

Study on in-situ reaction synthesis and mechanical properties of $\text{Si}_2\text{N}_2\text{O}$ ceramic

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Abstract

$\text{Si}_2\text{N}_2\text{O}$ ceramics were fabricated using an in-situ reaction synthesis method by nitriding powder mixture of Si and SiO_2 . The reaction mechanism in the Si– SiO_2 – N_2 system was discussed by analyzing the variation of Gibbs free energy with temperature. As the sintering temperature rises from 1450 to 1700 °C, the $\text{Si}_2\text{N}_2\text{O}$ phase increases with the Si_3N_4 phase decreasing and there is only $\text{Si}_2\text{N}_2\text{O}$ with hardly any Si_3N_4 after sintering at 1700 °C, the volume shrinkage and mean pore size decrease respectively from 26.1% to 18.0% and from 0.4 to 0.04 μm , the porosity increases from 20.8% to 29.1%, and the Vickers hardness decreases from 5.6 to 4.5 GPa. After sintering at 1600 °C, $\text{Si}_2\text{N}_2\text{O}$ ceramic possesses the highest flexural strength of 229 MPa and fracture toughness of 2.3 $\text{MPa m}^{1/2}$. © 2012 Elsevier Ltd and Techna Group S.r.l. All rights reserved.

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1. Introduction

Silicon oxynitride ($\text{Si}_2\text{N}_2\text{O}$) is the only compound in the SiO_2 – Si_3N_4 system [1]. It has many good properties, such as good thermal shock resistance, high thermodynamic stability temperature about 1800 °C, excellent oxidation resistance in air up to 1600 °C and high flexural strength up to 1400 °C [2–9]. A thermodynamic calculation of the stability of $\text{Si}_2\text{N}_2\text{O}$ indicates $\text{Si}_2\text{N}_2\text{O}$ is more stable than Si_3N_4 , so $\text{Si}_2\text{N}_2\text{O}$ is a promising high-temperature structural material in place of Si_3N_4 in many fields. $\text{Si}_2\text{N}_2\text{O}$ ceramics are usually fabricated by the in-situ reaction synthesis method, which is performed either through liquid phase by sintering an equimolar Si_3N_4 and SiO_2 mixture [9,10] or through gas phase by nitriding a mixture of Si and SiO_2 [11–13]. $\text{Si}_2\text{N}_2\text{O}$ has a low diffusion coefficient and strongly covalent bond, requires high sintering temperature [14], so $\text{Si}_2\text{N}_2\text{O}$ is difficult to be synthesized by reaction sintering an equimolar Si_3N_4 and SiO_2 mixture without the help of transition phase, and oxide additives are usually used to form a liquid phase with a eutectic melting

point low enough to permit sintering without excessive dissociation [2,6–8,14]. When $\text{Si}_2\text{N}_2\text{O}$ ceramic is fabricated by sintering equimolar Si_3N_4 and SiO_2 mixture by hot isostatic pressing method without adding any sintering aid, the maximum fraction of $\text{Si}_2\text{N}_2\text{O}$ is 91.1% though the sintering temperature is high as 1900 °C. When sintering aid such as Li_2O , ZrO, Al_2O_3 , MgO, Ln_2O_3 (where Ln = Yb, Er, Ce, Nb, Gd, Dy) is added, almost all Si_3N_4 and SiO_2 react with each other to produce $\text{Si}_2\text{N}_2\text{O}$, and $\text{Si}_2\text{N}_2\text{O}$ ceramics possess better mechanical properties [2,6–9,14–17]. When $\text{Si}_2\text{N}_2\text{O}$ ceramics are fabricated by nitriding a mixture of Si and SiO_2 , the strong exothermic reaction propagates spontaneously through the reactant mixture, converting it into product [18], but the incomplete reaction of Si or SiO_2 decreases the properties of $\text{Si}_2\text{N}_2\text{O}$ ceramic.

$\text{Si}_2\text{N}_2\text{O}$ ceramics have been considered to be a high-temperature functional engineering material with a variety of potential applications, such as high-temperature electric insulator, nuclear-reactor moderator or reflector and solid electrolyte [11]. The functional applications of $\text{Si}_2\text{N}_2\text{O}$ ceramics are seriously influenced by other phases, especially for Si, crystal SiO_2 and oxide additives, etc. When $\text{Si}_2\text{N}_2\text{O}$ ceramics are fabricated by sintering a mixture of Si_3N_4 and SiO_2 , the

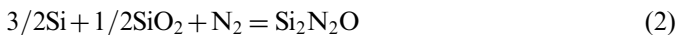
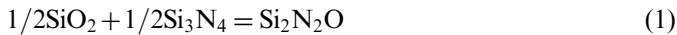
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sintering aids acting as impurities disturb the material system inevitably. Fabricating $\text{Si}_2\text{N}_2\text{O}$ ceramics by nitriding a powder mixture of Si and SiO_2 is more suitable for engineering applications due to its low-cost, but decreasing the residual Si or SiO_2 in $\text{Si}_2\text{N}_2\text{O}$ ceramics is an urgent problem needing to be solved.

In the present work, $\text{Si}_2\text{N}_2\text{O}$ ceramic is synthesized using a low-cost in-situ reaction synthesis method by nitriding powder mixture of Si and SiO_2 . With the aim to decrease the residual Si and SiO_2 in $\text{Si}_2\text{N}_2\text{O}$ ceramic, the reaction mechanism of possible reactions in the Si– SiO_2 – N_2 system is discussed by analyzing the variation of Gibbs free energy with temperature. Using raw materials with very small particle size and defining suitable sintering temperature range according to thermodynamics analysis results, the effects of sintering temperature on the phase composition, porosity, pore size distribution, microstructure and mechanical properties of $\text{Si}_2\text{N}_2\text{O}$ ceramic are investigated.

2. Thermodynamic considerations

Fig. 1 shows the variation of Gibbs free energy (ΔG) of possible reactions in the Si– SiO_2 – N_2 system as functions of temperature. The ΔG given in Fig. 1 is calculated using the Factsage Thermodynamics Software (Thermfact/CRCT and GTT-Technologies, Canada and Germany). Two reaction synthesis methods are commonly used for fabricating $\text{Si}_2\text{N}_2\text{O}$ according to following equations:



As shown in Fig. 1, as the temperature rises from 1200 to 1800 °C, $\Delta G_T^0(1)$ decreases slightly from -78.3 to -89.1 kJ/mol, while $\Delta G_T^0(2)$ increases significantly from -207.3 to -104.8 kJ/mol. Therefore, the suitable synthesis temperature of Eq.(1) depends on the eutectic melting point, while lower temperature is good for Eq.(2) to take place theoretically without considering the kinetic factors. If $\text{Si}_2\text{N}_2\text{O}$ is synthesized from Eq.(2) at lower temperature, lots of Si in the raw material could be nitrided into Si_3N_4 according to the following equation:



As shown in Fig. 1, $\Delta G_T^0(3)$ increases quickly from -258.0 to -31.3 kJ/mol as the temperature rises from 1200 to 1800 °C. At temperature below 1450 °C $\Delta G_T^0(3)$ is lower than $\Delta G_T^0(2)$, which means Si is preferred to be nitrided into Si_3N_4 . As the temperature rises to above 1450 °C, $\Delta G_T^0(3)$ becomes higher than $\Delta G_T^0(2)$, now the Si in the raw material reacts with SiO_2 and N_2 to produce $\text{Si}_2\text{N}_2\text{O}$. According to the above analysis, the sintering temperature should be higher than 1450 °C when fabricating $\text{Si}_2\text{N}_2\text{O}$ ceramic from Eq.(2).

The melting point of Si is 1412 °C. If $\text{Si}_2\text{N}_2\text{O}$ ceramic is fabricated from Eq.(2) directly at temperature above 1412 °C, the green body inevitably deforms due to the

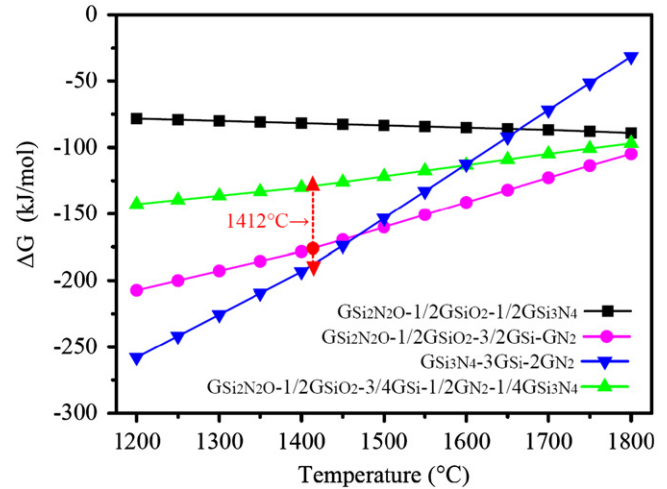
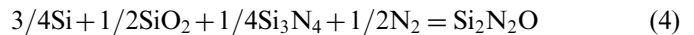


Fig. 1. Variation of Gibbs free energy of possible reactions in the Si– SiO_2 – N_2 system as functions of temperature.

flow of liquid Si. For holding the shape well, the green body should be pre-nitrided at temperature below the melting point of Si for a short time. In order to fabricate pure $\text{Si}_2\text{N}_2\text{O}$ ceramic, the best way for removing the produced Si_3N_4 is to make it react with SiO_2 to produce $\text{Si}_2\text{N}_2\text{O}$ from Eq.(1). As is known, it is difficult for Si_3N_4 react with SiO_2 without the help of oxide additive which is in liquid phase at high temperature. However, when the green body is sintered at temperature above the melting point of Si, it is possible for Si_3N_4 to react with SiO_2 with the catalysis action of liquid Si, and the potential reaction is as follows:



As shown in Fig. 1, $\Delta G_T^0(4)$ increases from -142.8 to -97.0 kJ/mol as the temperature rises from 1200 to 1800 °C, and $\Delta G_T^0(4)$ is lower than $\Delta G_T^0(1)$, so it is feasible for Si_3N_4 react with SiO_2 from Eq.(4).

3. Experimental

3.1. Raw materials

Si powder was made by ball-milling the Si powder with particle size of 45 μm for 20 h using agate balls in an agate barrel. Observed by SEM, the mean particle size of Si powder after ball-milling is about 1.0 μm . Aerosil 200, an amorphous high-purity SiO_2 powder with very high specific area of 200 ± 25 m^2/g and mean particle size of 50 nm, is the initial powder used in the present work.

3.2. Fabrication process

According to Eq.(2), the Si powder was mixed with SiO_2 powder with a mole ratio of 3:1. The powder mixture was wet-milled in distilled water for 24 h, and then the powder slurry was freeze-dried and sieved through a 50 mesh screen. Green body with dimension of 5 mm \times 10 mm \times 70 mm

was fabricated by cold-pressing the powder mixture under 100 MPa. Finally, the green body was placed in an alumina crucible and sintered in 0.1 MPa fluid nitrogen gas by following heating and cooling steps: (a) increase the temperature to 1200 °C at a temperature rising rate of 5 °C/min, and then to 1400 °C at a temperature rising rate of 0.2 °C/min, (b) increase the temperature to 1400, 1450, 1500, 1550, 1600, 1650 and 1700 °C in order with the temperature rising rate of 5 °C/min and then hold for 2 h, (c) decrease the temperature to room-temperature at a cooling rate of 5 °C/min.

3.3. Characterization and tests

The open porosity and bulk density were measured by the Archimedes method. Phase analysis was conducted by X-ray diffraction (XRD), via a computer-controlled diffractometer (XRD, X'Pert Pro, Philips, the Netherlands). Microstructure was observed by scanning electron microscopy (SEM, JSM-6360LV, Electronics Co., Ltd., Japan). The pore size distribution was measured by a Mercury Porosimeter (Poremaster 33, Quantan Ltd, USA). The flexural strength was measured via the three-point bending test (CMT4204, Jiehu Instrument Co., Ltd. Shanghai, China) with a support distance of 30 mm and a loading speed of 0.5 mm/min. Five specimens with a dimension of 3 mm × 4 mm × 36 mm were tested to obtain the average strength. The fracture toughness was measured by the single edge notched beam (SENB) method. The edge notch with 2.5 mm length was made by a diamond cutting wheel and then finished with a razor blade and diamond paste. Five specimens with a dimension of 2.5 × 5 × 30 mm³ were tested to obtain the average value. The Vickers hardness (Hv) was measured using a digital hardness tester (HBV-30 A, Huayin Experimental Apparatus Co, Shandong, China) with a pyramidal Vickers indenter. The specimens were indented with loads of 10 kg for 15 s and an average of eight indents was analyzed. Before mechanical property testing, all specimens were polished with 0.5 μm diamond paste as a final polishing step, then ultrasonically cleaned in acetone and air dried.

4. Results and discussion

4.1. Phase composition

The XRD pattern of Si₂N₂O ceramics sintered at different temperatures (Si₂N₂O-*n*, *n* means sintering temperature) is shown in Fig. 2. Besides unreacted Si, there are masses of α- and β-Si₃N₄ with little Si₂N₂O in Si₂N₂O-1400. Because of the restraining effect of nitrogen [19,20] and Si₃N₄ [21,22], most of SiO₂ in Si₂N₂O-1400 remains amorphous with little crystallized. Si₂N₂O-1450 possesses a primary phase of Si₂N₂O and secondary phase of α- and β-Si₃N₄. As is known, amorphous SiO₂ starts to crystallize when the temperature reaches to 1300 °C, and the nucleation of cristobalite is heterogeneous which starts from the surface of SiO₂ particles

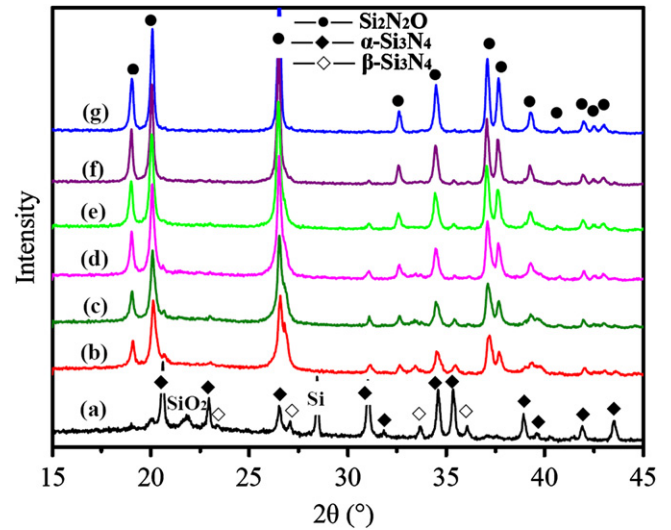


Fig. 2. XRD pattern of Si₂N₂O ceramics sintered at different temperatures.

[19,23,24], so when Si₂N₂O ceramic is fabricated by sintering at temperature above the melting point of Si, the SiO₂ particles in the green body may have chance to crystallize into cristobalite. However, liquid Si reacts with SiO₂ particles heterogeneously from the surface of SiO₂ particles to inside and the reaction speed is higher than the crystallization speed of SiO₂, so in the XRD pattern of Si₂N₂O-1450 there is no cristobalite detected and the amorphous phase is SiO₂ with a reason.

As known from Fig. 1, the $\Delta G_T^0(2)$ at 1400 °C is lower than that at 1450 °C, so the Si₂N₂O content in Si₂N₂O-1400 is theoretically more than that in Si₂N₂O-1450. $\Delta G_T^0(3)$ is lower than $\Delta G_T^0(2)$ when the temperature is below 1470 °C, which means Eq.(3) is easier to take place than Eq.(2) at 1450 °C. Actually, the Si₂N₂O content in Si₂N₂O-1450 is far higher than that in Si₂N₂O-1400. Fig. 3 gives the reaction model of the Si–SiO₂–N₂ system at temperatures below and above the melting point of Si respectively. When the green body is sintered at temperature below the melting point of Si (Fig. 3(a)), Eq.(2) could only take place at the contact area between Si and SiO₂ particles according to lattice diffusion, while Eq.(3) takes place rapidly to produce Si₃N₄ at the surface of Si particles. When the green body is sintered at temperature above the melting point of Si (Fig. 3(b)), the liquid Si surrounds the SiO₂ particles well, so Eqs.(2) and (4) take place easily, so there are lots of Si₂N₂O produced.

As the sintering temperature rises from 1450 to 1700 °C, $\Delta G_T^0(2)$ becomes far lower than $\Delta G_T^0(3)$, so Eq.(2) takes place even easier than Eq.(3). Besides, when the temperature rises to higher than 1650 °C, $\Delta G_T^0(4)$ also becomes lower than $\Delta G_T^0(3)$, leading to a faster decrease of Si₃N₄ in Si₂N₂O ceramic. As shown in Fig. 2, with temperature rising from 1450 to 1700 °C, Si₂N₂O ceramics increase in Si₂N₂O phase and decrease in Si₃N₄ phase, and there is only Si₂N₂O with hardly any Si₃N₄ in Si₂N₂O-1700.

4.2. Linear shrinkage and weight gain, density, porosity and pore size distribution

The linear shrinkage, weight gain, density and porosity of the $\text{Si}_2\text{N}_2\text{O}$ ceramics sintered at different temperatures are listed in Table 1. $\text{Si}_2\text{N}_2\text{O}$ -1400 is synthesized by solid-phase sintering, so the linear shrinkage of $\text{Si}_2\text{N}_2\text{O}$ -1400 is only 1.7% in three directions. When the sintering temperature rises to above the melting point of Si, the volume shrinkage of $\text{Si}_2\text{N}_2\text{O}$ ceramic gets large due to the liquid-phase sintering of $\text{Si}_2\text{N}_2\text{O}$ ceramic. The production of $\text{Si}_2\text{N}_2\text{O}$ has restraining effect on the shrinkage of $\text{Si}_2\text{N}_2\text{O}$ ceramic, and the more $\text{Si}_2\text{N}_2\text{O}$ produced, the more obvious the restraining effect shows. As listed in Table 1, due to the increase of $\text{Si}_2\text{N}_2\text{O}$ production speed with temperature rising from 1450 to 1700 °C, the linear shrinkage of $\text{Si}_2\text{N}_2\text{O}$ ceramic decreases from 9.6% to 6.4% in length (L) direction, from 9.0% to 6.3% in width (W) direction, and from 10.2% to 6.5% in thickness (T) direction. According to the linear shrinkage, $\text{Si}_2\text{N}_2\text{O}$ ceramic decreases in volume shrinkage from 26.1% to 18.0%.

As the Si in the green body reacts completely with nitrogen and SiO_2 from Eq.(2) or with nitrogen from Eq.(3), the theoretical weight gain of $\text{Si}_2\text{N}_2\text{O}$ ceramic is 38.9%. As shown in Table 1, the weight gain of $\text{Si}_2\text{N}_2\text{O}$ -1400 is only

12.9% because of the incomplete reaction of Si, while the weight gain of $\text{Si}_2\text{N}_2\text{O}$ -1450 is as high as 38.7% indicating an almost complete reaction of Si. At high temperature, SiO_2 and Si are volatile and the volatilization speed increases with temperature rising [25], so the $\text{Si}_2\text{N}_2\text{O}$ ceramic decreases little in weight gain from 38.7% to 37.9% with temperature rising from 1450 to 1700 °C.

The density and porosity of $\text{Si}_2\text{N}_2\text{O}$ ceramic are influenced together by the variation of volume shrinkage and weight gain. The density and porosity of $\text{Si}_2\text{N}_2\text{O}$ -1400 are 1.41 g/cm³ and 47.8% respectively. As the temperature rises to above the melting point of Si, $\text{Si}_2\text{N}_2\text{O}$ ceramic increases significantly in density due to its increase in volume shrinkage and weight gain. However, as the temperature rises from 1450 to 1700 °C, the volume shrinkage and weight gain of $\text{Si}_2\text{N}_2\text{O}$ ceramic decrease slowly, so the density of $\text{Si}_2\text{N}_2\text{O}$ ceramic decreases from 2.22 to 1.99 g/cm³ with the increase of porosity from 20.8% to 29.1%.

Fig. 4 shows the pore size distribution of $\text{Si}_2\text{N}_2\text{O}$ ceramics sintered at different temperatures. As can be seen, the $\text{Si}_2\text{N}_2\text{O}$ ceramics all show unimodal pore size distribution, and the pore size decreases with temperature rising. The pores in $\text{Si}_2\text{N}_2\text{O}$ -1400 are of micro-size. As the

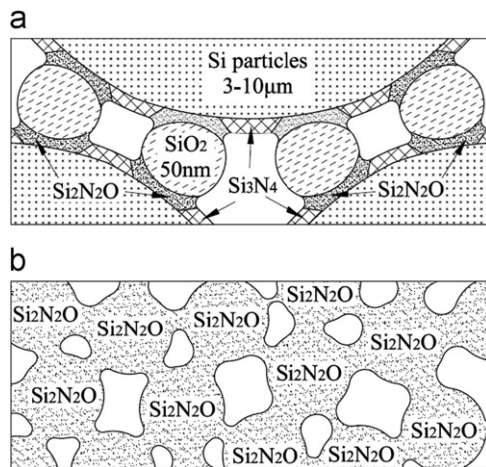


Fig. 3. Reaction model of the Si-SiO₂-N₂ system at temperatures (a) below and (b) above the melting point of Si.

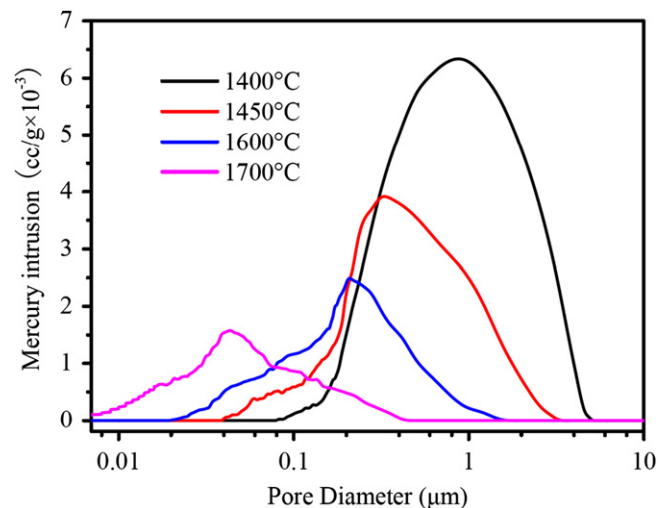


Fig. 4. Pore size distribution of $\text{Si}_2\text{N}_2\text{O}$ ceramics sintered at different temperatures.

Table 1
Shrinkage, weight gain, density and porosity of $\text{Si}_2\text{N}_2\text{O}$ ceramics sintered at different temperatures.

Temperature (°C)	Linear shrinkage (%)			Volume shrinkage (%)	Weight gain (%)	Density (g/cm ³)	Open porosity (%)
	L	W	T				
1400	1.7	1.7	1.7	5.0	13.5	1.41	47.8
1450	9.6	9.0	10.2	26.1	38.7	2.22	20.8
1500	8.6	8.1	9.1	23.6	38.6	2.15	23.4
1550	7.5	7.3	7.9	21.0	38.5	2.08	26.0
1600	6.9	6.8	7.1	19.4	38.3	2.03	27.6
1650	6.6	6.5	6.7	18.5	38.1	2.01	28.5
1700	6.4	6.3	6.5	18.0	37.9	1.99	29.1

temperature rises from 1450 to 1700 °C, the mean pore size of Si₂N₂O ceramic decreases from 0.4 to 0.04 μm.

4.3. Microstructure

Fig. 5 shows the micrographs at 10k-times magnification of Si₂N₂O ceramics sintered at different temperatures. During cold-pressing process, there are defects forms in the green body. Because of the small volume shrinkage of Si₂N₂O-1400, the defects are reserved and exist in the form of big pores in Si₂N₂O-1400 (Fig. 5(a)), and unreacted SiO₂ particles could be seen clearly. Due to the large volume shrinkage of Si₂N₂O-1450, the defects in Si₂N₂O-1450 (Fig. 5(b)) disappear, and the pores in Si₂N₂O-1450 are much smaller than that in Si₂N₂O-1400. As the temperature rises from 1450 to 1700 °C, Si₂N₂O ceramic increases in porosity from 20.8% to 29.1% and decreases in volume shrinkage from 26.1% to 18.0%, so the pores in Si₂N₂O ceramic theoretically increase. Seemingly, as can be seen by comparing Fig. 5(b)–(d), the pores in Si₂N₂O ceramic decrease on the contrary and the matrix gets denser with temperature rising.

Fig. 6 shows the micrographs at 50k-times magnification of Si₂N₂O ceramics sintered at different temperatures. In this magnification, it can be seen clearly that the matrix of Si₂N₂O ceramic is formed by stacking lots of 50 nm Si₂N₂O nanoparticles, and there are lots of nano-pores among the nanoparticles. By comparing Fig. 6(a)–(c), the pores in Si₂N₂O ceramic actually become more and bigger with temperature rising from 1450 to 1700 °C. The increase of the nano-pores makes the matrix of Si₂N₂O ceramic loose, and then influences the mechanical properties of Si₂N₂O ceramic.

4.4. Mechanical properties

From the above analysis, the phase composition, pore size distribution, porosity and microstructure of Si₂N₂O ceramic all vary with rising temperature, so the mechanical properties of Si₂N₂O ceramic are affected by these variations. Fig. 7 shows the flexural strength and fracture toughness of Si₂N₂O ceramics as functions of sintering temperature. Due to its high porosity and many big pores, Si₂N₂O-1400 possesses low flexural strength of 89 MPa and fracture toughness of 0.7 MPa m^{1/2}. Because of its low porosity and high Si₂N₂O content, Si₂N₂O-1450 possesses much higher flexural strength and fracture toughness than Si₂N₂O-1400. As the sintering temperature rises from 1450 to 1600 °C, the decrease of pore size makes the Si₂N₂O ceramic increase its flexural strength gradually from 183 to 229 MPa and fracture toughness from 1.9 to 2.3 MPa m^{1/2}. As the sintering temperature further rises to 1700 °C, because the matrix of Si₂N₂O ceramic gets loose, on the contrary Si₂N₂O ceramic decreases in flexural strength to 178 MPa and in fracture toughness to 1.9 MPa m^{1/2}.

Fig. 8 shows the Vickers hardness of Si₂N₂O ceramics as a function of sintering temperature. The Vickers hardness is affected both by phase composition and porosity. As to Si₂N₂O-1400, the Vickers hardness is only 2.4 GPa due to its high porosity. Si₂N₂O-1450 possesses high Vickers hardness due to its low porosity and high Si₂N₂O content. As the sintering temperature rises from 1450 to 1700 °C, the Si₂N₂O ceramic remains almost unchanged in phase composition but increases in porosity from 20.8% to 29.1%, so the Vickers hardness of Si₂N₂O ceramic decreases gradually from 5.6% to 4.5 GPa.

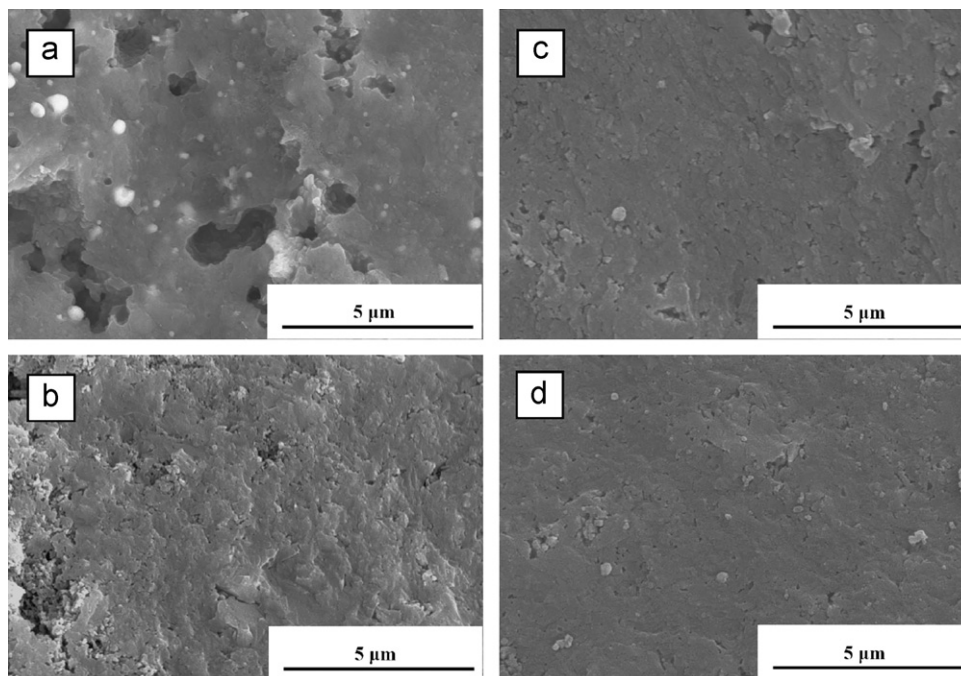


Fig. 5. Micrographs at 10k-times magnification of Si₂N₂O ceramics sintered at (a) 1400, (b) 1450, (c) 1600 and (d) 1700 °C.

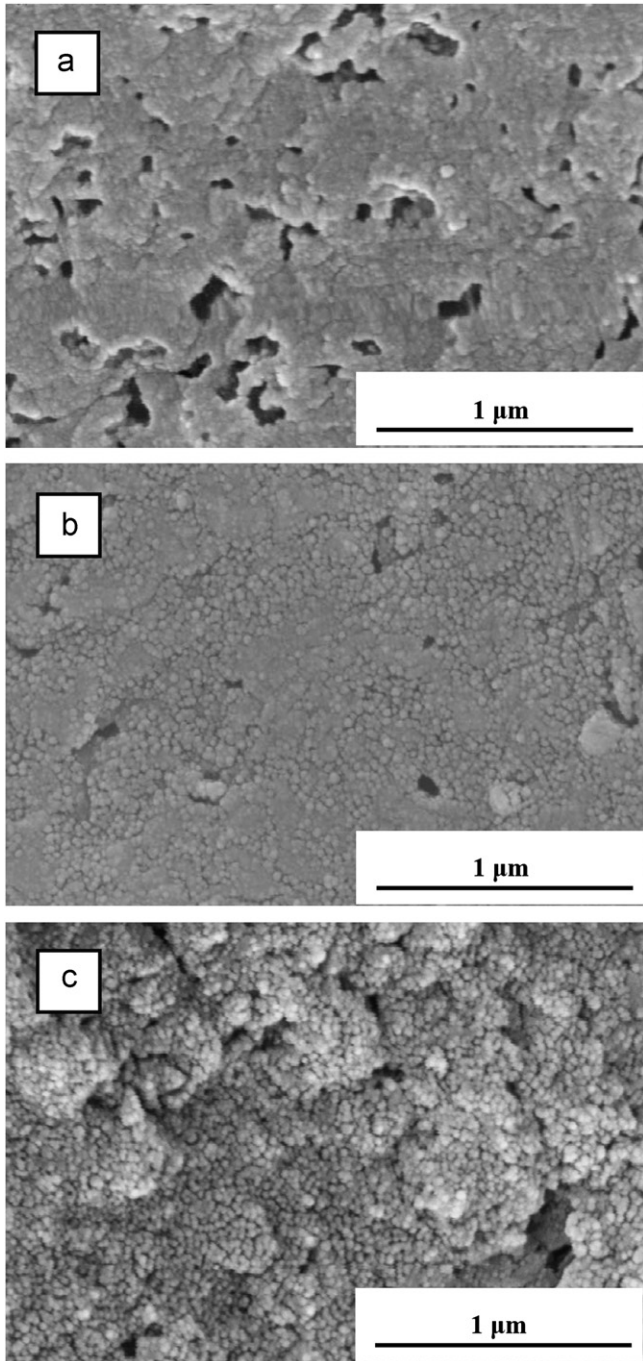


Fig. 6. Micrographs at 50k-times magnification of $\text{Si}_2\text{N}_2\text{O}$ ceramics sintered at (a) 1450, (b) 1600 and (c) 1700 °C.

5. Conclusions

$\text{Si}_2\text{N}_2\text{O}$ ceramics were fabricated by an in-situ reaction synthesis method. The sintering temperature has a great effect on the phase composition, porosity, pore size distribution, microstructure and mechanical properties of $\text{Si}_2\text{N}_2\text{O}$ ceramics.

1. The variation of phase composition with temperature coincides with the thermodynamics analyzing results of

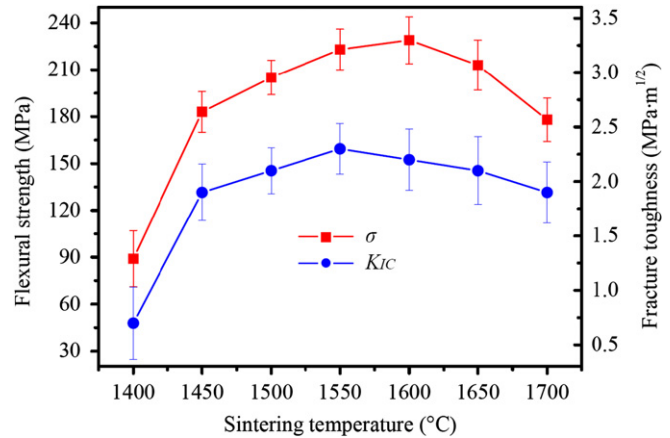


Fig. 7. Flexural strength and fracture toughness of $\text{Si}_2\text{N}_2\text{O}$ ceramics as functions of sintering temperature.

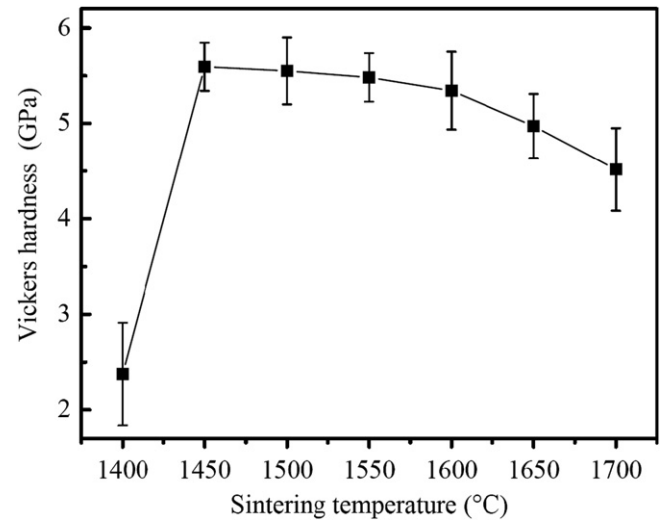


Fig. 8. Vickers hardness of $\text{Si}_2\text{N}_2\text{O}$ ceramics as a function of sintering temperature.

the Si– SiO_2 – N_2 system. When the ceramic is sintered at temperature below the melting point of Si, $\text{Si}_2\text{N}_2\text{O}$ is difficult to be produced. As the temperature rises to temperature above the melting point of Si, $\text{Si}_2\text{N}_2\text{O}$ is produced easily and Si_3N_4 content decreases with temperature rising. There is only $\text{Si}_2\text{N}_2\text{O}$ with hardly any Si_3N_4 in $\text{Si}_2\text{N}_2\text{O}$ -1700.

2. At temperature above the melting point of Si, liquid-phase sintering makes $\text{Si}_2\text{N}_2\text{O}$ ceramic shrink greatly, while the produced $\text{Si}_2\text{N}_2\text{O}$ retards the volume shrinkage of $\text{Si}_2\text{N}_2\text{O}$ ceramic. With the increase of $\text{Si}_2\text{N}_2\text{O}$ production speed as the sintering temperature rises from 1450 to 1700 °C, the $\text{Si}_2\text{N}_2\text{O}$ ceramic decreases gradually in volume shrinkage from 26.1% to 18.0% and in density from 2.22 to 1.99 g/cm³.
3. After sintering at temperature above the melting point of Si, the defects in $\text{Si}_2\text{N}_2\text{O}$ ceramic decrease significantly due to the large volume shrinkage, and there are hardly any big pores in $\text{Si}_2\text{N}_2\text{O}$ ceramic. With temperature rising

from 1450 to 1700 °C, the nano-pores in Si₂N₂O ceramic increase greatly with big pores decreasing.

4. After sintering at temperature above the melting point of Si, the high Si₂N₂O content, small pore size and low porosity make Si₂N₂O ceramic possess good mechanical properties. Si₂N₂O-1600 possesses the highest flexural strength of 229 MPa and fracture toughness of 2.3 MPa m^{1/2}.

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