Study on Molding of Fused-Silica Material by Pressure Slip-Casting from Alcohol-Based Slurry

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Abstract. In this manuscript a kind of alcohol-based slurry was prepared, in which the fused-silica powder, alcohol and thermosetting phenolic resin were the main components. Then, the fused-silica material was prepared by the process of pressure slip-casting, drying, curing and high temperature sintering. And at last, the properties of fused-silica material were analyzed. The slurry used for molding consisted of 82.5% fused-silica powder in which the 6 µm and 30 µm powders were at the ratio of 8:1, 17% alcohol and 0.5% phenolic resin, and settlement of the slurry was 3% after standing still for 2 hours. By pressure slip-casting at 0.5MPa pressure for 30 minutes, the green body with 12 mm thickness was obtained, and the strength of which could reach 2.3MPa after being cured at 90°C for 4 hours. After sintering at 1200°C for 2 hours, the strength of the fused-silica material reached 78MPa and the elastic modulus was 37.7GPa, and quartz and other crystalline phases did not appear in the test of XRD. Scanning electron microscopy showed that silica particles were uniformly distributed in the material.

1. Introductions
The fused-silica materials which mainly consist of fused-silica power or sand are sintered at high temperature [1]. With the characteristics of high strength, good thermal stability and low coefficient of thermal expansion, the fused-silica materials could be used as refractories and structural materials at medium high temperatures. And because of their low dielectric coefficient and stable dielectric properties at different temperatures, the fused-silica ceramics are also widely used in the field of wave-transmitting materials.

The slip-casting[2, 3]and gel-casting[4-6] are the two main widely used methods for molding silica material green bodies. In the slip-casting molding, the water-based slurry is made and then poured into the porous gypsum moulds. After a long time, the water is absorbed and the green body can be made. The gel-casting molding method was invented in the 1990s[7]. In the water-based fused-silica slurry, the monomers and cross-linking agents are added in and solidified by initiating the cross-linking reaction in hot water bath. The above two methods are based on the preparation of water-based slurry. These two technologies are mature and widely used in industry. However, because both slip-casting and gel-casting involve the preparation of water-based slurry and the milling time for the slurry is very long [8], the period for molding green body is much longer.

In this experiment, a certain amount of alcohol and phenolic resin were added in the fused-silica powder of 6 and 30 microns, then dispersed by ball milling, and aged for 24 hours at last. At the low pressure of 0.1MPa, the slurry was grouted into the mold and kept at the pressure of 0.4MPa to 0.7MPa for molding for 30 minutes. Then the green body was demoulded and cured at 90 °C. In this
process, alcohol was discharged from the green body and phenolic resin was cured to enhance the strength of the green body[9]. Then the green body was sintered and the fused-silica product was obtained. In the end of the experiment, the material was characterized by bending strength test, elastic modulus test, XRD and scanning electron microscopy.

2. Experiments and discussions

2.1. Determination of powder proportion and alcohol addition

In the experiment, the ratio of 6 and 30microns fused-silica powder was fixed to 8:1. The flow velocity and the suspension property of the slurry were investigated in the ways of Tu-4 cup empty time and 2 hours settlement. The flow velocity and the suspension property of the slurry were tested by adding 16.0%, 16.5%, 17.0%, 17.5% and 18.0% proportions of alcohol into and mixed with the fused-silica powder. The results are shown in Figure 1.

![Figure 1](image_url)

**Figure 1.** Effect of the alcohol content to Tu-4 cup empty time and 2 hours settlement of the slurry.

The Tu-4 cup empty time decreased with the increasing of alcohol content. But the 2 hours settlement of the slurry decreased with the decreasing of alcohol content. When the alcohol content was less than 17.0%, the Tu-4 cup empty time and the settlement amount were above 180s and below 3% respectively.

Considering both the flow velocity and the suspension property of the slurry, 17% alcohol was chosen to mix the slurry. Then the slip-casting experiment for making green body was carried out. The slip-casting time was set to 30 minutes, and the thickness of green body under the pressure of 0.4 MPa to 0.7 MPa was investigated. The results are shown in Figure 2.

The thickness of green body increased with the increasing of grouting pressure. The thickness of green body was 12 mm after 30 minutes of grouting at 0.5 MPa.

After being demoulded, the green body was dried and the bending strength was tested. In the test, the green body almost had no strength. The main reason for this was that it was difficult for the fused-silica powder reacting with alcohol to produce silica sol to increase the strength of the green body.

In order to increase the strength of fused-silica green body, a certain amount of thermosetting phenolic resin was added in the following tests.

2.2. Effect of phenolic resin addition to the strength of green body

According to the finished tests, the slurry with 17% alcohol content was selected to add a certain amount of phenolic resin.
By adding 0.1%, 0.2%, 0.5% and 1.0% phenolic resin, the green body was dried and curved at 90°C for 4 hours, and then the strength test was carried out. The result of bend strength was shown in figure 3.

In this test, the strength of the green body was obviously improved by adding resin. When the resin content was 0.5%, the strength of the green body was 2.3MPa.

Finally, the 17% ethanol and 0.5% ethanol were selected for the slurry and grouting at the pressure of 0.5MPa for 30minute was decided for slip-casting the green body in the following experiments. And some green bodies were produced, sintered and tested.

2.3. Effect of sintering temperature and time to material properties

2.3.1 Effect of sintering temperature on properties of materials. The density, strength and modulus of elasticity of the material were tested by sintering the green body at 1200°C, 1220°C, 1240°C and 1260°C for 2 hours. The results of tests were shown in figure 4 and figure 5.

It can be seen from figure 4 and figure 5 that with the increasing of the temperature, the density of products began to increase at first, and then stopped to increase when the temperature reached a certain value. The bend strength and elasticity modulus increased with the increasing of temperature, but when the sintering temperature was higher than 1240°C, the strength of materials decreased. The
main reason for this was due to the formation of a small number of crystalline phases during the material sintered at high temperature [10].

**Figure 4.** The density and shrinkage affected by sintering temperature.

**Figure 5.** The bend strength and elastic modulus affected by sintering temperature.

**Figure 6.** The density and shrinkage affected by sintering time.
2.3.2 Effect of sintering time to properties of materials. To investigate the sintering time to properties of the density, shrinkage, strength and elasticity modulus, the material was sintered at 1200°C for 1, 2, 3 and 4 hours respectively. The results are shown in figure 6 and figure 7.

When sintered at 1200°C, the density and linear shrinkage of the material increased with the increasing of the sintering time, and the flexural strength and elastic modulus also increased. The increase of sintering time leads to the increase of the density which is the main reasons for the obvious improvement of material properties.

![Graph showing the effect of sintering time on bend strength and elastic modulus.](image)

**Figure 7.** The bend strength and elastic modulus affected by sintering time.

![SEM images of the sintered material.](image)

**Figure 8.** The structure from SEM.

2.4. The microscopic morphology in sintering process
The product, sintered at 1200°C for 2hours, was analyzed by electron microscopy. The results are shown in figure 8, from(a) to (d).
The material contained some large particles of about 30 microns, which existed as the aggregates. The rest of the silica particles distributed uniformly, and the particles were tightly packed. But there was still a certain gap between the particles, and no crystal or liquid phase existed in the material.

2.5. Effect of different sintering temperatures on crystal form change of material

The materials which were sintered at 1200°C and 1260°C for 2 hours were tested by XRD to observe the crystalline phase changes. The result is shown in figure 9.

![XRD spectra of sintered materials](image)

**Figure 9.** The XRD of the material.

The crystalline phase was not found in the sintered material at 1200°C, but there was a small amount of crystal observed in the material sintered at 1260°C. The reason is that when the temperature was higher than 1240°C some crystalline phase was produced [11, 12], and this is the main reason for the strength reduction of the material after sintering at 1260°C.

3. The conclusions

From the experiments, these conclusions can be drawn from the experiment of high pressure slip-casting.

1. The Tu-4 cup empty time of the alcohol-based slurry mixed 30 and 6 microns fused-silica powers with 17% alcohol is 181 seconds and the 2 hours settlement amount is 3%. The slurry has good fluidity and suspensions.

2. The strength of the green body can be improved to 2.3MPa by adding 0.5% phenolic resin into the slurry, and the green body with a thickness of 12mm can be slip-casted in 30 minutes under the pressure of 0.5MPa.

3. After sintered at 1200°C for 2 hours, the strength of the material is 78MPa and the elasticity modulus is 37.7GPa. And in this material the particles distribute uniformly and no crystalline phase is formed.

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