Effects of different kinds of sillimanite minerals on the properties of mullite ceramic foams

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Abstract
In order to improve the mechanical properties of mullite ceramic foams, vacuum infiltration with different kinds of sillimanite minerals slurries (andalusite, sillimanite and kyanite) was used to prepare mullite ceramic foams. The effects of the kinds of sillimanite minerals on the mechanical properties, phase compositions, thermal shock resistance of the as-prepared mullite ceramic foams were systematically investigated. The results showed that the compressive strength and thermal shock resistance of mullite ceramic foams prepared with andalusite slurry were higher than the mullite ceramic foams prepared with kyanite slurry and sillimanite slurry. However, the apparent porosity and linear expansion of mullite ceramic foams with andalusite was lower than the mullite ceramic foams with kyanite because of the difference in decomposition temperature and decomposition rate of andalusite, sillimanite and kyanite. In addition, when the calcination temperature reached 1400 °C, it was beneficial to promoting the growth of mullite grains that andalusite slurries were used as infiltration slurries to produce ceramic foams. Furthermore, after calcination, the low thermal stresses were generated within the struts of mullite ceramic foams with andalusite, which positively influenced the thermal shock resistance and strength of the mullite ceramic foams.

1. Introduction
Mullite ceramic foams were commercially available for a wide range of technological applications such as catalyst supports, filters and insulation materials because of high levels of inner geometric surface areas, high porosity, chemical stability and mechanical properties [1, 2]. Over the previous decades, common techniques for preparation of ceramic foams included gel-casting [3], pyrolysis of organic additives. [4], direct-foaming [5], freeze-drying [6] and polymer replication [7]. Notably, the polymer replication was one of the most versatile techniques for preparing mullite ceramic foams with low density and open three-dimensional network structures from polyurethane template [8]. However, the hollow struts with voids and defects were generated due to burnout of the polyurethane template during calcination process. This process caused a weakening of mechanical strength and thermal shock resistance of ceramic foams.

Currently, various of methods had been developed to optimize hollow struts, resulting in improving the mechanical properties of ceramic foams [9]. They could be classified into three types: optimization of slurry, pretreatment of template and treatment of ceramic foam preforms [10]. For instance, Voigt et al improved the structure of ceramic foam via adjustment of ceramic slurry rheological behavior [11]. Pu et al optimized the coherence between ceramic slurry and templates by a surface treatment of templates using silica gel [12]. Jun et al promoted the mechanical strength of ceramic foam by using carbon coated polymeric sponge [13]. Zhu et al
used recoating approach to increase the struts thickness of ceramic foams and repair the flaws within struts [14]. Chen et al. had successfully prepared ceramic foams with excellent mechanical properties through in situ synthesis of whiskers [15]. Moreover, it had been illustrated in the literature that vacuum infiltration was an effective method to improve the mechanical strength and thermal shock resistance of ceramic foams by filling up the hollow struts and repairing the flaws [16].

In recent years, preparation of mullite ceramic foams from fly ash had been drawn much more attention due to its environment friendly features, which could promote the comprehensive utilization of resources [17]. However, it had a negative influence on the mechanical properties of the products that a lot of SiO₂ amorphous glass phase was abundant in fly ash [18]. In order to overcome the above problem, Zhu and co-workers reported that adding alumina powders improved the compressive strength of mullite porous ceramics produced from fly ash [19]. It had also been illustrated that adding andalusite could effectively optimize the mechanical properties of ceramic foams prepared with fly ash, owing to the decomposition of andalusite. Meanwhile, sillimanite and kyanite could also decompose to mullite and SiO₂ during calcination process, which improved the mechanical strength and microstructure of products [20, 21]. However, to the best of author’s knowledge, there were few reports in the literature investigating the effect of different kinds of clay slurries (andalusite, sillimanite and kyanite) on the properties of mullite ceramic foams.

Therefore, in this work, vacuum infiltration with different kinds of sillimanite minerals slurries (andalusite, sillimanite and kyanite) was used to prepare mullite ceramic foams. The effects of the different kinds of sillimanite minerals on the mechanical properties and thermal shock resistance of the mullite ceramic foams were studied. In addition, the phase compositions and microstructure of the mullite ceramic foams prepared with different kinds of sillimanite minerals were also studied. Finally, the thermal stresses within the skeleton of these materials were studied by finite element method.

2. Experimental procedure

2.1. Mullite ceramic foams preparation

The mullite ceramic foams were prepared by the replication with matrix slurry, followed by vacuum infiltration of infiltration clay slurries. The chemical compositions of raw materials were shown in Table 1. The raw materials of matrix slurry and infiltration clay slurries were shown in Table 2. Polyurethane foams (20 ppi) were impregnated with the matrix slurry and dried at room temperature, followed by heating at 750 ˚C to prepare mullite preforms. Then the as-prepared mullite preforms were immersed into infiltration slurry and a vacuum

| Table 1. The chemical compositions of raw materials. |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Raw materials   | SiO₂            | Al₂O₃           | Fe₂O₃           | CaO             | TiO₂            |
| Fly ash         | 45.50           | 40.35           | 3.69            | 4.94            | 1.31            |
| Andalusite      | 38.70           | 57.43           | /               | 0.15            | /               |
| Kyanite         | 41.84           | 50.68           | 0.40            | 0.20            | 1.41            |
| Sillimanite     | 38.92           | 58.91           | 0.94            | 0.79            | 0.15            |
| Kaolin clay     | 45.65           | 33.09           | 0.76            | 0.04            | 0.33            |
| Alumina         | ≤0.15           | ≥99.4           | ≤0.1            | /               | /               |

| Table 2. The formulations of mullite ceramic foams. |
|-----------------|-----------------|-----------------|-----------------|-----------------|
| Raw materials   | Matrix slurry   | Infiltration slurry-Si | Infiltration slurry-An | Infiltration slurry-Ky |
| Alumina         | 50              | 33.91           | 33.62           | 38.28           |
| Fly ash         | 20              | 20              | 20              | 20              |
| Kaolin clay     | 10              | 20              | 20              | 20              |
| Andalusite      | 20              | 46.30           | 46.38           | 46.30           |
| Sillimanite     | /               | 46.09           | 41.72           | 41.72           |
| Kyanite         | /               |                  |                  |                  |
| Polycarboxylates (dispersant) | ±1.1   | ±1.1          | ±1.1          | ±1.1          |
| Calcium lignosulfonate (binder)  | ±3.5  | ±3.5         | ±3.5         | ±3.5         |
| Carboxymethyl cellulose | ±1   | ±1           | ±1           | ±1           |
| Solid content   | 75              | 72              | 72              | 72              |
of 0.5 Pa was applied for 10 min. The mullite preforms were dried at 100 °C for 24 h and heated at different temperatures (1325 °C, 1400 °C, 1475 °C, and 1550 °C) to obtain mullite ceramic foams.

2.2. Characterization techniques
The bulk density of the ceramic foams specimen was calculated from their mass-to-volume ratios. The ceramic foam apparent porosity was measured by Archimedes method using distilled water as liquid media. The ceramic foam length was measured before and after heating to characterize the linear shrinkage. The thermal shock resistance was evaluated by the water quenching technique [22]. The compressive strength and residual compressive strength of ceramic foam were tested using a hydraulic universal testing machine (ETM304C, Wance, China) at a crosshead speed of 0.5 mm min⁻¹ (ASTM standard C36/C365M-05). The ceramic foam thermal shock resistance was determined from the residual strength ratio (compressive strength/residual compressive strength). In addition, the microstructure of ceramic foam was characterized by field-emission scanning electron microscopy (FESEM, Quanta 400; FEI Company, Hillsboro, OR, USA). The phase compositions of the RPCs were determined by x-ray diffraction (XRD, X’Pert PRO, Philips, Netherlands). Furthermore, the thermal stress distribution within the ceramic struts was investigated by the finite element method.

3. Results and discussions
3.1. The mechanical properties of mullite ceramic foams
The effects of heating temperature and kinds of clay on the mechanical properties of mullite ceramic foams are represented in Figure 1. When the heating temperature was below 1400 °C, the bulk density and linear shrinkage of the mullite ceramic foams increased marginally with an increase in the heating temperature. There were two reasons: on one hand, andalusite, sillimanite and kyanite could decompose to amorphous SiO₂ and mullite at high temperature, resulting in the volume expansion of the ceramic foams [23]. On the other hand, mullite grains were also generated by the secondary mullitization, which also led to expanding the ceramic foams volume. However, when the heating temperature was higher than 1400 °C, it revealed a great increase in the bulk density, linear shrinkage and compressive strength with increasing of the heating temperature. But the apparent
Ceramic foams had the following form: the residual compressive strength of the ceramic foams not only because of the degree of mullitization in ceramic foams but also because of the difference in thermal strength ratio of the ceramic foams that the andalusite slurry was used as in °C. According to the equation capacities. As mentioned above, the thermal expansion coefficient of the ceramic foams specimens, those heated at 1325 °C were the most suitable products for industrial applications. During the decomposition, the three minerals displayed a volume expansion that was very large for kyanite, relatively small for andalusite and sillimanite. The compressive strength of the ceramic foams-An was larger than the ceramic foams-An and Ky. However, when the heating temperature reached 1550 °C the compressive strength of the ceramic foams-Si exhibited the highest value among the as-prepared ceramic foams. This was because the decomposition temperature of sillimanite was between 1500 °C and 1550 °C. Therefore, a lot of mullite grains were formed in the ceramic foam-Si at 1550 °C, which was beneficial to improving the mechanical strength of the ceramic foams.

Table 3. The thermal shock resistance of different mullite ceramic foams.

| Specimens         | Compressive strength | Residual compressive strength | Residual strength ratio |
|-------------------|----------------------|-------------------------------|-------------------------|
| Ceramic foams-Si  | 2.174MPa             | 0.939Mpa                      | 43.2%                   |
| Ceramic foams-An  | 2.591Mpa             | 1.293Mpa                      | 49.9%                   |
| Ceramic foams-Ky  | 1.698Mpa             | 0.513Mpa                      | 31.9%                   |

The porosity decreased gradually. These phenomena were because the driving force of sintering was promoted at high temperature. Meanwhile, the nucleation and growth of mullite was accelerated, which positively affected the compressive strength of the ceramic foams [24]. During heating process a mass of liquid glassy phase with low viscosity was generated, and could fill the pores within the ceramic struts, which led to reducing the porosity of the ceramic foams. The main reasons for this phenomenon was that alkali metal oxide, alkaline earth metal oxide and impurities in fly ash, kaolin clay and sillimanite minerals could produce low melting glass with low viscosity at high-temperature [25].

It could be observed that the linear shrinkage and bulk density of the mullite ceramic foam prepared with infiltration slurry-Ky(ceramic foams-Ky) were less than those of other mullite ceramic foams at the same heating temperature, because the decomposition characteristics were different for the three sillimanite minerals. Sillimanite decomposed at the highest temperature, while kyanite did so at the lowest temperature [26]. During their decomposition, the three minerals displayed a volume expansion that was very large for kyanite, relatively small for andalusite and sillimanite [23]. The compressive strength of the ceramic foams-An was larger than the ceramic foams-An and Ky. However, when the heating temperature reached 1550 °C the compressive strength of the ceramic foams-Si exhibited the highest value among the as-prepared ceramic foams. This was because the decomposition temperature of sillimanite was between 1500 °C and 1550 °C. Therefore, a lot of mullite grains were formed in the ceramic foam-Si at 1550 °C, which was beneficial to improving the mechanical strength of the ceramic foams.

3.2. The thermal shock resistance of mullite ceramic foams

According to the mechanical properties analysis, the linear shrinkage and bulk density of the ceramic foams heated at 1550 °C and 1475 °C were too large to ensure product stability and the mechanical strength of the products heated at 1325 °C was not sufficiently high to ensure product quality. Therefore, among the mullite ceramic foams specimens, those heated at 1400 °C were the most suitable products for industrial applications.

The residual compressive strength and thermal shock resistance of the as-prepared ceramic foams heated at 1400 °C are shown in Table 3. Interestingly, it was beneficial to promoting the residual strength and residual strength ratio of the ceramic foams that the andalusite slurry was used as infiltration slurry. This phenomenon was not only because of the degree of Mullitization in ceramic foams but also because of the difference in thermal expansion coefficient between the ceramic foams-Ky, An and Si. The expression of thermal shock resistance of ceramic foams had the following form:

\[ R \propto \sigma \sqrt{\frac{\lambda}{C_p \rho}} / \alpha E \]  

Where R is the thermal shock stability coefficient, \( \sigma \) is the tensile strength, \( \lambda \) is the thermal conductivity, \( \rho \) is the density, \( \alpha \) is the thermal expansion coefficient, \( E \) is the Young’s modulus and \( C_p \) is the constant-pressure heat capacities. As mentioned above, the thermal expansion coefficient of andalusite was lower than other sillimanite minerals. According to the equation (1), as the thermal expansion coefficient decreased, the thermal shock resistance increased gradually. Therefore, using infiltration slurry-An to treat ceramic foams could improve the thermal shock resistance of the products. The thermal conductivity significantly influenced on the thermal stresses within ceramic struts, resulting in affecting the thermal shock resistance of the ceramic foams [27, 28]. The ceramic struts with multi-layered were obtained by using the vacuum infiltration process Figure 2. Owing to the difference in the thermal expansion and thermal conductivity for the matrix and coating layer, the thermal stresses could be found within the ceramic struts during thermal shock process [29]. It was clearly observed that the large thermal stresses were formed at the surface and edge of ceramic struts Figure 3. Particularly, the maximum thermal stresses were located at edge of the ceramic foams-Ky, which had a negative influence on the thermal shock resistance of the ceramic foams. This was attributed to the fact that a large amount of mullite grains and pores were formed within the ceramic struts of mullite ceramic foams-Ky, which led to decreasing the thermal conductivity of the coating layer. However, the minimum thermal stresses were formed within the ceramic foams-An, which was beneficial to improve the thermal shock resistance of the ceramic foams.
3.3. The phase compositions of mullite ceramic foams

Figure 4 reveals the XRD patterns comparison of the different ceramic foams. It was found that for the ceramic foams, corundum, quartz and mullite phases were obtained. Owing to the fact that kyanite decomposed at the lowest temperature among the three sillimanite minerals, a large amount of mullite and SiO₂ were generated in Figure 2.

Figure 2. Schematic example for multi-layered ceramic struts.

Figure 3. The thermal stresses distribution of the multi-layered struts of different mullite ceramic foam during thermal shock process (a) ceramic foams with-Si, (b) ceramic foams with-An and (c) ceramic foams with-Ky.

Figure 4. The XRD pattern of different mullite ceramic foams heated at 1400 °C. (A) ceramic foams-Si, (B) ceramic foams-An and (C) ceramic foams-Ky.

3.3. The phase compositions of mullite ceramic foams

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the ceramic foams-Ky by the decomposition of kyanite and the secondary mullitization. The decomposition temperature of andalusite was between the kyanite decomposition temperature and the sillimanite decomposition temperature. Thus, the content of mullite in the ceramic foams-An were also between the ceramic foams-Ky and the ceramic foams-Si. The content of mullite, corundum and quartz in the coating layer had a great influence on the thermal conductivity and thermal expansion of the coating layer, resulting in affecting not only the thermal stresses within the ceramic struts but also the residual stresses within the ceramic struts. The residual stresses within the multi-layered struts were determined according to equation (2).

\[
\sigma_R = \left( E_i t_i \int_{T_o}^{T} (\alpha_i - \alpha_o) dT \right) / \left[ t_i E_i (1 - \nu_o) / E_o + 2t_o (1 - \nu_i) \right]
\]  

(2)

where \( \nu \) is Poisson’s ratio, \( \alpha \) is the thermal expansion coefficient, \( E \) is Young’s modulus, and \( t \) is the layer thickness; \( T_o \) and \( T \) are 1400 °C and 25 °C, respectively; and the subscripts \( i \) and \( o \) stand for the matrix and coating layers, respectively. Based on the above reasons and equation, due to the generation of appropriate amount of mullite phases in the coating layer of the ceramic foams-An, the thermal expansion of coating layer matched with the matrix. Therefore, for the ceramic foam-An, the appropriate residual stresses were formed in the coating layer and the matrix, which positively influenced on the mechanical properties and the thermal shock resistance.

Figure 5. The microstructure of different mullite ceramic foams before thermal shock test.
3.4. The microstructure of mullite ceramic foams

Figure 5 represents the morphology of the different ceramic foams fractured surfaces, revealing that a complete infiltration of the ceramic struts could be achieved by the vacuum infiltration with different infiltration slurries. Owing the decomposition of three sillimanite minerals, pores were generated in the coating layer of the ceramic foams. Notably, the number of pores in the coating layer of the ceramic foams-Ky was much more than the other ceramic foams, which was attributed to the low decomposition temperature of kyanite and the large volume expansion caused by the kyanite decomposition. The amount of pores greatly influenced the physical properties of coating layer, such as mechanical strength, bulk density, Young’s modulus and thermal conductivity. Therefore, when the kyanite slurries were used as infiltration slurries to treat the ceramic foams, the thermal conductivity of coating layer of the ceramic foams decreased significantly, which led to increasing the thermal stresses within the ceramic struts during thermal shock test Figure 3. After thermal shock test, a lot of micro-cracks were generated in the coating layer of the ceramic foams-Ky (Figure 6). However, there were micro-cracks in the ceramic foams-An. The appropriate residual stresses within the ceramic struts of the ceramic foam-An could effectively restrain the micro-cracks propagation. During thermal shock process the low thermal stresses were formed in the ceramic struts of the ceramic foam-An, which prevented the nucleation and propagation of micro-cracks in the ceramic struts. Moreover, the mullite grains produced by the decomposition of andalusite and the secondary mullitization could also prevented the nucleation and propagation of micro-cracks, resulting in improving the mechanical strength and thermal shock resistance of the ceramic foams.

4. Conclusion

The mechanical properties and thermal shock resistance of mullite ceramic foams were improved by optimizing the kinds of sillimanite minerals in infiltration slurry. When the sillimanite slurries and andalusite slurries were used as infiltration slurries to treat ceramic foams the mullite ceramic foams (heated at 1400 °C) exhibited poor mechanical strength and thermal shock resistance. However, the mechanical strength and residual strength ratio of the mullite ceramic foams treated with andalusite infiltration slurries reached 2.591Mpa and 49.9% respectively. Therefore, it was beneficial to improving the mechanical properties of the mullite ceramic foams that the andalusite infiltration slurries were used to treat the mullite ceramic foams.

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