Effects of ammonium molybdate additive and sintering temperature on the properties of foam ceramics based on ceramic tile polishing waste

Cong WANG¹, Weiwei CHEN¹, Anze SHUI¹,³, Jianqiao LI¹, Wei TIAN¹, Juan MA¹, Bin DU¹ and Satoshi TANAKA²

¹School of Materials Science and Engineering, South China University of Technology, Guangzhou 510641, China
²Department of Materials Science and Technology, Nagaoka University of Technology, Nagaoka, Niigata 9402188, Japan

In this study, the foam ceramics based on ceramic tile polishing waste (CTPW) were successfully prepared by a simple solid phase sintering method. The CTPW was used as the main raw material, SiC inside CTPW as the foam agent and (NH₄)₆Mo₇O₂₄ as the sintering additive. Effects of the amounts addition of (NH₄)₆Mo₇O₂₄ (0–6 wt %) and sintering temperature (1080–1200°C) on the sintering properties, structural evolution, phase composition and mechanical properties have been investigated. During the sintering process, the SiC inside CTPW could react with oxygen and then produce the gases such as CO and CO₂, which caused a closed-pore structure. After adding (NH₄)₆Mo₇O₂₄ and increasing the sintering temperature, a better porous structure with suitable pore size and high compressive strength could be obtained. It was found that the foam ceramics doped with 2 wt % (NH₄)₆Mo₇O₂₄ and sintered at 1200°C for 30 min showed excellent properties: a low bulk density (0.362 g/cm³), the appropriate pore size (0.94 mm), a uniform pore size distribution and a very high compressive strength (8.16 MPa).

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1. Introduction

Porcelain tiles are widely applied in building materials due to outstanding properties such as high mechanical strength, wear and chemical resistance. And their very compact structure, vitrified throughout and extremely low porosity make the materials stain and frost resistant.¹,² In recent years, an increasing production of porcelain tiles has taken place in China according to a government report, which occupies more than half of overall production around the world. However, the porcelain tiles usually need to undergo the process of surface polishing to enhancing the aesthetical appearance of products. During this process, a relevant material with about 0.4–0.8 mm in thickness is removed from the tile surface, which creates a gloss surface level of 65–70%.³,⁴ As a result, it will generate a large amount of ceramic tile polishing waste (CTPW). It reported that the output of CTPW has reached about 6 million tons per year in China and it increases every year.⁵ Commonly, the CTPW is collected and temporarily stored in effluent treatment stations. After removing the residual water and then it is generally disposed in landfill sites. However, the landfill treatment process not only occupies the massive land, but also wastes the mineral resource.⁶ Thus, the recycling of CTPW is very important.

Recently, many researches have devoted their attention to recycling CTPW. Actually, the CTPW, generated by polishing process, mainly contains silica (SiO₂), aluminum oxide (Al₂O₃) and bits of Fe₂O₃, TiO₂, K₂O, Na₂O, CaO and MgO, which is similar to the ceramic material composition.⁵,⁷ For this reason, some reports indicated that it is feasible to use the CTPW to replace feldspar in ceramic industry or to manufacture traditional ceramic tiles.⁸–¹⁰ In addition, there are some other studies on the recycling of CTPW in concrete production to decrease the cost of production.⁵,¹¹,¹² However, the CTPW usually contains about 1–5 wt % silicon carbide (SiC) that derived from polishing tools. The SiC will react with oxygen and release gases such as CO and CO₂ in a sintering temperature (>1100°C) for the production of ceramic tiles. In this case, there are two problems with using the CTPW as a raw material for manufacturing traditional ceramic tiles: firstly, the generation of gas will hinder the process of sintering densification; secondly, the utilization rate of the CTPW is very low. Therefore, most studies have focused on preparing the porous or foam ceramics by using the CTPW as raw materials.⁵,¹³–¹⁶ At present, foamed ceramic is one of the most promising building insulation materials for its excellent advantages of chemically stable, low density, long-time stable in physical characteristics and water...
resistance. Moreover, the foam ceramic as an insulation material is produced by using the CTPW as the raw material, which could reduce the environment pollution and lower the cost of production. Most studies on preparing foam ceramics with CTPW needed to add additional foaming agent like SiC, carbon ash or others. It was reported that the CTPW usually contained about 1–5 wt% SiC. The SiC inside CTPW may act as the foaming agent. However, the oxidation reaction of SiC inside the CTPW are closely related to the production and content of liquid phase. As shown in Fig. 1, the SiO2 envelope layer generally formed in the surface of SiC, which prevented the contact of SiC and O2. And the diffusion rate of O2 in the SiO2 envelope layer was only \(10^{-14} - 10^{-15}\) cm²/s. In order to increase the O2 diffusion rate and enable more O2 react with SiC, the destruction of SiO2 envelope layer is very necessary. The use of the sintering aid is essential for getting the liquid phase in a lower sintering temperature in this study. Recently, the molybdenum oxides (MoO₃) have been used as one of sintering aid for reducing the sintering temperature in dielectric or electrolyte ceramics and porous ceramics. In optical glassy materials, MoO₃ can also serve as a network former and a network modifier in the presence of glass formers such as TeO₂, P₂O₅ and B₂O₃ based glasses. Moreover, some other researchers also reported that MoO₃ addition cannot only lower the mullitization temperature but also be used as a catalyst to promote the formation of mullite whiskers. Therefore, in this study, the foam ceramics were prepared by using the CTPW as main material, not adding any additional foaming agent but only making the most of SiC in the CTPW. The ammonium molybdate \([(NH₄)₆Mo₇O₄₄\] was an important raw material for production and preparation of MoO₃. The MoO₃ was achieved by the thermal decomposition of \((NH₄)₆Mo₇O₄₄\) and was utilized as sintering aid and a catalyst in the sintering process, which caused the formation of liquid glassy phase at a lower temperature and promoted the production of mullite. Thus, the porous structure of the ceramic with good mechanical properties could be obtained. The foam ceramics with various amounts of \((NH₄)₆Mo₇O₄₄\) ranging from 0 to 6 wt% were sintered at different temperature from 1080 to 1240°C for 30 min. The effects of the amounts addition of \((NH₄)₆Mo₇O₄₄\) and sintering temperature on the sintering properties, structural evolution, phase composition and mechanical properties of the foam ceramics were investigated in detail. The function mechanism of \((NH₄)₆Mo₇O₄₄\) additive was also discussed.

2. Experimental

2.1 Materials and experiment process

2.1.1 Raw materials

In this study, CTPW (Oceano Ceramics Co. Ltd., Guangdong, China) was the main raw material. The CTPW was first dried in drying oven at 100°C for 10 h, and then dry-ball-milled in a planetary ball mill (QM-ISPM4-CL, Instrument Plant of Nanjing University, China) for 2 h using alumina balls. The resulting powders were ground in an agate mortar and sieved through a 40-mesh sieve. The chemical composition of CTPW was shown in Table 1 and the phase composition of CTPW was seen in Fig. 2. It can be seen that the composition of CTPW was as follows: SiO₂ 71.64 wt%, Al₂O₃ 17.12 wt%, Fe₂O₃ 0.56 wt%, TiO₂ 0.32 wt%, CaO 0.73 wt%, MgO 1.28 wt%, K₂O 1.75 wt%, and Na₂O 3.23 wt%. The main phase in CTPW was quartz. There were also a few other phases such as amorphous phase, mullite and silicon carbide.

2.1.2 Preparation of foam ceramics

In this study, the ceramic tile polishing waste (as the main raw material) mixed with different content of \((NH₄)₆Mo₇O₄₄\) were wet-ball-milled in a planetary ball mill (the weight ratio of

| Table 1. The chemistry composition of CTPW (wt %) |
|-----------------------------------------------|
| wt%  | SiO₂ | Al₂O₃ | Fe₂O₃ | CaO | TiO₂ | MgO | K₂O | Na₂O |
| CTPW | 71.64 | 17.12 | 0.56  | 0.73 | 0.32 | 1.28| 1.75| 3.23 |

Fig. 1. The schematic illustration for oxidation reaction of SiC.
liquid/powders = 3) for 2 h with a constant speed of 400 rpm and then dried at 100°C for 10 h. The specific content of each sample was listed in **Table 2**. Later, the resulting powders were ground in an agate mortar and sieved through a 60-mesh sieve. The green bodies with 25 mm in diameter and 8 mm in thickness were formed by dry-pressing the compound powders at 15 MPa. Finally, the green bodies were fired in an electric furnace in air atmosphere at 1080–1240°C for 30 min at a heating rate of 5 °C/min.

### 2.2 Test and characterization

The composition of the ceramic tile polishing waste powders was measured with a silicate chemical composition analysis instrument (DHF86, Xiangtan Songshan instrument Co. Ltd., China). The phase identification of the sintering specimens was performed by an X-ray diffractometer (XRD; X’Pert Pro, PANalytical Co., Holland) using Cu Kα radiation at room temperature over a 2θ angle scan range of 5 to 80°. The microstructure of the sintered specimens was analyzed by scanning electron microscopy (SEM; Model EVO18, ZEISS, Germany). The macrostructure of the sintered specimens was analyzed by stereomicroscope (SZX 10-3121, OLYMPUS, Japan). The image analysis software (Image-Pro Plus6.0, Media Cybernetics, USA) was used to determine the pore size of the fired body. The bulk density and open porosity of the sintered samples were measured according to the ASTM C373-88. The compressive strength was measured by using a universal electronic tester (CMT4304) with load rate of 0.5 mm/min. The final result represented the average of 5 individual tests. Thermal behavior was examined by a thermogravimetry-differential scanning calorimetry instrument (TG-DSC; NETZSCH STA449C, Germany), and was carried out in Al2O3 crucibles at a heating rate of 10 °C/min in air and reached a final temperature of 1280°C.

### 3. Results and discussion

#### 3.1 Thermal analysis

**Figure 3** shows the TG-DSC curves of the samples S0 and S2 from 40 to 1280°C in air atmosphere. For two samples, there existed three endothermic peaks, respectively. The endothermic peaks at around 574°C in two samples was attributed to the crystal transformation (β-quartz to α-quartz). For sample S0 [as shown in Fig. 3(a)], the weight loss of 2.38% in 40–1049°C was due to the loss of water and the decomposition of impurities correlating with the second endothermic peak at 647°C. For sample S2 [as shown in Fig. 3(b)], the weight loss of 2.17% in 40–1000°C was related to the loss of water, the decomposition of (NH4)6Mo7O24 correlating with the first endothermic peak at 315°C and the decomposition of impurities, respectively.\(^{(30)}\) Besides, it could be seen that there

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**Table 2.** The weight ration of samples (wt %)

| Sample No. | Raw material   | CTPW | (NH₄)₆Mo₇O₂₄ |
|------------|----------------|------|--------------|
| S0         | 100            | 0    | 0            |
| S1         | 99             | 1    | 0            |
| S2         | 98             | 2    | 1            |
| S4         | 96             | 4    | 1            |
| S6         | 94             | 6    | 2            |

**Fig. 2.** The XRD pattern of CTPW.

**Fig. 3.** The TG-DSC results of samples: (a) S0 and (b) S2 sintered at air atmosphere.
was a slight weight increment of 0.11% from 1049 to 1280°C in sample S0 and 0.15% from 1000 to 1280°C in sample S2, which was caused by the oxidation of SiC. The last pronounced endothermic peak appeared at 1167°C for the sample S0 while at 1145°C for the sample S2, which was associated to the formation of liquid phase.\(^{16,31}\)

There were some significant differences in two samples because of adding \((\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\). The weight loss in sample S2 was less than that in sample S0, which indicated that the sublimation of MoO\(_3\) obtained from decomposition of \((\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\) was not happened and may be restrained by the existence of CaO or CaCO\(_3\) in CTPW.\(^{32,33}\) In addition, the initial temperature of weight increment (about 1000°C) that may be the starting temperature of SiC oxidation reaction in sample S2 were lower than that in sample S0. This variation showed that the MoO\(_3\) (like alkali metal oxide or alkali earth metal oxide) could promote the formation of liquid phase in a lower temperature.\(^{24}\)

### 3.2 Sintering properties

**Figure 4** shows the effects of the addition amounts of \((\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\) on the bulk density of sample S0 to S6 sintered at different temperature for 30 min. for sample S0 with no \((\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\) addition, the bulk density showed a lower value compared to sample S1 to S6 with \((\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\) addition. The reason of this phenomenon was the low oxidation reaction rate of SiC in CTPW, which caused less gases production. Thus, adding the sintering aid of \((\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\) was necessary for obtaining foam ceramic with low bulk density. In the temperature of 1080°C, it was clearly found that the bulk density of the sample (S0–S4) gradually decreased from 1.97 to 1.13 g/cm\(^3\) with the amounts of \((\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\) increasing from 0 to 4 wt% but showed a slight rise in the sample S6 (1.16 g/cm\(^3\)). It was worthy to note that there existed a similar tendency when the temperature increased from 1120 to 1200°C. However, the bulk density of the sample S2 to S6 was almost nothing changed. This trend illustrated that the moderate addition of \((\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\) was necessary for obtaining foam ceramic with low bulk density. In the temperature of 1240°C, the bulk density of the sample S2 to S6 increased gradually and showed a great value compared to former sintering temperature. The reason for these variations may be that the viscosity of silicate liquid was relatively low at excessive temperature after adding the \((\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\). A part of pores in CTPW may fracture and connect with each other under this viscosity. Then a small number of open pores could be obtained, leading to the increase of bulk density. Therefore, the \((\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\) addition of 2 wt% and sintering temperature of 1200°C was most suitable, which showed the lowest value of 0.362 g/cm\(^3\).

The effect of sintering temperature on samples was further researched. The results were shown in Figs. 5(a) and 5(b), and it was indicated the bulk densities and open porosities of sample S0 and S2 sintered at a temperature.
range from 800 to 1240°C. In Fig. 5(a), a first rises and then falls trend could be seen in both sample S0 and S2. However, from Fig. 5(b), it can be seen that the open porosities of sample S0 and S2 changed in the opposite trend. Thus, the sintering process could be divided into two stages. The first stage was sintering densification process that was from 800 to 1080°C for sample S0 and from 800 to 1040°C for sample S2. At this stage, the densification degree of samples increased with more generation of silicate liquid phase, but the bulk densities of sample S2 were all greater than sample S0 before reaching the maximum value. Moreover, the bulk density of sample S2 achieved a maximum value in advance compared to sample S0. Similarly, the open porosities of sample S2 were all less than sample S0 before reaching a lower value in advance. This phenomenon could be explained by the addition of (NH₄)₆Mo₇O₂₄ which could promote the formation of silicate liquid phase in a lower temperature. The increase of liquid phase further enhanced the densification process by filling the clearance between solid grains. In the second stage, the value of bulk density sharply dropped in two samples, but the value of open porosity only showed a slight increase in two samples. The reason for this was due to the more generation of silicate liquid phase with the sintering temperature increased, and then the SiO₂ protective layer of SiC was destroyed. The SiC inside CTPW could react with O₂, which caused the gas of CO₂ or CO released. These gases fixed in the liquid phase caused the formation of closed pores and prevent to the densification of samples. Furthermore, these closed pores began to grow because the pressure inside pores increased with the increasing temperature. Thus, the bulk densities got a decrease at the same time but the open porosities changed a little and had little difference in two samples. However, the values of bulk density in sample S2 were all less than sample S0. This difference between S0 and S2 was also due to the addition of (NH₄)₆Mo₇O₂₄. The oxidation of SiC was mainly depended on amount of liquid phase in the sintering process of CTPW. The effect of MoO₃ on the generation of liquid phase was obvious in first stage, so it was also more effective at higher temperatures. Therefore, more gases released and then the closed pores might burst and connect with each other [as seen in Fig. 8(e)] that was caused by the more oxidation of SiC, which caused a lower value of bulk density and a slight increase of open porosities in the subsequent sintering temperature. Similarly, the slight increase of open porosities in sample S0 might be caused by the burst and connection of closed pores in higher temperature.

3.3 Structural observations

Figure 6 shows optical images of cross section of sample S0 to S6 fired at 1200°C for 30 min with different amounts of (NH₄)₆Mo₇O₂₄. It can be seen that the porous structure or foam structure and many closed pores existed in all samples because of the oxidation reaction of SiC and generation of gas (CO and CO₂). The distribution of pore size, especially in sample S1 and S2, was relative uniform with adding the (NH₄)₆Mo₇O₂₄. Moreover, it was obviously found that the average pore size increased a lot (from 0.58 to 1.21 mm, as observed in Fig. 7) and the pore wall increased as well with increasing the amounts of (NH₄)₆Mo₇O₂₄. These results were probably due to that the MoO₃, decomposed from (NH₄)₆Mo₇O₂₄, caused the formation of liquid glassy phase at a lower temperature and reduced the viscosity of the liquid phase, which could lead to a more oxidation reaction of SiC and accelerated the diffusion of gas. While the excess of liquid phase gen-

![Fig. 6](image_url)  The optical images of cross section of samples fired at 1200°C for 30 min with different (NH₄)₆Mo₇O₂₄ addition: (a) S0: 0 wt %, (b) S1: 1 wt %, (c) S2: 2 wt %, (d) S4: 4 wt %, (e) S6: 6 wt %.
erated with adding excessive (NH₄)₆Mo₇O₂₄, it resulted in greater pore size and pore wall, destruction of pores and increase of bulk density. Thus, adding the proper amounts of (NH₄)₆Mo₇O₂₄ allowed obtaining a lower bulk density, greater pore size and more homogeneous porous structure.

Figure 8 shows the optical images of cross section of the sample S2 sintered from 1080 to 1240°C for 30 min. It was evident in the images shown by Figs. 8(a)–8(e) that these samples had a porous structure in all sintering temperature, even in a low temperature 1080°C but the difference could be found in the pore size. As shown in Fig. 9, an obvious increase of pore size (from 0.08 to 2.15 mm) happened as the sintering temperature increased. The reason for this was that amount of liquid phase increased with the increasing sintering temperature, and this could destroy the SiO₂ protective layer. The gas caused by the oxidation reaction of SiC would stay in the liquid phase, which could exert a pressure (Pg) on the pore wall. The pressure (Pg) inside pores increased with the increasing temperature, and the pores began to grow at the same time. However, the pore could burst and connect with each other in a higher temperature [as seen in Fig. 6(e)], and it caused a greater pore size and an increase of bulk density. Thus, the optimum sintering temperature was 1200°C.

Figure 10 shows the SEM images of cross section of sample S2 fired at 1200°C for 30 min. Figure 10(a) indicated that the microstructure of the sample and the microstructure of the inside pore wall was seen in Fig. 10(b). There existed a homogeneous porous structure and contained some small pores in pore wall or inside pore. From Fig. 10(b), it can be seen that some whiskers formed inside pore wall after magnification. The variation was due to that
adding (NH₄)₆Mo₇O₂₄, as a catalyst, could motivate in situ formation of whiskers.²⁹ In addition, these whiskers could be the mullite (as observed in Figs. 11 and 12).

### 3.4 XRD analyses

The XRD results of sample S0 to S6 fired at 1200°C for 30 min with different (NH₄)₆Mo₇O₂₄ addition (sample S0 to S6) were shown in Fig. 11. The shape of the peaks demonstrated that all the samples consisted of crystalline and amorphous phase corresponding to a board background. It was clearly found that the main crystalline phases were quartz and cordierite in sample doped without (NH₄)₆Mo₇O₂₄, meanwhile a few weak peaks of mullite, silicon carbide and tridymite can be also seen. After 1 wt% (NH₄)₆Mo₇O₂₄ was introduced, the diffraction intensities of quartz and silicon carbide decreased obviously, and the intensities of mullite and cordierite increased. When the addition amount of (NH₄)₆Mo₇O₂₄ was further increased from 2 to 6 wt %, the obvious peak of quartz (2θ ≈ 20.5 and 26°) even disappeared and only phases of mullite and cordierite were observed. Based on the above analysis of these results, it was concluded that the addition of (NH₄)₆Mo₇O₂₄ played an important role in the sintering process of foam ceramics. The melting of MoO₃ could promote formation of mullite, which can enhance the mechanical properties (as shown in Fig. 13).

The XRD results of sample S2 sintered at different temperature for 30 min were presented in Fig. 12. Obviously, the XRD patterns of all samples consisted of a board background.
background peak corresponding to the amorphous phase. With increasing the sintering temperature from 1080 to 1160°C for 30 min, the main crystalline phases were quartz and cordierite as well as including a few phases of mullite, silicon carbide and tridymite. However, the diffraction intensities of quartz (20 ∼ 20.5, 26 and 27.8°) gradually decreased as the sintering temperature increased. When the sintering temperature further raised to 1200 and 1240°C, the peaks of quartz disappeared but the peaks of mullite became more obvious. Hence, increasing the sintering temperature was also favorable for the formation of mullite. Moreover, the phase of tridymite may come from the crystal transformation (α-quartz to β-tridymite). This transformation caused volume expansion of 16%. If this change happened in the SiO₂ protective layer of SiC, the oxidation reaction rate could be accelerated due to the SiC inside directly reacted with O₂.16)

3.5 Mechanical properties

The results obtained from measurements of compressive strength with the different contents of (NH₄)₆Mo₇O₂₄ were plotted in Fig. 13. These results showed that the compressive strength increased first and then decreased when the (NH₄)₆Mo₇O₂₄ was introduced. The highest value of 8.76 MPa was the sample S1. When the content of (NH₄)₆Mo₇O₂₄ increased up to 2 wt.%, the compressive strength of sample decreased slightly (8.16 MPa) because of a greater pore size. Even in the sample with 6 wt.% (NH₄)₆Mo₇O₂₄, the compressive strength still was 4.48 MPa. Moreover, the bulk density of these samples was all less than 0.5 g/cm³ (as shown in Fig. 4). The enhancement of compressive strength was associated with the formation of many mullite whiskers inside the pore wall (as observed in Figs. 10 and 11). In addition, the mechanical properties of foam ceramics were significantly influenced by the porous structure such as pore size and thickness of pore wall. The compressive strength decreased gradually of the sample S1 to S6, which resulted from the increasing of pore size. The effects of sintering temperature on the compressive strength was presented in Fig. 14. It was clearly seen that the compressive strength declined sharply with the temperature increasing from 1080 to 1120°C (from 82.17 to 18.94 MPa). After rising to a higher temperature, the downward trend of the compressive strength was slowing down. The changing tendency was contrary to the changes in average pore size (as shown in Fig. 9).

4. Conclusions

The foam ceramics were fabricated by a simple solid phase sintering method, using CTPW as the main raw material, SiC inside the CTPW as foam agent and the (NH₄)₆Mo₇O₂₄ as additive, which presented a typical closed-pore structure. The addition amounts of (NH₄)₆Mo₇O₂₄ and sintering temperature exerted an effect on the bulk density, open porosity, structure, pore size, phase composition and compressive strength of the foam ceramics. Adding (NH₄)₆Mo₇O₂₄ could decrease the viscosity of the liquid phase and accelerate the diffusion of gases generated by oxidation reaction of SiC, as well as promote the formation of mullite whiskers and then enhance the compressive strength. Increasing sintering temperature mainly caused a greater pore size and changed the phase composition in the foam ceramics. The foam ceramics doped with 2 wt.% (NH₄)₆Mo₇O₂₄ and fired at 1200°C for 30 min displayed excellent properties: a low bulk density (0.362 g/cm³), the appropriate pore size (0.94 mm), a uniform pore size distribution and a very high compressive strength (8.16 MPa). Such high-strength ceramics foam could well apply in the building thermal insulation materials. In addition, it is believed that this simple method will have important scientific value and economic benefits on direct recycling of large amounts of ceramic tile polishing waste in the word.

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