

Mechanical and Dielectric Properties of Hot-pressed Si₃N₄ Ceramics with BaTiO₃ Addition

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Abstract: Si₃N₄ ceramics with improved and tailorable dielectric constant are desirable for microwave dielectric substrate application in harsh environment. Effect of BaTiO₃ addition on the mechanical and dielectric properties of the hot-pressed Si₃N₄ ceramics with Al₂O₃ and Nd₂O₃ as sintering additives were investigated. Relative density, flexural strength and Vickers' hardness of Si₃N₄ ceramics all decreased, while indentation fracture toughness increased monotonically with the increase of BaTiO₃ content. Flexural strength of Si₃N₄ ceramics was higher than 600 MPa and dielectric constant of Si₃N₄ ceramics increased to 9.26–11.50 when 5wt%–20wt% of BaTiO₃ was added. The dielectric loss was at the level of 10⁻³. The improvement of dielectric constant was attributed to the formation of TiN during hot pressing, as crystalline BaTiO₃ was not detected in the sintered samples. These findings can make a significant contribution to the new applications of Si₃N₄ ceramics.

Key words: Si₃N₄; fracture toughness; flexural strength; dielectric constant; hot pressing

With the development of communication and radar systems, the microwave dielectric materials have been extended to millimeter wave range, which requires that the dielectric ceramics possess low dielectric constant (<20, usually) to avoid the delay of electronic transition^[1-3]. Al₂O₃ ceramics have been extensively investigated as potential candidates for millimeter-wave application due to their high $Q \times f$, low ϵ_r and cost^[4-5]. However, the strength and toughness of Al₂O₃ are not adequate to withstand the increasingly harsh environment and achieve the miniaturization of devices. Hence, it is urgent to develop microwave dielectric ceramics with excellent mechanical property.

By comparison, Si₃N₄ ceramics are known for its superior mechanical properties^[6-8] as well as low dielectric loss, which is indispensable for the millimeter-wave applications^[9]. However, the dielectric constant is usually lower than 9 at 8–10 GHz even for the fully dense Si₃N₄ ceramics^[10], which is insufficient to meet the requirement of certain millimeter-wave application. Hence, it is the prime target to improve the dielectric constant of Si₃N₄ ceramics and achieve its tailorability. However, most of the work dielectric focus on the reduction of dielectric constant of Si₃N₄ ceramics for the wave-transparent application.

There is few reports on the improvement of the dielectric constant of Si₃N₄ ceramics^[11-12].

According to the Lichtenecker's logarithmic law of mixing, the introduction of particles with high dielectric constant could be an efficient strategy to enhance and tailor the dielectric constant of Si₃N₄ ceramics. Among the various ferroelectrics with high dielectric constant, BaTiO₃ possesses the advantages of high insulation, high permittivity, high thermal stability, low cost, wide application and so on^[13-15].

Hence, BaTiO₃ particles was adopted to enhance the dielectric property of Si₃N₄ ceramics in this investigation. In order to achieve higher density and reduce the impact of pores on the dielectric loss, which is difficult to be overcome in the pressureless sintered and gas pressure sintered Si₃N₄ ceramics, hot pressing was used for the densification of Si₃N₄ ceramics. Meanwhile, considering the low melting point (1625 °C) of BaTiO₃, Al₂O₃-Nd₂O₃ instead of conventional Al₂O₃-Y₂O₃ were used as sintering additives to lower the sintering temperature so as to avoid or decrease the volatilization of BaTiO₃. 5wt%–20wt% BaTiO₃ was added to improve the dielectric constant of Si₃N₄ ceramics. The flexural strength, fracture toughness and Vickers' hardness were measured to evaluate the

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effect of BaTiO₃ addition on the mechanical properties of Si₃N₄ ceramics. The dielectric constant and dielectric loss were measured to study the effect of BaTiO₃ addition on the dielectric properties of Si₃N₄ ceramics.

1 Experimental procedure

1.1 Materials preparation

α -Si₃N₄ (0.5 μ m, purity>99.9%, ANSM New Energy Technology Co., Ltd., Jiangsu, China) and BaTiO₃ (2 μ m, purity>99.9%, Qinhuangdao ENO High-Tech Material Development CO., Ltd., Hebei, China) were used as the starting materials. Al₂O₃ (0.4 μ m, purity>99.9%, Fenghe Ceramic Co., Ltd., Shanghai, China) and Nd₂O₃ (2 μ m, purity>99.9%, Qinhuangdao ENO High-Tech Material Development CO., Ltd., Hebei, China) were added as sintering additives.

Specimen powders were prepared by a conventional wet chemical method. Initially, four batches of powder mixtures consisting of Si₃N₄ powder, 3.68wt% Al₂O₃, 6.32wt% Nd₂O₃ and 5wt%–20wt% BaTiO₃ were homogenized in ethanol by ball milling at 300 r/min for 4 h. Subsequently, the resulting slurries were dried at 70 °C for 12 h. The dried powders were screened by a 149 μ m (100 mesh) stainless steel sieve. The as-received powders were placed into a graphite die and then hot pressed at 1680 °C for 2 h in a graphite furnace under flowing N₂ atmosphere. A uniaxial pressure of 20 MPa was applied through top graphite plungers. The obtained compacts were machined into rectangular bars with a size of 36 mm \times 4 mm \times 3 mm for mechanical tests and disks with the size of ϕ 15 mm \times 2 mm for dielectric tests.

1.2 Materials characterization

The apparent density (ρ) was measured by the Archimedes method. The flexural strength was measured by a three-point bending method with a span of 30 mm at a cross-head speed of 0.5 mm/min, using an Instron universal testing machine (Model 5566). Hardness was measured by Vickers' indentation applying a load of 49 N for 10 s on a polished section. The fracture toughness was estimated from the crack length generated by indents using the Evans and Davis's equation^[16]. All data were calculated according to the average values of six indents.

The dielectric constant (ϵ) and dielectric loss ($\tan\delta$) of the specimens were measured by RF impedance/material analyzer (Model 4291B, Agilent, USA) in the frequency range from 100 MHz to 1 GHz at room temperature.

The phase compositions of the specimens were determined using X-ray diffraction (D8 ADVANCE, Bruker, Germany) with Cu K α radiation. The diffraction data were collected within 10°–80° of 2θ range at a scanning step of 2 (°)/min. The morphologies of the polished and etched surfaces were

observed by scanning electron microscope (Magellan 400, FEI, USA).

2 Results and discussion

2.1 Phase composition and microstructure

Fig. 1 shows the XRD patterns of the hot-pressed Si₃N₄ ceramics with 5wt%–20wt% BaTiO₃ addition. Obviously, regardless of the addition of BaTiO₃, the major phase was β -Si₃N₄, indicating that the α -Si₃N₄ were transformed to β -Si₃N₄ due to the enhanced liquid phase flowing promoted by the external pressure during the hot pressing process, which facilitated the solution-precipitation process of Si₃N₄. No diffraction peaks of BaTiO₃ were detected, instead, diffraction peaks of TiN (JCPDS 65-0414) could be detected in these four specimens. In addition, diffraction peaks of Ba₅Al₂O₈ (JCPDS 28-0120) appeared when the amount of BaTiO₃ increased to 15wt%. The diffraction peaks of TiN and Ba₅Al₂O₈ intensified with the increase of BaTiO₃ content, indicating the continuous formation of TiN and Ba₅Al₂O₈. It has been reported that BaTiO₃ may decompose into BaO and TiO₂ during the sintering process^[17]. TiO₂ would react with Si₃N₄ to form TiN^[18]. Meanwhile, BaO would react with Al₂O₃ to form Ba₅Al₂O₈^[19]. Relevant reactions are shown as follows.

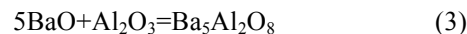
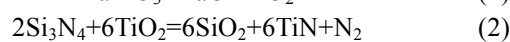
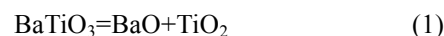


Fig. 2 reveals the microstructures of the Si₃N₄ specimens with 5wt%–20wt% BaTiO₃. Obviously, all these specimens were highly densified without visible voids or microcracks. The microstructure consisted of dark Si₃N₄ phase with elongated and equiaxed shapes, and bright grain boundary phase composed of the sintering additives and BaTiO₃-related reaction products. On the other hand, the grain size and amount of elongated Si₃N₄ grains decreased with the increase of BaTiO₃ content, due to the grain refinement effect of liquid phase which increased with more BaTiO₃ addition.

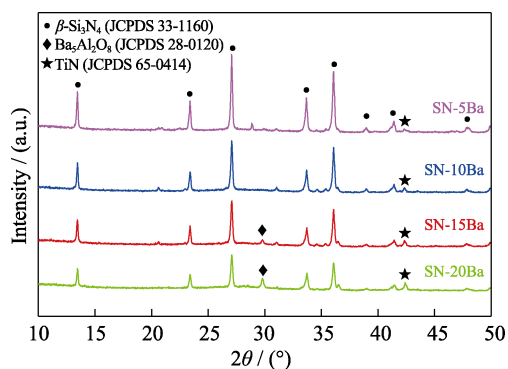


Fig. 1 XRD patterns of the hot-pressed Si₃N₄ ceramics with 5wt%–20wt% BaTiO₃

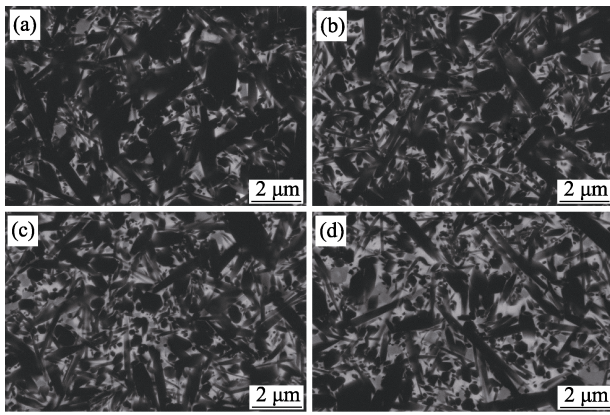


Fig. 2 Microstructure images of the Si₃N₄ specimens sintered with (a) 5wt%, (b) 10wt%, (c) 15wt% and (d) 20wt% BaTiO₃

2.2 Densification and mechanical properties

Fig. 3 illustrates the dependence of relative density and flexural strength of the Si₃N₄ ceramics on the BaTiO₃ content. Clearly, the relative density could reach as high as 96% without the addition of BaTiO₃, which was attributed to the fact that the uniaxial pressure was beneficial for the densification of ceramics by improving the fluidity of the liquid phase and elastic deformation of the grains. Nevertheless, the relative density exhibited slight decrease with the increase of BaTiO₃ content, which demonstrated that the liquid phase formed by the sintering additives (Al₂O₃ and Nd₂O₃) was already enough to promote the densification. Therefore, the additional liquid phase formed by the BaTiO₃-related products did not further improve the densification under hot pressing. Moreover, the redundant liquid phase would be squeezed out from the specimens due to the external pressure. Consistent with the trend of relative density, the flexural strength decreased obviously with the addition of BaTiO₃. The flexural strength of sample without BaTiO₃ was as high as (767.6±32.9) MPa. However, the flexural strength decreased to (752.3±41.1) MPa, (728.8±59.7) MPa, (681.2±63.2) MPa and (637.8±53.5) MPa with the BaTiO₃ addition of 5wt%, 10wt%, 15wt% and 20wt%, respectively.

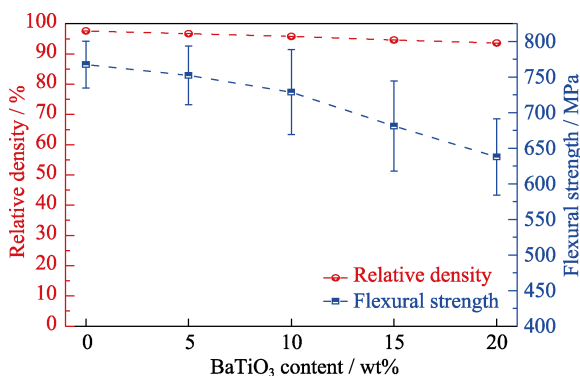


Fig. 3 Relative densities and flexural strength of the hot-pressed Si₃N₄ ceramics varying with the addition of BaTiO₃

On the one hand, the degradation of flexural strength is attributed to the addition of BaTiO₃ which has lower flexural strength inherently. On the other hand, the grain size and amount of elongated Si₃N₄ grains both decreased with the increase of BaTiO₃ addition. It is well acknowledged that the excellent mechanical property of Si₃N₄ ceramic is endowed by its unique microstructure of interlocked rod-like grains^[20]. Consequently, it is reasonable to speculate that the reduction of rod-like Si₃N₄ grains resulted in the flexural strength degradation to a great extent. Though the addition of BaTiO₃ played an adverse role in the flexural strength, it is noteworthy that the flexural strength of these Si₃N₄ ceramics still exceeds 600 MPa, which is much higher than those of traditional microwave dielectric ceramics.

The influence of BaTiO₃ addition on the indentation fracture toughness and Vickers' hardness is shown in Fig. 4. The fracture toughness of Si₃N₄ ceramics without BaTiO₃ was (4.92±0.24) MPa·m^{1/2}. When 5wt% BaTiO₃ was added into the Si₃N₄ ceramics, the fracture toughness increased to (5.72±0.22) MPa·m^{1/2}. With the further increase of BaTiO₃ content to 20wt%, the fracture toughness increased continuously to (7.26±0.14) MPa·m^{1/2}, which was 47.6% higher than that of the Si₃N₄ ceramics without BaTiO₃, indicating that the addition of BaTiO₃ was indeed beneficial for the improvement of fracture toughness. The reason for the increase of fracture toughness could be found from the crack propagation path induced by indentation tests. As shown in Fig. 5, in addition to the crack deflection induced by the elongated Si₃N₄ grains which endows Si₃N₄ ceramics with high fracture toughness in essence, the addition of BaTiO₃ introduced extra toughening mechanisms such as crack deflection, branching and bridging into the Si₃N₄ matrix due to the thermal expansion coefficient (TEC) mismatch between Si₃N₄ (3.0×10⁻⁶ K⁻¹)^[21] and TiN (9.35×10⁻⁶ K⁻¹)^[22]. Because of the TEC mismatch, these two phases shrink in different rates when cooled to room temperature, then residual stress field is created and the cracks propagation

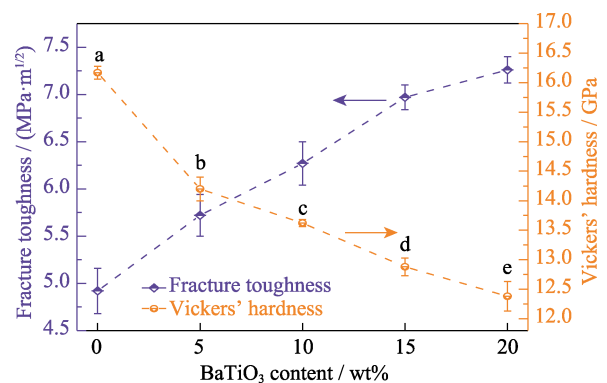


Fig. 4 Influence of BaTiO₃ addition on the indentation fracture toughness and Vickers' hardness of the hot-pressed Si₃N₄ ceramics

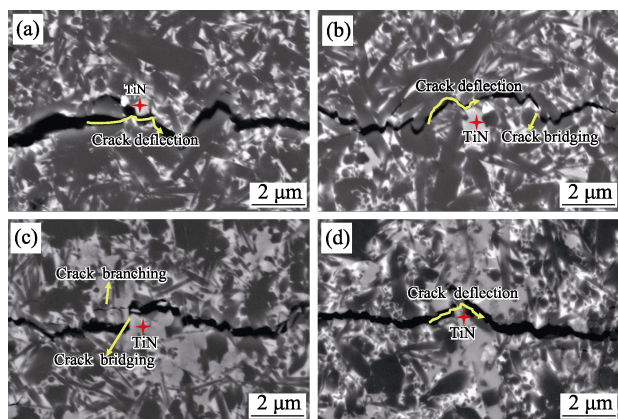


Fig. 5 Toughening mechanisms induced by the addition of BaTiO₃ to the hot-pressed Si₃N₄ ceramics (a) 5wt%; (b) 10wt%; (c) 15wt%; (d) 20wt%

is deflected when approaching the residual stress field. As a result, the cracks propagated in a more tortuous path and more fracture energy was dissipated, thus leading to the improvement of fracture toughness^[23].

On the contrary, the Vickers' hardness decreased with the increase of BaTiO₃ content. It can be seen that the slope of line ab (as labeled in Fig. 4) was steeper than that of line b-e, indicating the Vickers' hardness of Si₃N₄ ceramics dropped more sharply when the addition of BaTiO₃ increased from 0 to 5wt%. This demonstrated that the addition of BaTiO₃ dominated the mechanism of hardness decrease. Quite understandably, this was attributed to the fact that the Vickers' hardness of ceramics meet the rule of the mixture. Because the hardness of BaTiO₃-related reaction products is much lower than that of β-Si₃N₄ (14–16 GPa), it is reasonable that the Vickers' hardness decreases continuously with the addition of BaTiO₃ increasing.

The hardness of ceramics also strongly depends on its porosity according to Eq. (4):

$$H = H_0 e^{-bP} \quad (4)$$

where H represents the hardness, H_0 is the hardness without pores, b is a pre-exponential constant (which ranges from 2 to 8 relying on the ceramic investigated), and, P is the porosity of the ceramic^[24].

Obviously, the porosity intensively reduces the resistance of the microstructure against the applied loads^[25-26]. As mentioned in Fig. 3, the porosity in the Si₃N₄ matrix increased with the increase of BaTiO₃ content. As a result, the Vickers' hardness decreased with the increase of BaTiO₃ content, following the trend given by Eq. (4).

2.3 Dielectric properties

The dielectric constant is defined as the ratio between the absolute permittivity of material and the absolute permittivity of vacuum. The higher the dielectric constant, the stronger the ability to bind charge. For the dielectric

substrate application, higher dielectric constant is favored for the miniaturization of resonator. The dielectric constant of the hot-pressed Si₃N₄ ceramic without BaTiO₃ was 7.56. Fig. 6 summarizes the dielectric constant (ϵ) and dielectric loss ($\tan\delta$) of specimens with 5wt%–20wt% BaTiO₃ frequency. Obviously, the dielectric constant increased monotonically from 9.26 to 11.50 for the Si₃N₄ ceramics blended with BaTiO₃ content increasing, which increased by 23.5%–53.3% compared to the Si₃N₄ ceramics without BaTiO₃. Considering the fact that the dielectric constant of the composites obeys the logarithmic law^[27], the addition of BaTiO₃ was indeed effective for the improvement of the dielectric constant of Si₃N₄ ceramics. The trend of growth slowed down with the increase of BaTiO₃ addition from 15wt% to 20wt% may be ascribed to the appearance of pores, of which the dielectric constant was regarded as 1^[28]. As mentioned above, some of the BaTiO₃ particles were consumed by hot decomposition and reaction with N₂, sintering additives and Si₃N₄. Consequently, the improvement of the dielectric constant was probably attributed to the formation of TiN grains which have a dielectric constant about 40. Dielectric loss is defined as the loss of energy that goes into heating a dielectric material in a varying electric field. The dielectric loss increased with the increase of BaTiO₃ content as well, which was probably due to the extrinsic losses caused by the secondary phase. Generally speaking, higher dielectric constant will lead to higher dielectric loss which may cause the thermal failure of the device. However, in this investigation, they were all at the level of 10⁻³, which were similar to those of the specimens without BaTiO₃ addition and applicable as dielectric substrates.

3 Conclusions

In the present work, the mechanical and dielectric properties of hot-pressed Si₃N₄ ceramics with the addition

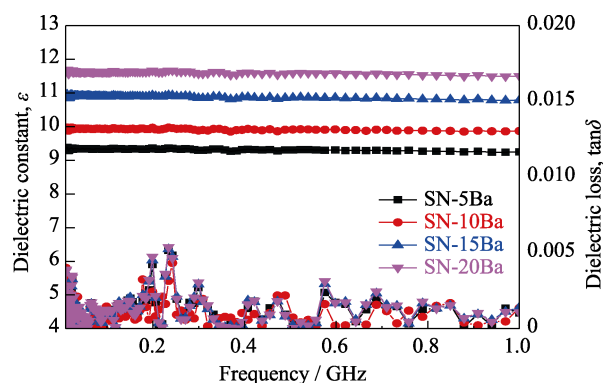


Fig. 6 Change of dielectric constant and dielectric loss with frequency for the hot-pressed Si₃N₄ ceramics with different BaTiO₃ additions

of 5wt%–20wt% BaTiO₃ have been investigated. While the relative density, flexural strength, Vickers' hardness and dielectric constant decreased with the increase of BaTiO₃ addition, the fracture toughness increased. The flexural strength of these Si₃N₄ ceramics exceeded 600 MPa, much higher than those of traditional microwave dielectric ceramics. The dielectric constant of hot-pressed Si₃N₄ ceramics could be improved by the addition of BaTiO₃. The dielectric loss was at the level of 10⁻³, which is tolerable for application.

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添加 BaTiO₃ 的热压烧结 Si₃N₄ 陶瓷的力学和介电性能

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摘要: 本研究以 Al₂O₃ 和 Nd₂O₃ 为烧结助剂, 采用热压烧结法制备 Si₃N₄ 陶瓷, 系统研究了添加 BaTiO₃ 对 Si₃N₄ 陶瓷力学和介电性能的影响。研究表明, 随着 BaTiO₃ 含量的增加, 相对密度、抗弯强度和维氏硬度都随之降低, 而断裂韧性有所升高; 即使添加 5wt%~20wt% 的 BaTiO₃, Si₃N₄ 陶瓷的抗弯强度依然可以保持在 600 MPa 以上。Si₃N₄ 陶瓷的介电常数可以提高到 9.26~11.50, 而介电损耗保持在 10⁻³ 量级。在 Si₃N₄ 陶瓷中未检测到 BaTiO₃ 结晶相, 可以认为 Si₃N₄ 陶瓷介电常数的提高主要来源于烧结过程中形成的 TiN。这些结果有助于拓展 Si₃N₄ 陶瓷的应用领域。

关键词: 氮化硅; 断裂韧性; 抗弯强度; 介电常数; 热压烧结

中图分类号: TQ174 文献标志码: A