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Fabrication of molybdenum disilicide components by slip casting

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Abstract

Molybdenum disilicide ($MoSi_2$) has many desirable properties which make it an excellent material for aerospace applications. This demands a reliable processing route to manufacture components with complicated shapes. The suitability of using slip-casting technique to fabricate $MoSi_2$ components has been studied. In this study, we have optimized the pH of the aqueous solution to get a reasonable suspension without adding any dispersing agents or sintering aids. Further, it is found that the 50 wt.% solid loading is best to get a compact of 92% density.

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1. Introduction

Ceramic materials can be compacted by various processing routes and each of them has its advantages as well as disadvantages. In any processing method, the ultimate aim is to consolidate the powdered sample to get a desired shape and densification by sintering the sample at high temperatures [1]. Slip casting or slurry casting is one of the oldest techniques in ceramic processing and can be used for manufacturing any complicated shaped ceramic components [2,3]. Its simplicity, low cost, flexibility and uniform particle packing are the added advantages for adopting this processing method [4]. There are limited reports available about the slip casting process of non-clay ceramic materials.

The green state processing plays an important role in reducing micro structural imperfection and hence increasing the reliability of the final ceramic products. The sintered density of the sample is directly dependent on the density and uniformity of the green microstructure [5]. The particle packing behavior in the green body is determined by the capability of the particles to remain separated from each other in the colloidal suspension [6]. The repulsion in this suspension can be enhanced by dispersants such as polyelectrolytes. However, the removal of these organic matters becomes very difficult at the time of sintering. Thus, it is preferable to have a colloidal suspension without adding a dispersant. The dispersion quality of any powder particles in aqueous media can be characterized by the zeta potential. The absolute maximum value of the zeta potential is essential for the production of highly dispersed electrostatically stabilized slips [7,8]. In case of $MoSi_2$, the highly negative zeta potential lies in aqueous alkali so that the basic media is good to form well-dispersed slips.

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Apart from inter-particle potentials, the other factors that influence the packing behavior of the powder compacts are the particle-size distribution, the particle shape and solid loading [5,9]. Generally, in slip casting the specific gravity of the slurry is kept as high as possible without compromising the fluidity of the slip, and this can be achieved by more extended particle size distribution [9]. Because extended particle size distribution reduces the interstitial pore space, the initial liquid goes into the interstitial pores and further addition will contribute to the fluidity of the slip [9]. However, there are different opinions about the effect of solid loading on green density, some authors have observed a linear increase in packing density whereas others have found an initial increase of green density followed by a decrease with increased solid loading [5,10].

Molybdenum disilicide ($MoSi_2$) based composites are potential structural materials for high temperature applications because of their properties such as high temperature strength, wear resistant properties and oxidation resistance in aggressive environments [7, 11,12]. Because of these properties, many applications including seals in internal combustion engines are being contemplated upon. We have optimized the conditions for slip casting of $MoSi_2$ compacts and the results are presented in this communication.

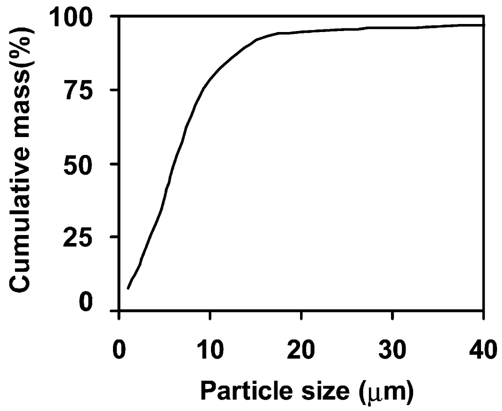

Fig. 1. Particle size distribution of $MoSi_2$ powder (cumulative mass).

2. Experimental procedure

The ceramic used in this experiment is 99.5% pure, micronized $MoSi_2$ powder (International Advanced Research Center for Powder Metallurgy and New Materials, Hyderabad, India). The average particle size measured by sedimentation technique (Sedigraph 5100, Micromeritics, USA) is 6 μm. The particle size distribution of the powder sample is found to be wide and is given in Fig. 1.

2.1. Settling experiment

Sedimentation test is done to find out the suitable pH value in which the slip possesses maximum colloidal suspension. Four different pH aqueous solutions (4, 7, 9 and 10.7) are taken in test tubes. The pH values are adjusted by adding hydrochloric acid and ammonium hydroxide solution to the aqueous media.1 wt.% of $MoSi_2$ powder is added to each test tube, the solution is well stirred and allowed to settle. The sedimentation heights of the different suspensions are measured as a function of time.

2.2. Rheological study

Viscosity measurements are made using a rotational viscometer (viscotester VT-500, Haake, Germany). This is a Searle type instrument where the outer cylindrical container remains stationary while the inner cylinder is rotated. Four aqueous suspensions were prepared with different solid loading starting from 45 wt.% to 70 wt.% of $MoSi_2$ powder and well dispersed using magnetic stirring. For all the viscosity measurement of slips, the pH of the aqueous solution is maintained at 10.7.

2.3. Slurry preparation and casting

Five different slurries are made with different solid loadings, starting from 45 wt.% ($MoSi_2$ powder) to 75 wt.% in the 10.7 pH aqueous solution. The green compacts made out of these slips are dried in air for 24 h and in an oven at 50 °C for 24 h. The dried compacts are sintered at 1800 °C for 15 min in argon atmosphere. The densities of the sintered samples were measured by using the Archimedes principle. The various important steps involved in the slurry preparing process are described in the flow chart (Fig. 2).

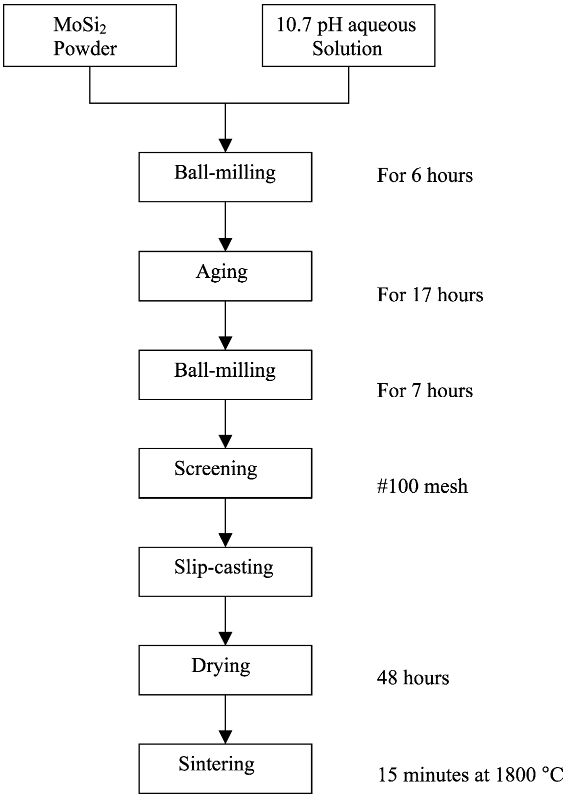

Fig. 2. Schematic diagram of the fabrication process of $MoSi_2$ monolithic components.

2.4. Fabrication of cylinders and seals

Fig. 2 shows the schematic diagram of fabrication of components using $MoSi_2$ powder. $MoSi_2$ powder (50 wt.%) is added with 50 wt.% of 10.7 pH deionized water in a pot mill. The charge material to ball ratio is

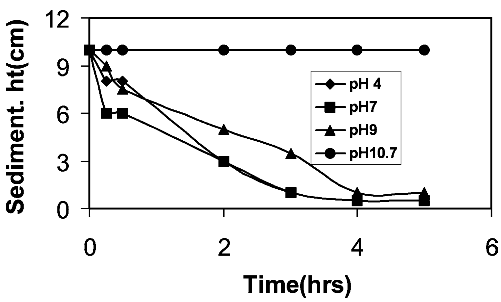

Fig. 3. Sedimentation height as a function of time for $MoSi_2$ powder at different pH.

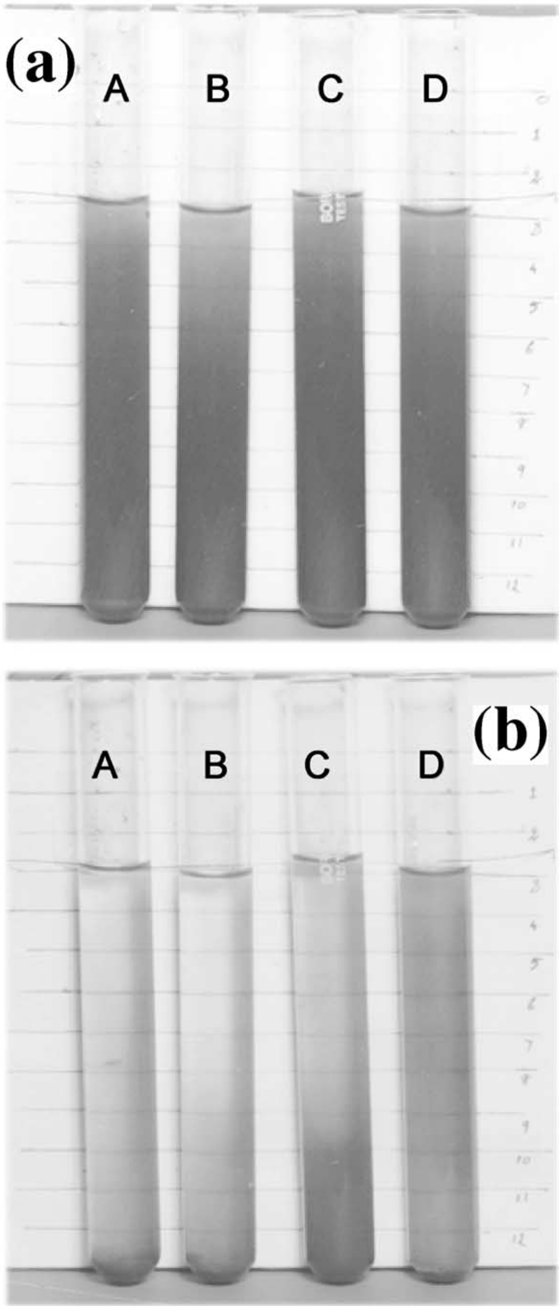

Fig. 4. Photograph of the sedimentation behavior of $MoSi_2$ powder: A—4 pH; B—7 pH; C—9 pH; D—10.7 pH. (a) Initial heights, (b) after 5 h.

kept at 1:3. After 6 h milling, the slip is allowed to age for 17 h. Then the slip is once again ball-milled for 7 h. The slip is strained through a 100 mesh net and the slip is cast into a plaster of Paris mould. The green components are dried in an oven and sintered at 1800 °C for 15 min.

3. Results and discussion

The sedimentation heights of all pH solutions measured as a function of time is given in Fig. 3. At initial stages all suspensions are well dispersed (Fig. 4a). But after 5 h, all systems have settled down except the 10.7 pH system (Fig. 4b), indicating that aqueous solution with a pH of 10.7 is good for casting $MoSi_2$ powder. The pH 11.5 aqueous solution also showed similar tendency as the pH 10.7 solution. For the longevity of the mould, however, it is always good to choose a pH value that has a minimum deviation from neutral pH. So pH 10.7 is taken as the optimum value for all studies.

Slurry preparation is an important step to get a good green compact with uniform density throughout the sample. One of the important parameters affecting the casting behavior and the densities is its viscosity. The measured viscosity values as a function of solid loading is plotted in Fig. 5. Viscosity increases with increasing solid loading. But this increase is marginal when the solid loading goes from 45 to 50 wt.% and it is drastic when the solid loading is further increased. So, 50 wt.% should be the optimum solid loading for casting.

Different slips are prepared with solid loading starting from 45 to 75 wt.% solid loading. The milling and aging times were followed as per the processing flow chart. All samples are dried and sintered under identical conditions. The resulting densities are plotted in Fig. 6. It is observed that the densities of the sintered samples are increasing with solid content in the slip and attains a maximum at 50 wt.% then decreasing with further increase in solid loading. Thus, 50 wt.% solid loading is preferred for further casting of shapes. This maximum density at 50 wt.% could be because of the drastic increase in viscosity of the slip as the solid content increased beyond this

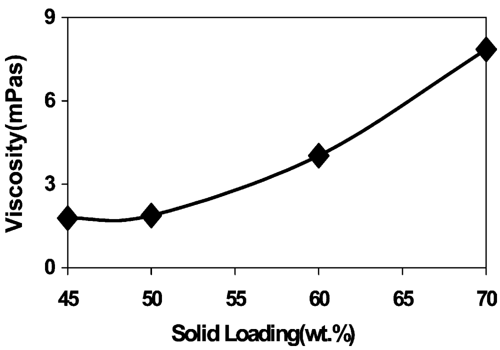

Fig. 5. Effect of solid loading on viscosity (at constant shear rate of 2700 s^{-1}; 10.7 pH).

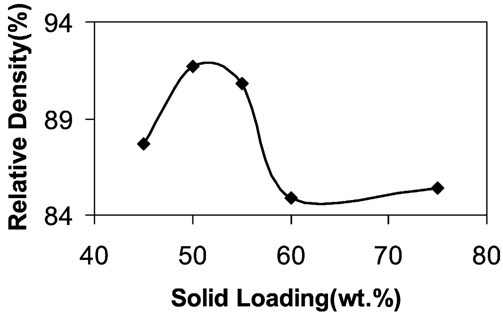

Fig. 6. Effect of solid loading on sintered density.

Fig. 7. Cylinder and seal fabricated using slip-casting technique.

value. The increase in viscosity can prevent the close packing of particles in the compact and consequently the density of the sintered samples.

Using the above optimized conditions, i.e. pH of 10.7 and solid loading of 50 wt.%, $MoSi_2$ cylinders (i.d. = 11 mm, o.d. = 20 mm and height = 15 mm) and seals (length = 40 mm, width = 6 mm and thickness = 2 mm) are cast and sintered. They are shown in Fig. 7.

4. Conclusions

The suitability of the slip casting to fabricate complicated shapes of $MoSi_2$ is studied. It is found that the optimum pH of aqueous solution for stabilizing the slip is 10.7. A maximum density is obtained when solid loading in the slip is around 50 wt.%. We have cast $MoSi_2$ cylinders and seals by slip casting, using the optimized conditions without using either dispersing agents or sintering aids.

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