

As is well known, Al and SiO gas are important intermediate reactants during the synthesis of β-SiAlON whiskers.[21] The concentrations of these two phases are strongly dependent on such factors as temperature, time and reaction atmosphere from both the thermodynamic and kinetic viewpoint.

Gunn[22] predicted the Gibbs free energies (J mol^{-1}) of some β-SiAlON phases based on the Calphad model as follows

$$\Delta_f G^\theta_{0{\cdot}67Si_3Al_3O_3N_5} = -1978480+575{\cdot}1T \quad (2)$$

$$\Delta_f G^\theta_{Si_4Al_2O_2N_6} = -2598080+868{\cdot}1T \quad (3)$$

From the above equations, it can be seen that β-SiAlON can be easily produced at a wide temperature range. According to the reports in the literature, the synthesis temperature of β-SiAlON is usually carried out in the temperature range of 1700–1873 K.[20] The dependence of temperature on the morphology of β-SiAlON whiskers will be discussed in the later section.

In view of the effect of atmosphere, two systems, Si–O–N and Al–O–N, were involved during the formation of β-SiAlON whiskers. According to the phase diagram of Si_3N_4–AlN–SiO_2–Al_2O_3 (Fig. 2), X-phase and O′-SiAlON exist between the phases of oxides and β-SiAlON, indicating that X-phase and O′-SiAlON may appear during β-SiAlON synthesis depending on the

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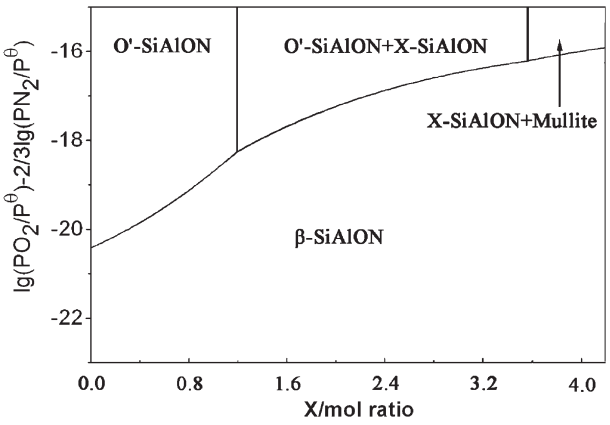


3 Stable phase region of SiAlON at 1800 K

reaction atmosphere. The transformation equations can be described as follows

$$\beta\text{-SiAlON } (0<z\leq 1{\cdot}2)+\frac{3}{2}O_2\rightleftharpoons 3O'\text{-SiAlON}+N_2 \quad (4)$$

$$6\beta\text{-SiAlON } (1{\cdot}2<z\leq 3{\cdot}6)+(7{\cdot}2+1{\cdot}5z)O_2\rightleftharpoons (27-7{\cdot}5z)O'\text{-SiAlON}+(0{\cdot}5z-0{\cdot}6)\text{X phase}+(4{\cdot}8+z)N_2 \quad (5)$$

$$18\beta-\text{SiAlON } (3{\cdot}6<z\leq 4{\cdot}2)+10{\cdot}5zO_2\rightleftharpoons(18-4z)\text{X}-\text{SiAlON}+(15z-54)3Al_2O_{3\cdot 2}SiO_2+7zN_2 \quad (6)$$

The standard Gibbs free energies (J mol^{-1}) of β-SiAlON and O′-SiAlON at 1800 K can be assessed by using the thermodynamic quasi-parabolic rules[23] as follows

$$\Delta_f G^{\Theta}_{\beta\text{-SiAlON}}\ (0<z\leq 4{\cdot}2)=7576{\cdot}7z^2-417333{\cdot}3z-231140 \quad (7)$$

$$\Delta_f G^{\Theta}_{O'-\text{SiAlON}}\ (0<x\leq 0.4)=99838.5x^2-354623.5x-428625 \quad (8)$$

Setting the ratio of Al to Si in molar, $6n(\text{Al})/n(\text{Si}+\text{Al})$ as x axis and $\log\left(P_{O_2}/P^{\theta}\right)-{}^2\!/_3\log\left(P_{N_2}/P^{\theta}\right)$ as y axis, the stable phases as a function of oxygen partial pressure at 1800 K can be calculated from equations (4)–(6), and the results are shown in Fig. 3. From Fig. 3, it can be seen that the phase of β-SiAlON exists only at extra low oxygen partial pressure.

From above thermodynamic calculation, the factors of the reaction temperature and the reaction atmosphere play an important role during the synthesis of β-SiAlON whiskers.

Results and discussion

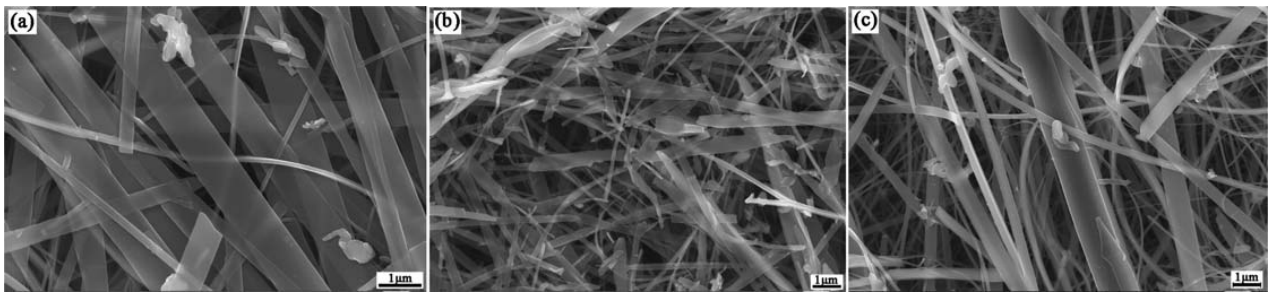
Effect of reaction atmosphere

Experiments under the following three different conditions were conducted:

(i) weak reductive atmosphere using β-Si_3N_4 powder to control the oxygen pressure in flowing high purity nitrogen
(ii) neutral atmosphere using flowing high purity nitrogen
(iii) strong reductive atmosphere using carbon powder to control the oxygen pressure in flowing high purity nitrogen.

Figure 4 shows the FE-SEM images of β-SiAlON whiskers (nominal $z=1{\cdot}6$) prepared at 1773 K for 6 h in the three reaction atmospheres. The production rate of β-SiAlON whiskers under condition (i) were the highest, and white wool-like materials covered the entire surface of the sample. Figure 4*a* showed the microstructure of β-SiAlON synthesised under condition (i) to be whiskers with uniform morphology. The diameter was ~700 nm, and the length was up to a few hundreds of micrometres. By comparison, the white wool-like materials were also formed on overall the surface of the sample under condition (ii). However the production rate of the product synthesised was a little bit less. The elongated β-SiAlON whiskers formed under this condition was also uniform as shown in Fig. 4*b*. These whiskers were of ~400 nm in diameter, while the length was much shorter than those synthesised under condition (i), which implies of being little significance for practical application. In view of the morphology of β-SiAlON whiskers synthesised under condition (iii), the distribution of the diameter of the whiskers varied quite wide from 200 to 1100 nm and the production rate was quite low.

From analysis above, β-SiAlON whiskers with uniform morphology can be synthesised under condition (i), i.e. controlling the oxygen pressure via flowing high purity nitrogen and using β-Si_3N_4 powder. From the thermodynamic calculation, it indicates that Al and SiO are important intermediate reactants during the synthesis of β-SiAlON whiskers.[21] In the experiment, β-Si_3N_4 powder was used to control the oxygen partial pressure to be $\sim 10^{-20}$ atm in the experimental temperature range, which enables high concentrations of Al and SiO



a weak reductive atmosphere; *b* neutral atmosphere; *c* strong reductive atmosphere

4 Images (FE-SEM) of β-SiAlON (nominal $z=1{\cdot}6$) whiskers synthesised at 1773 K for 6 h under different reaction atmospheres

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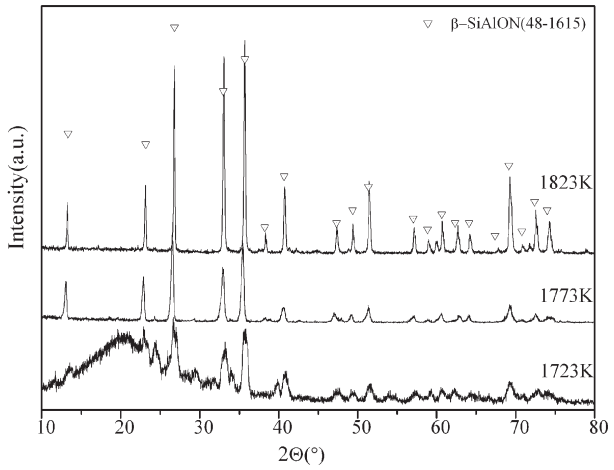


5 XRD pattern of β-SiAlON (nominal z=1·6) whiskers synthesised at different temperature for 6 h

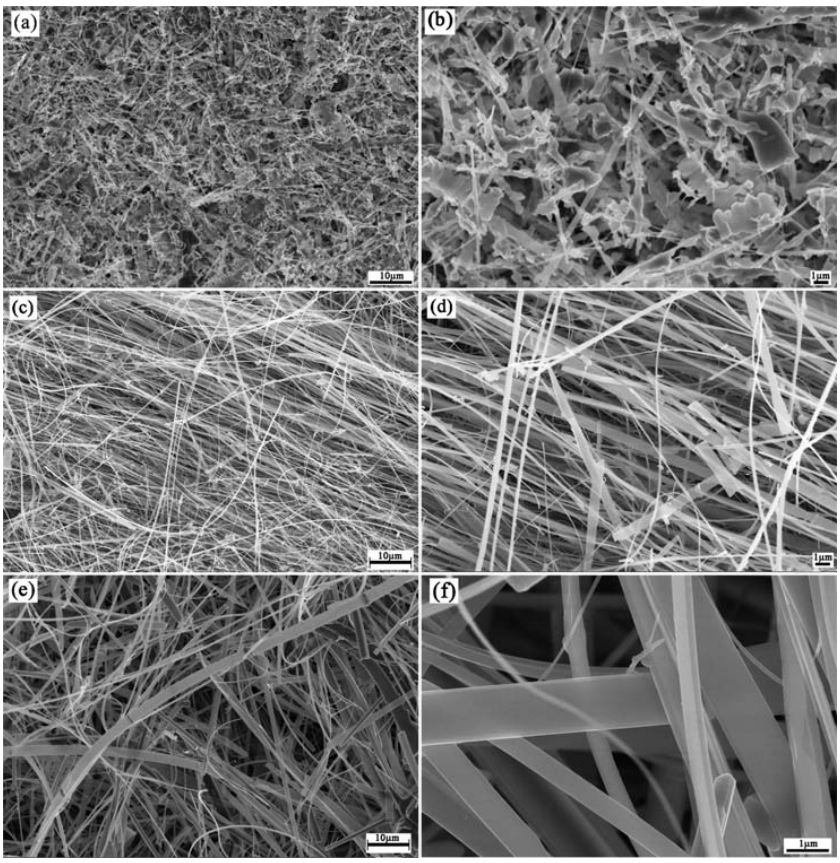
phases.[21] At the same time, Si_3N_4 powder can provide the phase of SiO during the synthesis process, which makes the phase of SiO stable.

Besides introducing β-Si_3N_4 powder in the system, the following measurements have been adopted to enable β-SiAlON whiskers grow in a stable way in the experiment by adjusting the flowrate of nitrogen, i.e. the flowrate was kept at 0·6 L min^{-1} before heating to 1773 K. It was slowed down to 0·4 L min^{-1} at 1773 K and then kept at 0·5 L min^{-1} during cooling.

Effect of reaction temperature

Figure 5 shows the XRD results of the sample prepared (nominal z=1·6) at different temperature, i.e. 1723, 1773 and 1823 K for 6 h at weak reductive atmosphere. It can be seen from the results that almost all the characteristic peaks of the sample prepared were well matched by the monophasic β-SiAlON (PDF card no. 48-1615, i.e. z=1·0). While a big lump appeared at the range of 10–25° in the sample synthesised at low temperature, i.e. 1723 K, indicating that the crystallisation of β-SiAlON whiskers synthesised at this temperature was not well. When the temperature was increased to 1773 K, the characteristic peaks became stronger and sharper, indicating that the reaction and crystallisation of β-SiAlON whiskers were improved. Further, temperature increased to 1823 K seems to improve the crystallisation of β-SiAlON.

The corresponding FE-SEM images of the β-SiAlON samples prepared at 1723, 1773 and 1823 K respectively are shown in Fig. 6. As shown in Fig. 4*a*, the morphology of β-SiAlON synthesised at 1723 K was composed of various shapes including whiskers. Higher magnification (Fig. 4*b*) revealed that the whiskers in the product prepared at this temperature were very tiny. Their diameter was relatively small. This was in agreement with the XRD results at the same temperature (Fig. 5). When the temperature was increased to 1773 K, as shown in Fig. 6*c* and *d*, tremendous amount of elongated β-SiAlON whiskers were produced, and they also grew longer to become more uniform in their morphology. The diameter was ~100 nm, and the length was extended to a few hundreds of micrometres. TEM image together with the result of the selected area electron diffraction (SAED) of β-SiAlON whiskers synthesised at this temperature is shown in Fig. 7*a*. The diffraction patterns (the lower right corner of



6 Images (FE-SEM) of β-SiAlON (nominal z=1·6) whiskers synthesised at *a*, *b* 1723, *c*, *d* 1773 and *e*, *f* 1823 K for 6 h

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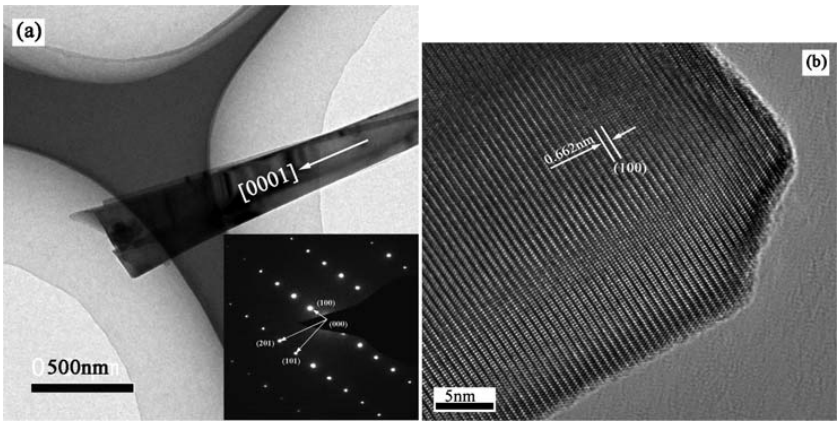


a image (TEM) with SAED pattern; *b* HRTEM image of the whisker's head

7 Results (TEM) of β-SiAlON (nominal z=1·6) whisker

Fig. 7*a*) indicated that the synthesised β-SiAlON whisker was a single crystal, and it grew along the c axis. HRTEM was also employed to analyse the structure of the single crystal, and the result is shown in Fig. 7*b*. Lattice image indicated that the plane distance of single β-SiAlON was ~0·662 nm, which was close to the $(10\bar{1}0)$ lattice spacing of β-SiAlON (0·661 nm, PDF card no. 48–1615, z=1·0). In addition, the longitudinal direction of this crystal was parallel to the [001] direction, namely, c axis from the SAED pattern. HRTEM image of the crystal clearly showed that the crystal developed perfectly, and the preferred growth direction is [001] and its edge was parallel to (100) planes, although the edges of these whiskers were not always parallel to (100) but to (110) or (210) planes sometimes.

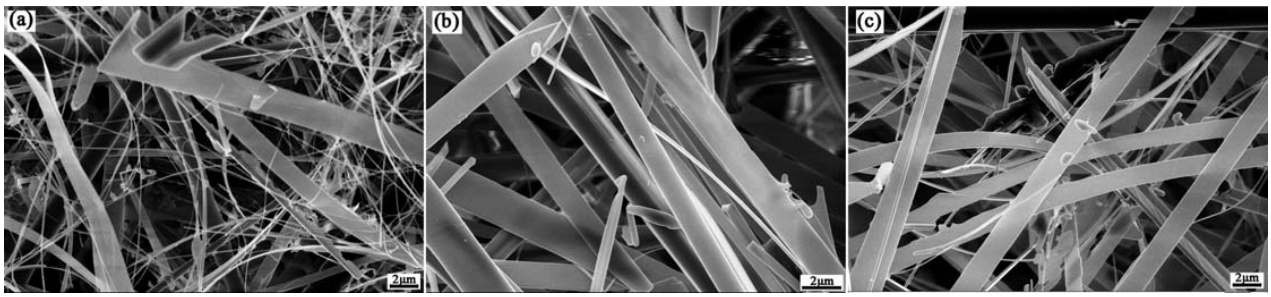
Further increasing the sintering temperature, the morphology of β-SiAlON did not change much except that the diameter increased up to 300 nm or so (shown in Fig. 6*e* and *f*). A small amount of tiny crystals which were <100 nm in diameter were also produced. The main reason was probably that the growth rate along the width direction, i.e. $(10\bar{1}0)$ plane, was much quicker initially caused by the anisothropic grain growth of β-SiAlON.[21] However, the growth rate along the other direction might occur at higher sintering temperature. This is consistent with several previous studies on the growth of β-Si_3N_4 whiskers[24] and α-Si_3N_4 nanowires.[25]

Effect of holding time

In order to determine the optimal conditions for preparing the β-SiAlON whiskers with uniform morphology, the effect of the holding time on the morphology was also investigated between 3 and 9 h at 1773 K. The experimental results showed that the production rate of β-SiAlON whiskers (nominal z=2·5) on the surfaces of the sample increased with the holding time increasing. The microstructures of the β-SiAlON whiskers synthesised at 1773 K and holding for different times are shown in Fig. 8. It can be seen that β-SiAlON whiskers with uniform morphology were obtained at 1773 K for 6 h. The diameter was ~700 nm, and the length was up to a few hundreds of micrometres (Fig. 8*b*). In addition, the SEM observation showed that the surfaces of the whiskers were smooth and clean. However, when the holding time was increased to 9 h, as shown in Fig. 6*c*, the morphology was no longer uniform and part of the surface became coarse. The main reason was probably that the growth rate along the width direction, i.e. $(10\bar{1}0)$ plane, was much quicker initially caused by the anisotropic grain growth of β-SiAlON.[12,26] At longer holding time, the growth rate along the other direction may occur due to the thermodynamic force. This is confirmed in the foregoing section, and it indicated that β-SiAlON whiskers with uniform morphology were an in-process product, appearing only under optimal preparation conditions.

Effect of ratio of raw materials

The effect of the ratio of raw material on morphology of β-SiAlON whiskers was also investigated. Figure 9 showed the FE-SEM images of the morphology of β-SiAlON with nominal z values of 1·0, 1·6 and 2·5 prepared at 1773 K for 6 h. By comparison, the morphology of synthesised β-SiAlON whiskers using different ratio of raw materials did not change significantly, indicating that the raw material ratio had little effect on the morphology of β-SiAlON whiskers.



a 3 h; *b* 6 h; *c* 9 h

8 Images (FE-SEM) of β-SiAlON (nominal z=2·5) whiskers synthesised at 1773 K for different holding times

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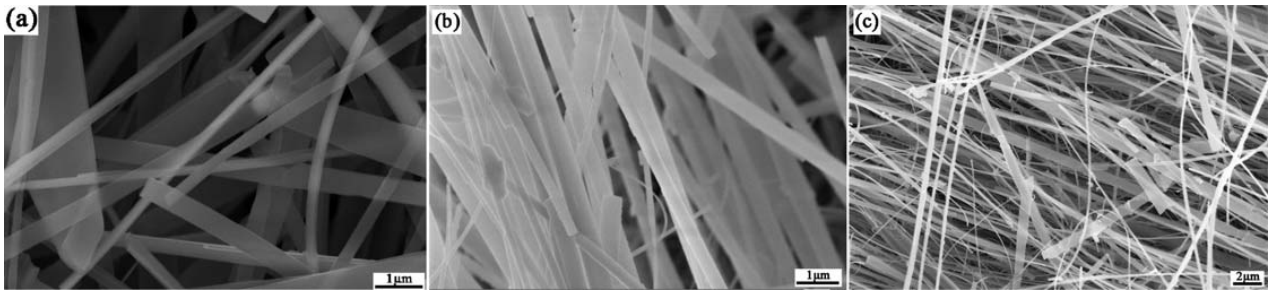

a z=1·0; *b* z=1·6; *c* z=2·5

9 Images (FE-SEM) of β-SiAlON whiskers synthesised at 1773 K for 6 h using different ratio of raw materials

Vapour–solid (VS) and vapour–liquid–solid (VLS) are two well known mechanisms for the formation of whiskers.[26] In the VLS mechanism, a liquid droplet will be initially formed, and then reactant molecules in the vapour are transported by diffusion to the liquid–solid interface, where precipitation occurs and, with crystal growth, the droplet is detached from the substrate. During the cooling stage, the liquid droplet forms a nodule at the top of the whisker, which is considered a characteristic morphology of the VLS mechanism. In the micrographs of the β-SiAlON whiskers observed in this study, no nodule was observed at the ends of those whiskers. As shown in Fig. 5, the top of the whiskers was well developed, indicating that β-SiAlON whiskers were mainly nucleated by VS mechanism.[12,25] The morphology of β-SiAlON whiskers prepared in this work appeared to be belt-like. This is attributed to an anisotropic growth at an early nucleation/growth stage. The preferred growth direction of the crystal was [001], and the growth rate along this direction was faster, which led to the formation of the belt-like β-SiAlON whiskers. This phenomenon has also been reported in the literature during the synthesis of plate-like and branched single crystalline Si_3N_4 whiskers.[24]

Conclusions

This work provides, for the first time, a systematic study of β-SiAlON whiskers with uniform morphology using reaction sintering method from both thermodynamic and experimental results. The results showed that the preparing conditions such as sintering temperature, holding time and reaction atmosphere have significant influence on the morphology of β-SiAlON whiskers. The introduction of β-Si_3N_4 powder in the reaction system cannot only help to provide SiO phase but also control the oxygen partial pressure, resulting in the formation of β-SiAlON whiskers with uniform morphology. The reaction temperature and holding time are crucial for the improvement of the morphology of β-SiAlON whiskers because the whiskers are an in-process product depending on both thermodynamic and kinetic factors. Low reaction temperature and shorter holding time result in β-SiAlON whiskers with low crystalline structure and non-uniform morphology. The ratio of raw materials has little influence on the morphology of the whiskers. According to the characteristic morphology of β-SiAlON whiskers, its growth was mainly nucleated by VS mechanism. This work sheds light on the factors that govern the growth of β-SiAlON whiskers with uniform morphology and gains access to the controlled fabrication of nitride whiskers by reaction sintering.

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