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The effect of sintering temperatures on alumina foam strength

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Abstract

An idea to improve the strength of ceramic foams by controlling the sintering temperature was proposed in this paper. The effect of sintering temperatures on alumina foams strength was investigated by discussing three factors: shrinkage, porosity and grain size, which changed with sintering temperatures. When the sintering temperature increased from 1400 to 1500 °C, the samples shrunk increasingly and the porosity decreased from 89.5 to 86.75%, which lead to the alumina foams strength increased from 0.27 MPa (at 1400 °C) to 0.627 MPa (at 1500 °C). When the sintering temperature increased from 1500 to 1550 °C, the porosity changed a little but the grains of samples grew up and contacted more tightly which resulted in the improvement of foam strength. When the sintering temperature is up to 1600 °C, a few grains preferentially grew too large compared to other grains, which caused a little decrease of strength. Therefore, to get strong alumina foams, the sintering temperature of 1550 °C was suggested in our work.

Keywords: A. Sintering; B. Porosity; B. Grain size; C. Strength; D. Al_2O_3

1. Introduction

Molten metal, particularly molten aluminum, in practice generally contains entrained solids which are deleterious to the final cast metal product. These entrained solids appear as inclusions in the final cast product after the molten metal is solidified and cause the final product to be less ductile or to have poor bright finishing and anodizing characteristics [1,2]. Therefore, it is naturally highly desirable to select a filter for use in removing or minimizing entrained solids from the final cast product, particularly when the aluminum is to be used in a decorative product. The common filter to cleaning molten aluminum is alumina foams which has some unique properties such as high porosity, low pressure drop and high resistance to molten aluminum attack [3]. The alumina foams are usually produced by the polymeric sponge method which involves the impregnation of polyurethane sponges with slurries containing ceramic particles and appropriate binders followed by pyrolysis and sintering at elevated temperature [4–6]. Alumina foams produced by this method are of low strength and fracture toughness as a result of large porosity and thin webs with holes in their center, making them sensitive to structural stress and limiting their structural application [7]. One of the most common ways to improve the mechanical properties of such foam is by the addition of fibers to the ceramic structure [8,9]. Typically, about 1–5 wt.% of fibers is added to the ceramic slurry to enhance its compressive strength up to 1–2 MPa [2] which is necessary to filter molten aluminum. However, uniformly impregnating polymeric sponges with the ceramic slurry containing fibers is difficult although various approaches such as applying a vacuum during infiltration and spraying the slurry onto the sponge have been used [6]. So a simple and efficient method to improve strength is urgently required.

Because sintering temperatures determine porosity and sintering degree which can affect the strength of ceramic foams, sintering temperatures have a significant effect on the foams strength. It is a pity that few literatures in our research pay more attention on the relationship of sintering temperatures and alumina foams strength. Some author sintered alumina foams at 1450 °C [10], others sintered them at 1650 °C [11]. The effect of sintering temperature on foam strength has not

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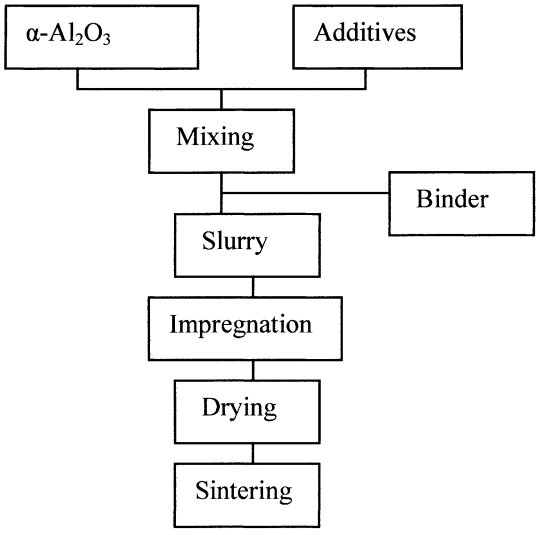

Fig. 1. Typical scheme of experimental procedure.

been investigated in detail. In this paper, an idea to improve the strength by controlling sintering temperatures was proposed. The effect of sintering temperatures on alumina foams strength was studied in detail. An alumina foams (about 1 mm diameter) with 1.3 MPa compressive strength was prepared in our experiment.

2. Experimental details

2.1. Foam preparation

α-Alumina (95 wt.%) and 5 wt.% additives (which consist of 2 wt.% TiO_2, 1 wt.% CaO and 3 wt.% others) were mixed by ball mill for 24 h and sieved by 60-mesh screen. Binder (5 wt.% PVA aqueous solution) was poured into the mixture while stirring to obtain well-dispersed slurry. The polymeric sponge with pore size of 12 ppi was immersed in the slurry and was compressed while submergence in order to fill all the pores. Subsequently, the impregnated sponge was removed from the slurry, and excess slurry was squeezed out of the sponge by means of pressing it with two parallel plates having a constant gap. Then the impregnated foams were aged in air for 12 h, dried at 80 °C for 24 h, and sintered to different temperatures in auto-controlled stove (LHT 04/17, Nabertherm, German). The typical scheme of this procedure was depicted in Fig. 1.

2.2. Property measurements

The linear shrinkage of samples during the course of heating was determined using the following equation:

$$\text{shrinkage} = (l_{g-} l_p)/l_g \times 100\%$$

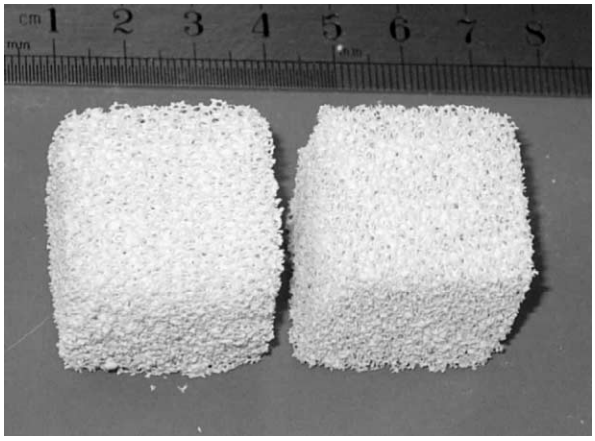

Fig. 2. Photos of alumina foams.

where l_g is the height of green disc and l_p the height of fired product. The height was measured by sliding gage.

The porosities of fired products were calculated by the following equation:

$$\text{porosity} = (\rho_r - \rho_b)/\rho_r$$

where ρ_r and ρ_b are the real and bulk densities respectively. Since the discs were rather uniform and flat, their bulk densities were simply calculated from their weights and volumes. As for the real densities, the discs were first ground to powder and then measured with a pyconmeter (Micromeitics 1305).

The compressive strength of fired product was measured using a universal testing machine (SJ-IA, made in China) fitted with flat steel plates closing with a circular head at the speed of 0.5 mm/min.

2.3. Characterization

X-ray diffraction (XRD) was carried out on samples after sintering to different final temperatures in air. A D/max –RB X-ray diffractometer (RIGAKU, Japan) with filtered CuKα radiation of wavelength 0.15418 nm was used. The voltage and current settings of the diffractometer were 40 kv and 30 mA, respectively. The scan angle ranges from 5° to 90° with a step size of 0.02° and a scan speed of 8°/min.

The microstructures of the fired samples were investigated by a JSM-6301F (JEOL, Japan) scanning electron microscope (SEM). The photo of samples was taken by digital camera (Casio, Japan) and shown in Fig. 2.

3. Results

The linear shrinkage of samples sintered to different temperatures(1400, 1500, 1550, 1600 °C) was shown in Fig. 3. With the increase of sintering temperatures, the

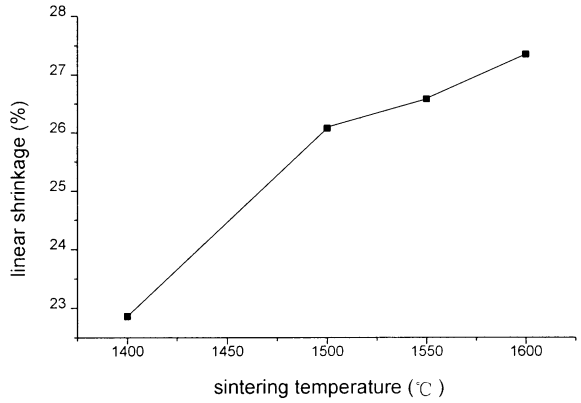

Fig. 3. Shrinkages of samples sintered to 1400, 1500, 1550, 1600 °C.

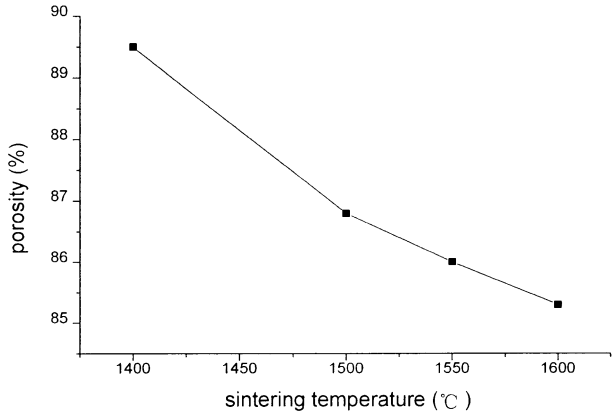

Fig. 4. Porosity of samples sintered to 1400, 1500, 1550, 1600 °C.

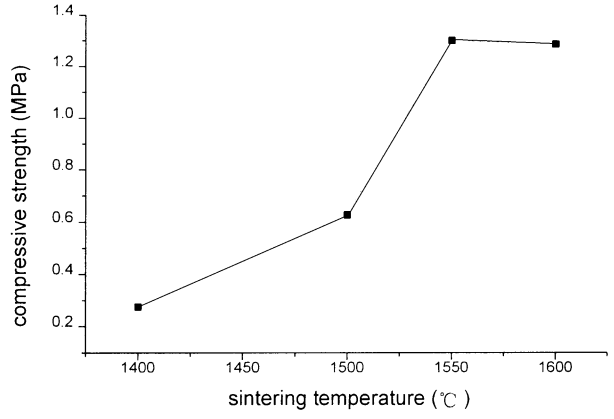

Fig. 5. Compressive strength of products sintered to 1400, 1500, 1550, 1600 °C.

foam shrunk increasingly. The shrinkage of samples changed from 22.86 to 27.35% when the sintering temperature increased from 1400 to 1600 °C. The change of shrinkage between 1400 and 1500 °C was larger than others. When the sintering temperature was up to 1500 °C, the shrinkage increased slowly.

Fig. 4 shows the porosities of samples after sintering to different temperatures (1400, 1500, 1550, 1600 °C).

Fig. 6. The holes in the center of webs.

The porosities decreased with the increase of sintering temperature, and it decreased obviously between 1400 and 1500 °C. After 1500 °C, the porosities of samples decreased slightly.

Fig. 5 shows the compressive strength of samples sintered to different temperatures (1400, 1500, 1550, 1600 °C). The compressive strengths were enhanced with the increase of sintering temperature below 1550 °C. It was up to 1.3 MPa at 1550 °C. After 1550 °C, the compressive strength decreased slightly with the increase of sintering temperature.

4. Discussion

All the samples discussed in this paper were prepared at the same condition except the sintering temperature. Hence, we can assume that the difference of each sample only resulted from the change of sintering temperatures.

For each foam, a substantial shrinkage occurred as the polymers (PU and PVA) were removed and the particles, which were initially packed loosely, approached and contacted. The removal of polymers left holes in the center of webs as shown in Fig. 6. These holes were the source of void that would move from the center to the outer surface during the procedure of sintering, at the same time the particles moved toward the internal surface of webs. These kind of movements led to the shrinkage of foams. The shrinkage became intensive with the increase of sintering temperature. As a result of shrinkage, the holes in the center of the webs became smaller as shown in Fig. 6. The shrinkage of holes and webs led to the decrease of porosity. In our experiment, the porosity of alumina foams decreased from 89.5% (1400 °C) to 85.25%(1600 °C).

Studies of brittle porous materials have demonstrated a relationship between the compressive strength and relative density. Generic expression have the following form [12]:

$$\sigma_{fc} = C\sigma_{fs}(R)^{3/2}$$

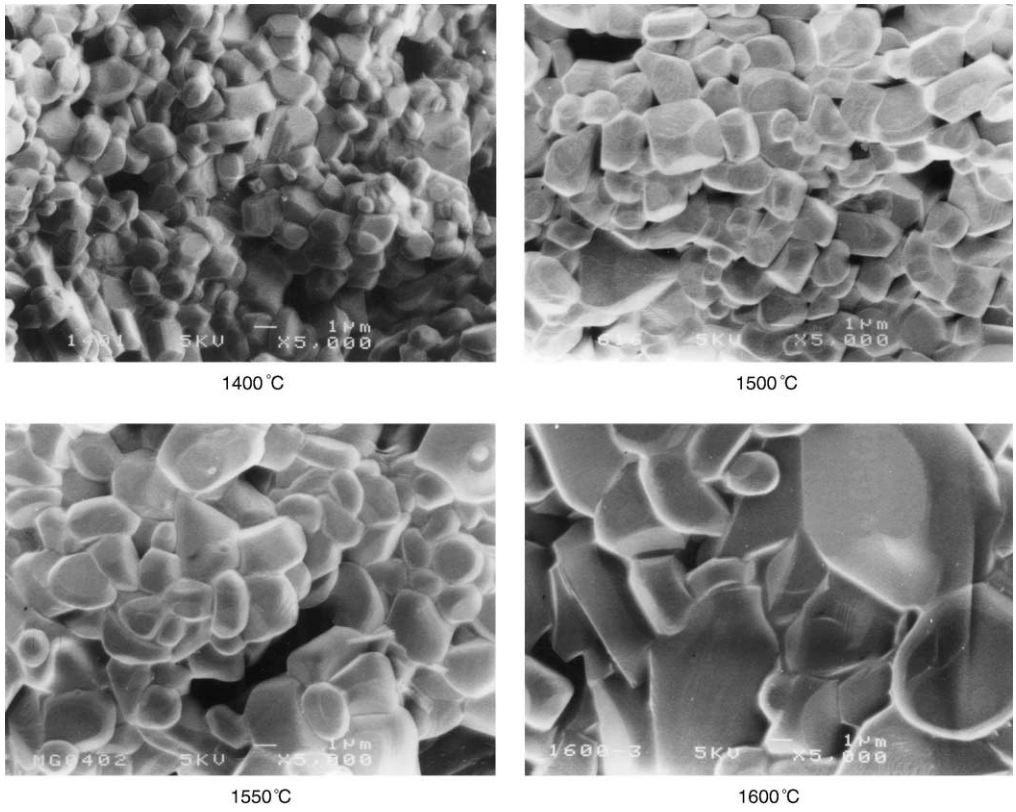

Fig. 7. The micrograph of samples sintered to 1400, 1500, 1550, 1600 °C.

where σ_{fc} is compressive strength, C a geometric constant characteristic of the unit cell shape, σ_{fs} the webs strength and R relative density which can be expressed by porosity as the following:

$$R = 1\text{-P}.$$

So the relationship between compressive strength and porosity can be expressed as the following form:

$$\sigma_{fc} = C\sigma_{fs}(1 - \mathrm{P})^{3/2}$$

Because porosity decreased with the increase of sintering temperature as discussed above, the compressive strength should increase with the increase of sintering temperature.

With the increase of sintering temperature, the microstructure of samples changed obviously as shown in Fig. 7. The SEM micrograph of 1400 °C shows that the particles are irregular and stack loosely, and only a few of the regular grains distributed among the particles. When the sintering temperature increased, the irregular particles grew up and turned into large grains. The SEM micrograph of 1550 °C show that the large grains are solidly bonded together and they have more contacting area, which would lead to the improvement of strength.

The porosity and grains contacting area are two key factors affecting the strength of alumina foams. When the sintering temperature increased from 1400 to 1500 °C, the porosity of samples decreased obviously which is the main reason causing the improvement of foams strength. When the sintering temperature increased from 1500 to 1550 °C, the porosity decreased a little but the grains grew up and have more contacting areas which became the main reason for causing the improvement of foams strength at that time. When the sample sintered to 1600 °C, a few grains preferentially grew very large compared to other grains, which was caused by overfiring. The overfiring usually results in a little decrease in strength, so the sample sintered to 1600 °C has a lower compressive strength than that of 1550 °C.

5. Conclusions

An idea to improve foams strength by controlling sintering temperature was proposed in this paper. The effect of sintering temperatures on alumina foams strength was studied by discussing three factors: shrinkage, porosity and grain size. When the sintering temperatures increased from 1400 to 1600 °C, the alumina foams shrunk increasingly, which resulted from the holes in the center of the webs becoming smaller and

the alumina particles approaching and contacting each other. The increasing shrinkage led to the porosity decreasing with the increase of sintering temperature. A lower porosity usually lead to higher strength. So the sample sintered to 1500 °C has a higher strength than that of 1400 °C. With the increase of sintering temperature, the grains size of samples grew up. When the sintering temperature is up to 1550 °C, the large grains solidly bonded together, which lead to the improvement of foam strength. The increase of strength from 0.627 MPa (1500 °C) to 1.3 MPa (1550 °C) mainly result from the growing up of grains because the minor change of porosity between 1500 and 1550 °C is not enough to account for the increase of strength. When the sample sintered to 1600 °C, a few grains preferentially grew too large compared to other grains, which caused a little decrease of strength. Therefore, to get a strong alumina foams, the sintering temperature of 1550 °C was suggested in our work.

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