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Effects of sintering additives on mechanical properties and microstructure of Si_3N_4 ceramics by microwave sintering

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ABSTRACT

Si_3N_4 ceramics with excellent mechanical properties and microstructure were fabricated by microwave sintering. The effects of sintering additives on the densification, phase transformation and grain growth of Si_3N_4 ceramics were investigated. The results showed that Y_2O_3 can promote the phase transformation, MgO can decrease the sintering temperature, Al_2O_3 can increase the Vickers hardness of the samples and the combination of Y_2O_3 and MgO was more effective in enhancing the densification and phase transformation in the microwave sintering. The samples with 5 wt%Y_2O_3, 5 wt%MgO and 2 wt%Al_2O_3 sintering additives had the optimum mechanical properties when sintered at 1700 °C for 10 min. Its relative density, fracture toughness and Vickers hardness were 98.52 ± 0.13%, 6.44 ± 0.02 MPa $m^{1/2}$ and 14.92 ± 0.20 GPa, respectively. Compared to other microwave sintered Si_3N_4, the hardness and fracture toughness were enhanced by 6.6–24.3% and 5.6–11%, respectively, while the sintering holding time was reduced by 33.3–83.3%.

1. Introduction

Silicon nitride is considered as one of the promising ceramic materials which has excellent high temperature mechanical properties, wear resistance and thermal properties and has been widely used in high-temperature gas filter, heat insulators, cutting tools, etc. [1].

Nowadays, the main conventional sintering techniques [2–6] of fabricating silicon nitride ceramics are pressureless sintering, reaction bonding sintering, gas pressure sintering, hot pressing sintering and hot isostatic pressing sintering, etc. For these conventional sintering, high sintering temperature, slow heating rate and long holding time are usually needed in order to fully dense the materials. The long sintering period is not cost effective and limits the wide use of the ceramic materials. With the development of science technology, some new sintering techniques are developed which include spark plasma sintering [7], microwave sintering [8], etc. The new sintering techniques not only improve the mechanical properties of ceramics but also greatly shorten the sintering period, while spark plasma sintering assisted with mechanical pressure which is similar with hot pressing sintering can only fabricate products with simple shape, moreover its equipment is very expensive and single furnace has low production. Microwave sintering is a potential way of promoting industrialization production of ceramic materials.

In recent years, microwave sintering of materials have attracted the attention of many researchers due to its advantages over conventional sintering techniques in fabricating of silicon nitride ceramics, such as enhancing densification and phase transformation, reducing sintering temperature and holding time, improving mechanical properties and capability of producing unique microstructure. There have been several studies on preparation of silicon nitride ceramics which make comparisons between microwave sintering and conventional sintering using the same sintering schedule. Yoon Chang Kim et al. [9] made comparisons between microwave sintering and conventional pressureless sintering. It was observed that the relatively density of microwave sintered specimens reached more than 99% and phase transformation rate reached about 100% at the sintering temperature of 1600 °C, while in the case of conventional pressureless sintering, such high density and phase transformation rate could not be obtained even at a sintering temperature of 1850 °C. Jones et al. [10,11] sintered silicon nitride ceramic by 28 GHz microwave sintering at the sintering temperature from 1200 °C to 1750 °C. It showed that the temperature of densification and phase transformation sintered by microwave energy would be 200 °C lower than the conventional pressureless sintering. Chockalingam et al. [12] investigated the mechanical properties of

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Table 1
The characteristics of the raw powders of silicon nitride ceramic materials.

Powder	Grain size (μm)	Purity (%)	Manufacturer
α-Si_3N_4	0.7	99.9	Shanghai
Al_2O_3	0.5	99.9	Shanghai
MgO	1	99.9	Shanghai
Y_2O_3	1	99.9	Shanghai

Table 2
The composition ratios of three series silicon nitride ceramic materials (wt%).

Symbol	Si_3N_4	Y_2O_3	MgO	Al_2O_3
SYM	90	5	5	/
SMA	90	/	5	5
SYA	90	5	/	5

2.45 GHz microwave sintered Si_3N_4 ceramics. They reported that microwave sintered samples exhibited higher relative density and hardness compared with the conventional sintered samples which performed in Centorr Industry's tungsten heating element furnace, its hardness was enhanced by about 40% and its holding time was reduced by 75%. Hirota et al. [13] reported microwave sintering can promote the production of columnar grain and bimodal microstructure compared with the conventional pressure sintering. From these reports, it can be found that microwave sintering is a potential way of fabricating Si_3N_4 ceramics with high properties.

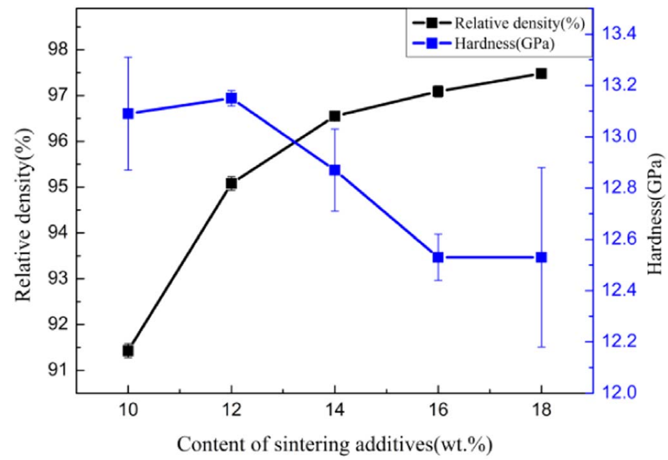

Fig. 2. The relative densities and hardness of samples with 5 wt%–13 wt% Y_2O_3 and 5 wt% Al_2O_3 as additives microwave sintered at 1700 ℃ for 10 min.

Si_3N_4 is difficult to densify by solid state sintering for its high degree of covalent bonding, thus liquid-phase sintering with suitable additives is a preferred method of promoting densification. In the conventional sintering, various metal oxides, lanthanide oxides and rare earth oxides were used as sintering additives [14–17], and kinds of conventional sintered silicon nitride ceramics with high relative densities and superior mechanical properties had been fabricated using the compositions of two or three sintering additives [18–20]. Especially

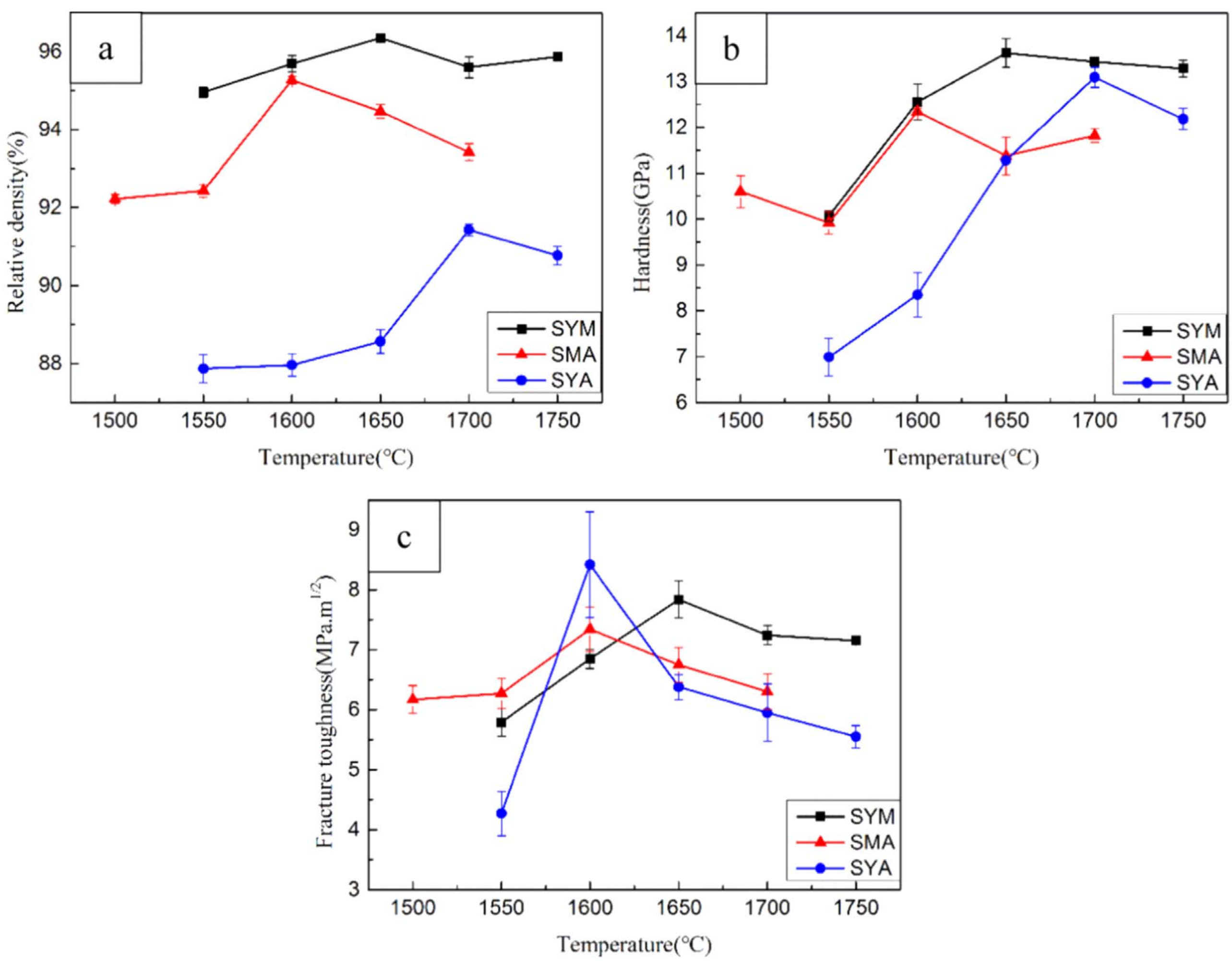

Fig. 1. (a) relative densities, (b) hardness and (c) fracture toughness of SYM, SMA and SYA sintered at different sintering temperatures.