Preparation and Characterization of SiC/Al₂O₃ Composites

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Abstract. The present research was focus on various SiC content adding to Al₂O₃ matrix to improve the mechanical properties of Al₂O₃ ceramic. The mechanical properties of the composites were characterized by XRD and SEM. The effect of SiC content on the mechanical properties of SiC/Al₂O₃ composites was studied. The results show that SiC/Al₂O₃ composites have the best comprehensive mechanical properties when the SiC content is 5vol.%. With the increase of SiC content, agglomeration phenomenon was obvious, pores at grain boundaries increased, fracture mode changed from transgranular fracture to intergranular fracture, and mechanical properties of the material got worse.

1. Introduction
With the development of modern industry and high and new technology, single component materials have been difficult to meet this situation. Ceramic composites have become one of the hot spots in material research for its high toughness, high strength, high hardness, high temperature resistance, corrosion resistance and other properties[1-3]. Aluminium oxide (Al₂O₃) has been widely used in machine manufacturing, medical treatment, automobile and other domains owing to its high hardness, high chemical stability, corrosion resistance (acid and alkali corrosion), good thermal conductivity, high insulation strength, high resistivity, wear resistance and a series of outstanding performance[4-6]. However, the remarkable defect, high brittleness is the greatest problem that limit the application of Al₂O₃ ceramic. Therefore it is significative to search an effective method to enhance the strength and toughness of Al₂O₃[7-9]. In recent decades researchers have tried various materials such as metal, ceramic, fibers to intensify Al₂O₃ ceramic[10]. Various volume content of SiC has been used in the study of Y.K. Jeong to enhance Al₂O₃, illustrating that SiC was an excellent sintering aids and flexibilizer to form Al₂O₃[11]. The research exhibited that the composite acquire the best mechanical properties of bending strength beyond 650 MPa and fracture toughness 4.8 MPa·m¹/² when the SiC volume content at 5%. T. Sekino pointed that carbon nano tube was an ideal material to toughen Al₂O₃ ceramic that cause the fracture toughness of the composite exceed twice than that of the onefold Al₂O₃[12].

In the present work, nano SiC particles with different volume content were employed as the sintering aids and flexibilizer to synthesize Al₂O₃ via vacuum heating-press sintering. At the end thermal etching experiment has been adopted to evaluate the heat resisting capacity of the SiC/Al₂O₃ composite.

2. Experimental Procedure
The raw materials, Al₂O₃ (99.9%, average size 1 μm, Shanghai ST-Nano Science and Technology, China) and SiC (99.9%, 50 nm, Shanghai ST-Nano Science and Technology, China), were prepared in
the experiment with five components which were exhibited in table 1. The microstructure of the raw materials has been exhibited in figure 1.

|   | 1  | 2  | 3  | 4  | 5  |
|---|----|----|----|----|----|
| Al₂O₃(vol.%) | 95 | 90 | 85 | 80 | 75 |
| SiC(vol.%)   | 5  | 10 | 15 | 20 | 25 |

Figure 1. Microstructure of the raw materials

The mixing powders were stirred by the dispersion medium absolute ethyl alcohol and abrasive medium corundum balls. To obtain the preferable dispersion effect, the mass ratio of the powders, mill balls, and ethyl alcohol was kept as 1:2:1. Then the sizing agents were put into resin ball mill pot for grinding via a planetary with the ball mill process of rotate speed 200 r/min, grinding time 360 min. After milling the slurry was dried at a vacuum drying oven at 60°C until the liquid phase exhaust. The desiccative powders will be filled into a columniform graphite die with the diameter of 45 mm. Finally the graphite was put into a vacuum hot pressing furnace for fabricating the block composite accompanying the sintering temperature of 1600°C, sintering pressure 30 MPa, soaking time 90 min.

The block composite will be cut into several strips with the shape size of 36×4×3 mm in order to measure the bending strength. Besides, few specimens have been processed as the shape size of 36–40×2×4 mm with a groove of 0.2 mm in breadth, 0.5–1 mm in altitude for characterizing the fracture toughness of the composite. Hardness was calculated with the size of indentation by a Vickers. The phase transformation and microstructure of the composite have been investigated by XRD and SEM tests respectively. The relative density was measured based on Archimedes method.

3. Results and Discussion

3.1. Phase Analysis

XRD image of SiC/Al₂O₃ composites has been exhibited in figure 2. There were no new phases appeared in the sintered samples. The peaks of SiC became higher from sample 1 to 5 that can be ascribed to the increase of SiC volume content, which was corresponding with the component of these samples. All peaks of Al₂O₃ and SiC were evident and poignant, which indicated the favourable crystallinity of the composites.
3.2. Morphology Analysis

Figure 3. exhibited the microstructure of the fracture surface of 5 vol.% SiC/Al2O3 detected by a scanning electron microscope. Nano SiC had not grew singularly and dispersed uniformly in the interface and surface of Al2O3 ceramics, which could act as a nail binding to restrict the growth of Al2O3 particles that caused the composite excellent mechanical properties. Few pores could be found in the fracture surface. In addition, there were partial Al2O3 particles presented transcrystalline fracture that were signed in Figure 3.(1). The reason for this phenomenon may be that the SiC particles distributed in the grain boundary of Al2O3 restrict the volume of Al2O3, making it difficult for the matrix to be destroyed along the grain boundary. Therefore, the addition of nano SiC would play a positive role in strengthening and toughening Al2O3 ceramics.

The microstructure of fracture section of 10 vol.% SiC/Al2O3 had been showed in figure 3. It could be seen more pores in sample 2 than that of sample 1, and the combination of matrix particles was loose. Additionally, the number of grains breaking through the grain also decreased significantly comparing to sample 1. According to Kunadson's formula:

$$\sigma = \sigma_\infty \exp(-bP) \quad (1)$$
Where $\sigma$ was actual strength, $\sigma^\infty$ was theoretical strength, $b$ was coefficient, $P$ was porosity. The strength of materials and porosity showed an exponential change, therefore smaller changes of porosity could also cause bigger changes of strength. The mechanical strength of sample 2 was poorer than that of sample 1 theoretically.

**Figure 4.** SEM images of 10 vol.%SiC/Al$_2$O$_3$ composites

**Figure 5.** SEM images of 15 vol.%SiC/Al$_2$O$_3$ composites

**Figure 6.** SEM images of 20 vol.%SiC/Al$_2$O$_3$ composites
Figure 7. SEM images of 25 vol.%SiC/Al₂O₃ composites

Figure 5-7. illustrated the fracture surfaces of 15, 20, 25vol.%SiC/Al₂O₃ (sample 3-5) multiphase ceramics, respectively. It was certain that nano SiC particles had been aggregated due to the more addition which was found in the SEM images. The Al₂O₃ had started to grow singularly without of the limit of nano SiC when the volume content of SiC exceeded 15%. Meanwhile, the fracture mechanisms of matrix had transformed from transgranular fracture to intergranular fracture. Furthermore, it was clearly found that the sintered Al₂O₃ grain particles had grown singularly showed in figure 6-7. The fracture surface of sample 4-5 also tended to be smooth than that of sample 1-2. The agglomerate SiC granules could be seen obviously inseting between the Al₂O₃ particles inhomogeneously, which would damage the mechanical property of the composite. As a result, the mechanical property of the composite was predicted gradually decrease opposite to the nano SiC addition. It would be confirmed in the following discussion on mechanical properties.

3.3. Mechanical Property

The mechanical properties of sample 1-5 had been tested and calculated and the results were listed in table 2.

### Table 2. Mechanical properties of sample 1-5

| Sample | Relative density/% | Bending strength/MPa | Fracture toughness/MPa·m⁻¹/² | Vickers hardness/HV |
|--------|--------------------|----------------------|------------------------------|---------------------|
| 1      | 99.47              | 507.82               | 4.75                         | 1824.96             |
| 2      | 99.09              | 419.81               | 4.50                         | 2019.11             |
| 3      | 98.69              | 330.66               | 4.34                         | 1693.23             |
| 4      | 97.78              | 323.27               | 4.12                         | 1394.74             |
| 5      | 97.34              | 284.46               | 4.01                         | 1273.23             |

The samples sintered via vacuum hotpressing showed very high relative density beyond 97%. The change tendency of relative density was on the contrary to the increase of SiC volume content. Sample 1, 5vol.% SiC/Al₂O₃ composite was endowed the best relative density, which was consistent with the most compact structure by analysing SEM images. Based on Kunadson's formula, the samples with lesser pores would get the higher bending strength. Results of three point test indicated the best bending strength and fracture toughness were 507.82 MPa and 4.75 MPa·m⁻¹/² at 5vol.% SiC/Al₂O₃ sample, respectively, which was ascribed to the symmetrical distribution of nano SiC in the matrix and its pinning effect. Nanomaterials have high activity, which makes it difficult to disperse them evenly. Therefore, when the nano SiC content increased, homogeneity of the composite might become worse, so the mechanical properties of sample 2-5 decreased gradually, including the Vickers hardness.
Structure dependenced performance, the loose particle binding had caused the Vickers hardness decreased obviously in corresponding with the relative density of sample 1-5. In short, the mechanical properties of the composites getted the best values at 5 vol.%SiC/Al₂O₃ composites, and then decreased with the more nano SiC addition.

4. Conclusions
SiC/Al₂O₃ composites were successfully fabricated via vacuum hot pressing sintering. According to the XRD analysis and SEM images of sample 1-5, as well as the mechanical properties analysis, the following conclusions were obtained:

(1) XRD analysis showed that no new phases appeared in the sintered samples.

(2) The results of three point test indicated that when SiC content was 5 vol.%, the mechanical properties of the composite reached the optimal by the best bending strength and fracture toughness of 507.82 MPa and 4.75 MPa·m¹/², respectively. While the density reached to 99.47% and vickers hardness of 1824.96 HV. With the increase of SiC content, agglomeration phenomenon was obvious, pores at grain boundaries increased, fracture mode changed from transgranular fracture to intergranular fracture, and mechanical properties of the material got worse.

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6. References
[1] T. Nagai, H.J. Hwang, M. Yasuoka, M. Sando and K. Niihara 1998 J. Am. Ceram. Soc. 81 425
[2] X.M. Chen and B. Yang 1997 Materials Letters. 33 237
[3] B. Yang and X.M. Chen 2000 J. Eur. Ceram. Sci. 20 1687
[4] D. Chakravarty, S. Bysakh, K. Muraleedharan and T.N. Rao 2008 J. Am. Ceram. Soc. 91 203
[5] M.L. Cheng, H.L. Liu, B. Zhao, C.Z. Huang, P. Yao and B. Wang 2017 Ceram. Int. 43 13869
[6] Z.B. Yin, C.Z. Huang, J.T. Yuan, B. Zou, H.L. Liu and H.T. Zhu 2015 Ceram. Int. 41 7059
[7] Shengfang Shi, Sunghun Cho, Tomoyo Goto, Takafumi Kusunose and Tohru Sekino 2018 Ceram. Int. 44 18382
[8] Mingxian Yu, Jingxian Zhang, Xiaoguang Li, Hanqin Liang, He Zhong, Yinsheng Li, Yusen Duan, Dongliang Jiang, Xuejian Liu and Zhengren Huang 2015 Ceram. Int. 41 14845
[9] K. Poser, K.H. Zum Gahr and J. Schneider 2005 Wear 259 529
[10] Liu Zhang, Zhi Wang, Junyan Wu, Guopu Shi and Hanqing Xu 2017 Ceram. Int. 43 2143
[11] Y.K. Jeong and K. Niihara 1997 Nanostruct. Mater. 9 193
[12] T. Sekino and K. Niihara 1995 Nanostruct. Mater. 6 663