



Formation mechanism of Sialon in alumina-ferro-silicon-nitride composite under nitrogen atmosphere at high temperatures

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ABSTRACT

Sialon bonded Al_2O_3 composites were successfully synthesized using ferro-silicon nitride and different alumina sources at 1500 °C and 1600 °C under N_2 atmosphere. Fused corundum, sintered alumina and the mixture of both were used as different alumina sources to evaluate their effects on the formation of Sialon phases. The samples were characterized by X-ray diffraction (XRD), Scanning electron microscope (SEM) and Energy-dispersive X-ray spectroscopy (EDAS). The results show that the Sialons (β -Sialon ($\text{Si}_2\text{Al}_4\text{O}_4\text{N}_4$) and 15R-Sialon ($\text{SiAl}_4\text{O}_2\text{N}_4$)) contents are dependent on the sintering temperature and alumina sources. Formation mechanism of Sialon in samples prepared with different alumina sources is different. Sintered alumina can react with Si_3N_4 directly to form Sialon. In sample prepared with fused corundum, AlON is formed first and then it reacts with Si to form Sialon. Sintered alumina exhibits better reactivity than fused corundum. Sialon is more stable than AlON under N_2 atmosphere at high temperature with the existence of carbon.

1. Introduction

Alumina composites with non-oxide materials have attracted many research focuses since they can be applied to environment in which low carbon content is required. Sialon- Al_2O_3 composite, which has already been used in long-life blast furnace [1,2], is one representative composite.

Sialon has been being researched for high temperature engineering material because of its excellent chemical property and physical property, such as oxidative resistance [3] and thermal shock resistance [4–6]. Normally, Sialon was synthesized by reaction-bonded sintering [7–9] or hot isostatic pressing earlier [10,11] and sintering aids was often required. In recent years, preparing Sialon by carbothermal reduction nitridation of aluminosilicate [12–14] or combustion synthesis [15,16] has been studied a lot owing to their low fabrication costs and the production of pure Sialon.

Usually, Sialon- Al_2O_3 composites are produced by in-situ synthesis of metallic Al powders, Si powders and Al_2O_3 through carbothermal reduction nitridation. However, using fine metal powders as raw materials results in increased costs and process complexity. Ferro-silicon nitride, the composite of Fe_3Si and Si_3N_4 , is produced by the combustion synthesis of Fe–Si alloy in industry [17,18]. It has been widely used in blast furnace because of its special property. Ferro-silicon nitride is stable below 1500 °C, while Si_3N_4 decomposes rapidly at

1600 °C. So, it can be used to replace Si_3N_4 in some cases. Moreover, liquid ferro-silicon melt will play the role of plastic reinforcement in liquid phase sintering at high temperature [19]. So, in this work, ferro-silicon nitride is used as precursor for the in-situ synthesis of Sialon- Al_2O_3 composite. At the same time, the difference of alumina sources is also considered in this work. Fused corundum and sintered alumina have different properties because of their different production methods. So, their efforts on the reaction between Al_2O_3 and ferro-silicon nitride may be different.

Therefore, fused corundum, sintered alumina and the mixture of both were used as different alumina sources in the present to prepare samples that composite with ferro-silicon nitride. And samples were sintered at 1500 °C and 1600 °C due to the decomposition of ferro-silicon nitride at 1600 °C.

2. Experimental

Different alumina (fused corundum, sintered alumina and the mixture of both) powders, ferro-silicon nitride were used as raw materials, and phenolic resin was added with a concentration of 12 wt% (relative to the total weight) as a binding agent. Their relative amounts are listed in Table 1. The raw materials were mixed and then compressed to cylindrical shape under 200 MPa pressure. Samples were dried in the oven at 200 °C for 48 h and then heated in graphite vacuum furnace under N_2

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Table 1
Relative amounts of raw materials.

Raw material	Size/ μm	Mass fraction/wt.%					
		M1	M2	M3	M4	M5	M6
Fused corundum	44	100	50		46	23	
Sintered alumina	44		50	100		23	46
Ferro-silicon nitride	44				54	54	54

flow (0.1 MPa, purity of 99.99%) with a ramp speed of 5 °C/min to 1100 °C and 3 °C/min to 1500 °C and 1600 °C, respectively. The holding time was 3 h. After that, the samples were cooled naturally to room temperature.

Sintered samples were ball-milled into powders for X-ray diffraction measurements. The patterns were collected on a Smart Lab equipped with Cu K α radiation (with a scan speed of 5°/min in the continuous mode and 2θ range of 10–90°). Morphology of fracture surface of samples were observed by Scanning electron microscopy (JSM-6510A Smart Lab, Japan). Elements in specific areas were analyzed by EDAS.

3. Results and discussions

3.1. Phase composition

XRD patterns of samples sintered at 1500 °C and 1600 °C are presented in Fig. 1. The results show that different Sialon phases including

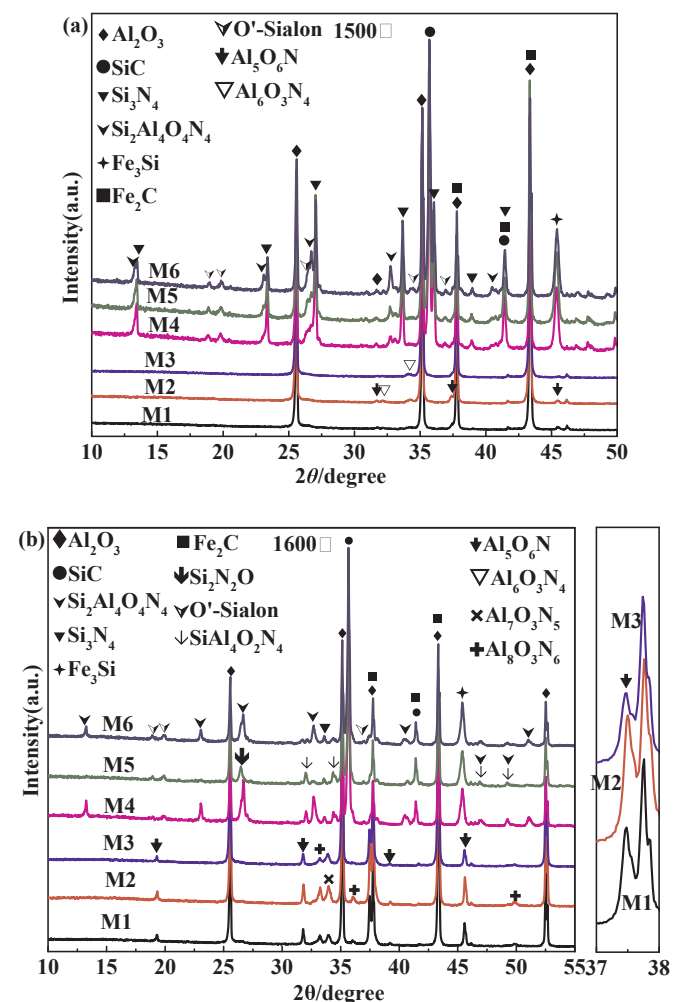


Fig. 1. XRD patterns of samples sintered at (a) 1500 °C and (b) 1600 °C.

β -Sialon ($z = 4$), 15R-Sialon and O'-Sialon, and SiC were formed in samples containing ferro-silicon nitride after the samples were sintered. While only AlON is formed in samples free of ferro-silicon nitride. To better show the differences of the AlON content in sample M1-M3 sintered at 1600 °C, enlarged X-ray diffraction patterns at 37–38° are illustrated in Fig. 1 (b). For all products, SiC and Al₂O₃ are the major phases. At 1500 °C, more Si₃N₄ phase existed in samples compared to that at 1600 °C. The contents of Sialon phases are dependent on sintering temperature and species of alumina. At 1500 °C, the sample prepared with sintered alumina (M6) possesses more β -Sialon content than the other two samples. At 1600 °C, the amount of β -Sialon in sample prepared with fused corundum (M4) increased sharply and some 15R-Sialon was formed in this sample. However, there is no obvious increase in β -Sialon content in sample M6. Specifically, no β -Sialon was formed in sample prepared with the mixture of sintered alumina and fused corundum (M5). Instead, some Si₂N₂O and 15R-Sialon were formed in this sample. There is also little O'-Sialon formed in all samples.

The differences of products in samples with and without ferro-silicon nitride suggest that Sialon is more stable than AlON under N₂ atmosphere at high temperature when carbon exists.

3.2. Morphology

To further study the formation pathway of Sialon, SEM was applied to observe the fracture surface of samples. The major focus of the observation is the differences of morphology of SiC and β -Sialon. Morphology of SiC in samples are illustrated in Fig. 2 and that of β -Sialon are illustrated in Fig. 3.

Most ferro-silicon alloy particles are observed bonded together with SiC in all samples, as shown in Fig. 2 (a) and (b), bright white areas represent ferro-silicon alloy. Specifically, some unreacted Si₃N₄ is found next to these areas in samples sintered at 1500 °C, which suggests that in the conversion of Si₃N₄ to SiC, Fe–Si alloy that bonded together with SiC may be an intermediate phase.

Morphology of β -Sialon is found to be strongly dependent on the species of alumina, as shown in Fig. 3. In sample M4 sintered at 1600 °C, which is prepared with fused corundum, the β -Sialon is found in regular hexagonal prism shape and it is mainly found on the surface of fused corundum particles, as shown in Fig. 3 (b). While morphology of most β -Sialon is found in irregular shape in sample M6, which is prepared with sintered alumina, as shown in Fig. 3 (a) and (d). XRD results show that there is no β -Sialon formed in sample M5 sintered at 1600 °C, which is prepared with the mixture of both alumina sources, while Si₂N₂O and 15R-Sialon were formed in this sample. Amorphous Si₂N₂O is observed at the fracture surface of this sample, as shown in Fig. 3 (c). The element ration of point A, B and C in Fig. 3 (b) and (c) is shown in Table 2, which suggests that phases in A, B and C are β -Sialon, 15R-Sialon and Si₂N₂O, respectively.

Si–Al–O–N ratio of different Sialon has been studied previously, and phase diagram of Si₃N₄–AlN–SiO₂–Al₂O₃ is presented in Fig. 4 [20]. In the Si₃N₄–AlN–SiO₂–Al₂O₃ system, the proportion of Al–N in 15R-Sialon is higher than β -Sialon, which suggests that β -Sialon may be

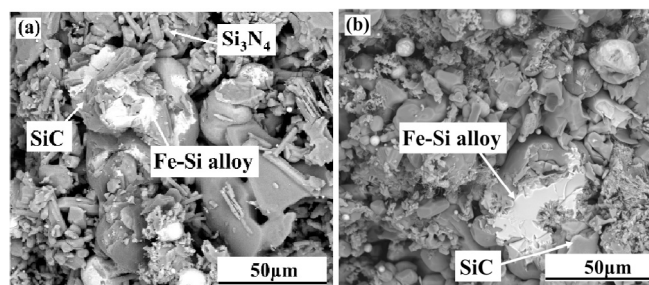


Fig. 2. SEM images of sample M5 sintered at (a) 1500 °C and (b) 1600 °C.

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