Fabrication and properties of \( \text{Si}_2\text{N}_2\text{O} \) ceramics for microwave sintering furnace

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Abstract

The \( \text{Si}_2\text{N}_2\text{O} \) ceramics with low dielectric constant, low thermal diffusivity and high thermal shock resistance were successfully prepared by vacuum sintering. The amorphous \( \text{Si}_3\text{N}_4 \) was used as raw material with \( \text{Li}_2\text{CO}_3 \) as sintering additive. The phase, microstructure, oxidized resistance, mechanical and dielectric properties of the \( \text{Si}_2\text{N}_2\text{O} \) ceramics were investigated. XRD analysis showed that the suitable content of \( \text{Li}_2\text{CO}_3 \) could promote the generation of \( \text{Si}_2\text{N}_2\text{O} \) ceramics. However, the excess or insufficient amount of \( \text{Li}_2\text{CO}_3 \) additive would cause decomposition of \( \text{Si}_2\text{N}_2\text{O} \) phase. The \( \text{Li}_2\text{O} \) volatilized at high temperature leaving highly pure (99.63\%) porous \( \text{Si}_2\text{N}_2\text{O} \) ceramics. The flexural strength of the porous \( \text{Si}_2\text{N}_2\text{O} \) ceramics (with \( \approx 49.19\% \) of open porosity) was about 30 MPa, the residual strength ratio was more than 70\% after 1300 °C quenching in air. The \( \text{SiO}_2 \) layer formed by oxidization could prevent \( \text{Si}_2\text{N}_2\text{O} \) ceramics from further oxidizing. Therefore, these \( \text{Si}_2\text{N}_2\text{O} \) ceramics will be excellent thermal insulation and wave-transparent materials for high temperature microwave sintering furnace.

Keywords: porous \( \text{Si}_2\text{N}_2\text{O} \), \( \text{Li}_2\text{CO}_3 \) additive, vacuum sintering, dielectric properties, thermal shock resistance

I. Introduction

As an advanced sintering technology, microwave sintering plays a key role in the field of ceramics fabrication and it has attracted considerable attention in recent years [1–4]. However, the lack of insulation and microwave transparent materials with excellent thermal shock resistance and dielectric properties limits the high temperature (>1300 °C) application [5]. Therefore, it is necessary to develop thermal insulation and wave-transparent materials for high temperature application in microwave sintering technique. So far, there are several high performance wave-transparent materials, such as \( \beta \)-SiAlON ceramics [6–8], \( \text{Si}_3\text{N}_4 \) ceramics [9–11], \( \text{SiO}_2 \) ceramics [12,13], \( \text{Si}_2\text{N}_2\text{O} \) ceramics [14–16] and related composite materials [17–20]. Among them, the \( \text{Si}_3\text{N}_4 \) ceramics is the only compound in the \( \text{SiO}_2 \cdot \text{Si}_3\text{N}_4 \) system and it has the lowest dielectric constant (6.0). The dielectric constants of \( \beta \)-SiAlON and \( \text{Si}_3\text{N}_4 \) are 8.8 and 7.9, respectively [8,21]. In addition, the \( \text{Si}_2\text{N}_2\text{O} \) ceramics possesses relatively low theoretical density (2.81 g/cm\(^3\)), high hardness (17–22 GPa), low thermal expansion coefficient (3.5×10\(^{-6}\) K\(^{-1}\)), high thermal stability up to 1750 °C and oxidation resistance, making it an ideal choice for thermal insulation and wave-transparent materials [8,14,21–24]. Many studies reveal that pure and dense \( \text{Si}_2\text{N}_2\text{O} \) ceramics exhibit excellent mechanical and dielectric properties, making it suitable for use in the wave-transparent field, such as the microwave sintering and missile radomes [20,21,25]. However, the sintered dense materials show poor thermal shock resistance (critical temperature difference of about 600 °C) and higher dielectric constant (6.17 at 1 MHz) [21,26], which is still too high for microwave sintering. Therefore, appropriate methods should be tested to improve thermal shock resistance and reduce

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Si\(_2\)N\(_2\)O ceramics was about 30 MPa, the residual strength ratio was more than 70\% after 1300 °C quenching in air. It would cause decomposition of Si\(_2\)O\(_2\), promote the generation of Si\(_2\)N\(_2\)O ceramics. The flexural strength of the porous Si\(_2\)N\(_2\)O ceramics (with \( \approx 49.19\% \) of open porosity) was about 30 MPa, the residual strength ratio was more than 70\% after 1300 °C quenching in air. The Si\(_2\)O\(_2\) layer formed by oxidization could prevent Si\(_2\)N\(_2\)O ceramics from further oxidizing. Therefore, these Si\(_2\)N\(_2\)O ceramics will be excellent thermal insulation and wave-transparent materials for high temperature microwave sintering furnace.

Keywords: porous Si\(_2\)N\(_2\)O, Li\(_2\)CO\(_3\) additive, vacuum sintering, dielectric properties, thermal shock resistance

I. Introduction

As an advanced sintering technology, microwave sintering plays a key role in the field of ceramics fabrication and it has attracted considerable attention in recent years [1–4]. However, the lack of insulation and microwave transparent materials with excellent thermal shock resistance and dielectric properties limits the high temperature (>1300 °C) application [5]. Therefore, it is necessary to develop thermal insulation and wave-transparent materials for high temperature application in microwave sintering technique. So far, there are several high performance wave-transparent materials, such as \( \beta \)-SiAlON ceramics [6–8], Si\(_3\)N\(_4\) ceramics [9–11], Si\(_2\)O\(_2\) ceramics [12,13], Si\(_2\)N\(_2\)O ceramics [14–16] and related composite materials [17–20]. Among them, the Si\(_3\)N\(_4\) ceramics is the only compound in the SiO\(_2\)-Si\(_3\)N\(_4\) system and it has the lowest dielectric constant (6.0). The dielectric constants of \( \beta \)-SiAlON and Si\(_3\)N\(_4\) are 8.8 and 7.9, respectively [8,21]. In addition, the Si\(_2\)N\(_2\)O ceramics possesses relatively low theoretical density (2.81 g/cm\(^3\)), high hardness (17–22 GPa), low thermal expansion coefficient (3.5×10\(^{-6}\) K\(^{-1}\)), high thermal stability up to 1750 °C and oxidation resistance, making it an ideal choice for thermal insulation and wave-transparent materials [8,14,21–24]. Many studies reveal that pure and dense Si\(_2\)N\(_2\)O ceramics exhibit excellent mechanical and dielectric properties, making it suitable for use in the wave-transparent field, such as the microwave sintering and missile radomes [20,21,25]. However, the sintered dense materials show poor thermal shock resistance (critical temperature difference of about 600 °C) and higher dielectric constant (6.17 at 1 MHz) [21,26], which is still too high for microwave sintering. Therefore, appropriate methods should be tested to improve thermal shock resistance and reduce
the dielectric constant. Many researchers have reported that porous ceramics possess both better thermal shock resistance and lower dielectric constant due to the lower air dielectric constant (1.0) [9,15]. Thus, the lower dielectric constant and better thermal shock resistance of porous Si$_3$N$_4$O ceramics could satisfy the requirements of the microwave sintering furnace.

Various processing techniques have been developed to prepare porous Si$_3$N$_4$O ceramics, such as gel-casting [27,28], adding pore former [15,29,30], and polymer-derived ceramics [31]. However, the impurities were left due to the incomplete reaction with each other, which seriously affected dielectric properties of materials. Recently, some reports utilized the characterization of rapid volatilization of Li$_2$O sintering additives at high temperature to reduce sintering additives residues [32–34], which might be beneficial for the fabrica tion of the pure porous Si$_3$N$_4$O ceramics.

In this paper, the Li$_2$CO$_3$ was used as precursor additive to form Li$_2$O at high temperature. By vacuum sintering, the Li$_2$O was discharged from porous Si$_3$N$_4$O ceramics. The influence of Li$_2$O content on the phase, morphology, mechanical and dielectric properties of Si$_2$N$_2$O ceramics was investigated. The effects of porosity and oxidation on dielectric properties were also discussed.

II. Experimental details

2.1. Materials preparation

Nano-sized amorphous Si$_3$N$_4$ powder (>99% purity, ~20 nm, Shanghai Chao Wei Nanotechnology Co., Ltd, China) was used as starting material (Fig. 1). It can be seen that the raw powder has homogeneous distribution and the average size of Si$_3$N$_4$ particles is about 50 nm. Li$_2$CO$_3$ (99% purity, Xilong Scientific Co., Ltd. China) in different amount (1.0, 2.0, 3.0 and 5.0 wt.%) was added to the amorphous Si$_3$N$_4$ powder as sintering additive. Si$_3$N$_4$ powders were attrition milled with different sintering additive content (1.0, 2.0, 3.0 and 5.0 wt.%) using PyNN, SPEX-8000M. The mixed powders were ultrasonically dispersed in ethanol for 15 min and then mixed by high energy milling for 1 h using a 375 ml Si$_3$N$_4$ bottle with Si$_3$N$_4$ balls.

In order to increase oxygen content in the raw materials, the amorphous Si$_3$N$_4$ powder was pre-oxidized at 1000°C for 2 h. The pre-oxidized amorphous Si$_3$N$_4$ powder and different content of the sintering additive were mixed and milled by Si$_3$N$_4$ balls media in ethanol for 30 min. Then, the mixtures were dried at 100°C for 8 h under vacuum. The mixtures were sieved though a 100 mesh screen, then the powder mixtures were uniaxially pressed under 50 MPa to form rectangular green body (6 mm × 9 mm × 36 mm). The green pellets were placed in an alumina crucible and sintered in vacuum furnace with packing of Si$_3$N$_4$ powder by a multi-step sintering approach. Firstly, sintering temperature was increased to 1000°C with the heating rate of 8 °C/min, then increased to 1450°C with the heating rate of 5 °C/min. Secondly, the temperature was further increased to 1650°C with the heating rate of 2 °C/min and held for 2 h, followed by natural cooling to room temperature.

The porosity was measured by the Archimedes displacement method. Three-point bending strength was determined at room temperature using a universal testing machine at a crosshead speed of 0.5 mm/min with a span of 30 mm. Fracture toughness ($K_{IC}$) was performed using the single-edge notch beam (SENB) method, the size of specimens was 3 mm × 4 mm × 20 mm with a notch of 2 mm depth and 0.2 mm width and a 16 mm span. The fracture toughness was calculated using the following equation:

$$K_{IC} = \frac{3PL \sqrt{a}}{2bh^2} \left[ 1.93 - 3.07 \frac{a}{h} + 14.53 \left( \frac{a}{h} \right)^2 - 25.07 \left( \frac{a}{h} \right)^3 + 25.08 \left( \frac{a}{h} \right)^4 \right]$$

where $P$ is the applied indentation load, $L$ is the span, $a$ is the specimen pre-crack depth, $b$ is the sample cross section width and $h$ is the sample cross section height [35].

Thermal shock resistance experiments were carried out in a muffle furnace at temperatures between 900 and 1300°C in air. After the furnace was heated to the desired temperature, the ground and polished rectangular bars were put into the furnace and kept for 20 min. Then the samples were quenched in air and the residual flexural strength was performed using a universal testing machine. Five samples were characterized to get each final average value. The mass change was measured to evaluate Li content and the oxidation degree.

The microstructures were characterized by scanning electron microscopy (SEM, JEOL JSM-6700F, Japan). The phase was identified by X-ray diffraction (XRD, Beijing Purkinje General Instrument Co. Ltd, Cu-Kα radiation). The quantitative analysis of Si$_2$N$_2$O, Si$_3$N$_4$ and
SiO$_2$ was calculated based on the peak intensity using the following equation:

$$X_i = \left( \frac{k_i}{\sum_{i=1}^{n} I_i k_i} \right)^{-1}$$  

(2)

where $k$ is the internal standard and $I_i$ is the strongest peak’s integrated intensity [36,37].

The residual contents of Li in the as-prepared samples after sintering were determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES, Thermo Electron Corporation, MA). The samples were crushed into powders and ultrasonically etched in HNO$_3$/HF solution for 12 h to ensure complete dissolution of the Li in the liquid. The dielectric constant ($\varepsilon$) and loss tangent (tan $\delta$) of the Si$_2$N$_2$O ceramics were measured on the Agilent N5244A vector network analyser system in the frequency range of 2–18 GHz at room temperature.

III. Results and discussion

3.1. Phase and microstructure

In order to study the influence of oxygen content on the crystallized product of amorphous Si$_3$N$_4$, the amorphous Si$_3$N$_4$ raw material was pre-oxidized in the muffle furnace in air at 1000°C and the heating rate was 10°C/min with 2 h holding. Comparison of the un-oxidized and pre-oxidized samples is shown in Fig. 2. It can be seen that amorphous Si$_3$N$_4$ is more crystallized at the same temperature after pre-oxidation. On the other hand, the Si$_2$N$_2$O and SiO$_2$ peaks appeared when the amorphous Si$_3$N$_4$ was oxidized, which proved that per-oxidation can provide a lot of oxygen and produces Si$_2$N$_2$O.

Figure 3 shows the phase evolution of the sintered bodies with 0, 1, 2, 3 and 5 wt.% of sintering additives after sintering at 1650°C for 2 h. It can be seen from Fig. 3, that the sintered sample without any additives consisted of $\alpha$-Si$_3$N$_4$, Si$_2$N$_2$O and $\beta$-Si$_3$N$_4$. The residual Li content in the as-prepared samples has been detected by inductively coupled plasma-atomic emission spectrometry (ICP-AES, Thermo Electron Corporation, MA). The residual Li content decreased from 0.022% to 0.015% with the decrease of additive content from 5 to 1 wt.%.

The $\alpha$-Si$_3$N$_4$ was the major phase, while Si$_2$N$_2$O and $\beta$-Si$_3$N$_4$ could also be detected. The single phase Si$_2$N$_2$O was prepared when the additive content was 2 wt.% The $\alpha$-Si$_3$N$_4$ peaks of the sample with 1 wt.% additive content can be observed in Fig. 3. This phenomenon indicates that the conversion of $\alpha$-Si$_3$N$_4$ to Si$_2$N$_2$O was not complete due to the relatively low Li$_2$O liquid amount. The $\beta$-Si$_3$N$_4$ and SiO$_2$ peaks appeared when the additive content was higher than 3 wt.%, which proved that the decomposition of Si$_2$N$_2$O took place. In addition, the peaks of $\beta$-Si$_3$N$_4$ and SiO$_2$ were enhanced with the increasing of additive content as seen in Fig. 3. These results suggested that the decomposition of Si$_2$N$_2$O was easier in the rich Li$_2$O liquid envi-
The decomposition reaction of $\text{Si}_2\text{N}_2\text{O}$ mainly included the following equations [14,24]:

$$3 \text{Si}_2\text{N}_2\text{O}(s) \rightarrow 2 \text{Si}_3\text{N}_4(s) + 3 \text{SiO}(g) + \text{N}_2(g) \quad (3)$$

$$2 \text{Si}_2\text{N}_2\text{O}(s) + 3 \text{O}_2(s) \rightarrow 4 \text{SiO}(g) + 2 \text{N}_2(g) \quad (4)$$

Figure 4 shows the typical morphology of the porous $\text{Si}_2\text{N}_2\text{O}$ ceramics with different additive content after sintering at 1650°C for 2 h. As shown in the Fig. 4a, small rod-shaped grains were present in the porous $\text{Si}_2\text{N}_2\text{O}$ ceramics. Furthermore, it can be seen from Fig5. 4b-e that the samples with different $\text{Li}_2\text{O}$ content exhibited a rod-shaped grain morphology. However, when the additive content was 3 wt.% or 5 wt.%, the typical elongated $\beta$-$\text{Si}_3\text{N}_4$ grains were visible as shown in Figs. 4d,e. According to the previous XRD analysis, the elongated $\beta$-$\text{Si}_3\text{N}_4$ grains came from the decomposition of $\text{Si}_2\text{N}_2\text{O}$. Granular morphology with a short grown rod-shaped crystal phase was present in $\text{Si}_2\text{N}_2\text{O}$ ceramics when the additive content was 1 wt.% and the average size of $\alpha$-$\text{Si}_3\text{N}_4$ was about 1 µm. However, typical elongated and needle-like $\beta$-$\text{Si}_3\text{N}_4$ grains were visible when the additive content was 5 wt.% and the average size of $\beta$-$\text{Si}_3\text{N}_4$ was about 2.5 µm.

The porosity and density of samples are listed in Table 1. The open porosity has been slightly decreased when the additive content was more than 2 wt.%. According to mass loss, three reasons were responsible for the porosity and mass loss: i) the decomposition of $\text{Si}_2\text{N}_2\text{O}$, ii) the decomposition of $\text{Li}_2\text{CO}_3$ and iii) the $\text{Li}_2\text{O}$ evaporated when the vapour pressure of $\text{Li}_2\text{O}$ was higher than 5 Pa at 1650°C. The decomposition of $\text{Li}_2\text{CO}_3$ and evaporation of $\text{Li}_2\text{O}$ lead to porosity increasing. So, the porosity decreasing indicates that the decomposition of $\text{Si}_2\text{N}_2\text{O}$ can decrease porosity. In the Table 1, quantitative analysis showed that the fraction of $\text{Si}_2\text{N}_2\text{O}$ reached minimum when the amount of additive content was 5 wt.%. This result is consistent with XRD patterns in Fig. 3. It was reported in literature [8,24,32] that the atomic diffusion became easier when the $\text{Si}_2\text{N}_2\text{O}$ crystals were wetted by a low viscous melt of $\text{LiO}_2$-$\text{SiO}_2$. So the viscosity of the melts reduced with the increase of additive content, which facilitated the decomposition of $\text{Si}_2\text{N}_2\text{O}$.

![Figure 4. SEM morphology of the Si₂N₂O ceramics sintered at 1650°C for 2 h with different Li₂O contents:](image)

<table>
<thead>
<tr>
<th>Additive content [wt.%]</th>
<th>Open porosity [%]</th>
<th>Density [g·cm⁻³]</th>
<th>Mass loss [%]</th>
<th>Fraction of Si₂N₂O [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>57.11</td>
<td>1.12</td>
<td>1.60</td>
<td>37.90</td>
</tr>
<tr>
<td>1</td>
<td>52.16</td>
<td>1.23</td>
<td>5.89</td>
<td>85.56</td>
</tr>
<tr>
<td>2</td>
<td>49.19</td>
<td>1.34</td>
<td>8.71</td>
<td>99.63</td>
</tr>
<tr>
<td>3</td>
<td>47.43</td>
<td>1.41</td>
<td>10.66</td>
<td>83.83</td>
</tr>
<tr>
<td>5</td>
<td>45.82</td>
<td>1.51</td>
<td>15.47</td>
<td>71.07</td>
</tr>
</tbody>
</table>
3.2. Mechanical and dielectric properties

The mechanical properties of the sintered samples with different additive content were listed in Table 2. It can be seen from Table 2 that the bending strength, elastic modulus and fracture toughness appeared to have a linear growth and they significantly increase with the additive content from 18 MPa, 8.16 GPa and 1.12 MPa·m$^{1/2}$ to 72 MPa, 31.7 GPa and 1.79 MPa·m$^{1/2}$. The mechanical properties of the specimens were obviously quite sensitive to the additive content.

Figure 5 shows the influence of temperature and additive on the thermal diffusivity. The thermal diffusivity of the material decreased with the increase of temperature. It has been reported that the phase composition and porosity of the Si$_3$N$_4$O ceramics have important influence on the thermal diffusivity. When the sintering additive content was 2 wt.%, Si$_3$N$_4$O acted as the main crystalline phase. The thermal diffusivity of Si$_3$N$_4$O was lower than that of Si$_3$N$_4$, so the sample with 2 wt.% additive has the lowest thermal diffusivity. This result indicates that the Si$_3$N$_4$O ceramics has good thermal insulation properties and can meet the requirements of refractory properties for thermal insulation performance.

Figure 6. Weight gain of the different samples versus oxidation time at 1300 °C

3.2.3. Thermal shock resistance

The thermal shock resistance performances of the samples were shown in Fig. 8. It can be seen that the residual strength showed little change from room temperature to 900 °C. The residual strength dropped sharply when the quenching temperature was above 1100 °C. However, for the sample with 2 wt.% sintering additive, its residual strength ratio was more than 70%, even though the quenching temperature was above 1300 °C. This result indicates that the porous Si$_3$N$_4$O ceramics prepared with 2 wt.% of sintering additive has excellent thermal shock resistance. These were caused by the difference of the thermal expansion coefficient between Si$_3$N$_4$O and oxidized layer (SiO$_2$) during cooling procedure. Therefore, the different thermal shock resistance of the samples could be attributed to the sintering additive and oxidation degree [40–42].

Apart from mechanical properties and high temperature properties, dielectric properties were also important.

Table 2. Mechanical properties of the obtained samples with different additive content

<table>
<thead>
<tr>
<th>Additive content [wt.%]</th>
<th>Bending strength [MPa]</th>
<th>Elastic modulus [GPa]</th>
<th>Fracture toughness [MPa·m$^{1/2}$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>18 ± 5.1</td>
<td>8.16 ± 1.5</td>
<td>1.12 ± 0.05</td>
</tr>
<tr>
<td>2</td>
<td>30 ± 4.7</td>
<td>13.7 ± 2.6</td>
<td>1.19 ± 0.08</td>
</tr>
<tr>
<td>3</td>
<td>44 ± 6.6</td>
<td>19.4 ± 2.2</td>
<td>1.34 ± 0.06</td>
</tr>
<tr>
<td>5</td>
<td>72 ± 6.2</td>
<td>31.7 ± 3.1</td>
<td>1.79 ± 0.09</td>
</tr>
</tbody>
</table>

Thümmler [38]. Figure 7 shows XRD patterns (A) and SEM images (B) of the surface of the different oxidized samples. According to the XRD pattern of the sample after oxidation, the formed oxidation product was recognized as SiO$_2$, which was expected according to Eq. 4. As can be seen from SEM images of the oxidized surfaces, very small particles just appeared on the surface of the sample with 1 wt.% sintering additive. However, the sample’s surface was completely covered by glassy phase when the amount of sintering additive was more than 1 wt.%. The formed coherent SiO$_2$ layer blocked the oxygen diffusion to a certain degree, so further oxidation can be suppressed [24,39].

It was reported that Si$_3$N$_4$O was unstable at high temperatures when exposed to oxidation atmosphere. It can be seen from Fig. 6 that the oxidation reaction of the Si$_3$N$_4$O causes weight increase. It is worth noticing that the initial oxidation rate of the porous Si$_3$N$_4$O ceramics was high and then the oxidation rate dropped rapidly with increase of the oxidation duration. The effects of the porosity on the oxidation resistance of the porous Si$_3$N$_4$ ceramics were described according to Porz and Thümmler [38].
Figure 7. XRD patterns (A) and SEM images (B) of the surface of the different samples oxidized at 1300 °C for 30 h: 
a) 1, b) 2, c) 3 and d) 5 wt.%

Figure 8. Residual strength and residual strength ratio of the Si$_2$N$_2$O ceramics with different Li$_2$O contents as a function of quenching temperature difference

Figure 9. Effects of additive content (a) and oxidation time (b) on the dielectric constant and loss tangent of porous Si$_2$N$_2$O ceramics at 2.45 GHz
parameters for wave-transparent applications. Figures 9a and 9b showed the dielectric constant (ε) and loss tangent (tan δ) of the samples with different sintering additive content and oxidation time respectively, which were measured at the frequency of microwave sintering around 2.45 GHz. It can be found that all the samples exhibited reasonably ultra-low dielectric constant (around 2.45 GHz. It can be found that all the samples were measured at the frequency of microwave sintering furnace.

Another reason influencing the dielectric constant was the difference in composition, including different Si₃N₄ and Si₂N₂O content in the final composition and different sintering additive content in starting material. It has been discovered that residual Li⁺ content was lower than 1/10 of the starting material because of the easy volatilization of Li₂O above 1300 °C [32,34,45]. On the other hand, the increasing amount of Si₃N₄ and SiO₂ caused by Si₂N₂O decomposition would increase dielectric constant. However, the content of those phases was also low. Therefore, the effect of sintering additive content and other phase on dielectric constant can be ignored, and porosity was considered to be the major factor influencing the dielectric constant. As for oxidation time, there are two aspects of changes happening in the products, namely the decrease of porosity and the rise of SiO₂ phase content as discussed above. Although the porosity decreased evidently after the oxidation, the oxidation products of Si₂N₂O can reduce the dielectric constant greatly. Because the dielectric constant of SiO₂ is lower than Si₂N₂O [14], the dielectric constant of Si₂N₂O ceramics was stable overall.

IV. Conclusions

Porous Si₂N₂O wave-transparent ceramics were prepared using Li₂CO₃ additive and vacuum sintering. The phase composition, microstructure, mechanical and dielectric properties can be tailored via directly adjusting the Li₂CO₃ amount. It was found that the generated Si₂N₂O became the main phase when the additive content was above 1 wt.%. The room temperature mechanical properties increased with the increase of additive content. The oxidation resistance and the thermal shock resistance were better when the additive content was 2 wt.%. The highest critical temperature difference (ΔTc) could reach 1300 °C (residual strength ratio >70%). Furthermore, the dielectric properties were excellent (ε = 3.13–5.12, tan δ = 0.00268–0.00422), which can be attributed to the porosity, Si₂N₂O phase and formed SiO₂ by oxidation. In conclusion, the porous Si₂N₂O ceramics with good mechanical properties and low dielectric properties have excellent potential for application in microwave sintering furnace.

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