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Rheological properties of slurries prepared using a planetary mixer

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Abstract

Preparation of ceramic slurries is an important process in ceramic colloidal processing. We investigated the rheological properties of slurries prepared using a planetary mixer. Planetary mixing for a short time enhanced the fluidity of the slurry. With longer-term planetary mixing, however, a film was observed on the surface of the slurry similar to the Ramsden phenomenon, which was due to the increases in temperature of the slurry by mixing. Slurries with this surface film showed high viscosity. A combination of planetary mixing for a very short time and ball milling was useful to enhance the fluidity of slurries.

Keywords: A. Milling; D. Zirconia; Planetary mixer; Slurry; Rheology

1. Introduction

Preparation of ceramic slurries is an important process in ceramic colloidal processing [1,2]. Especially, a high-solids slurry is necessary for near net shape techniques, such as slip casting, gel casting [3], direct coagulation casting [4], etc. In colloidal processing, the preparation of a high-solids slurry is needed to fabricate a fine green and sintered body. Therefore, various methods for preparation of ceramics slurries have been studied to improve their dispersion and fluidity characteristics [5,6].

Generally, slurries are dispersed by electrostatic or electrosteric stabilization mechanisms [7–9]. Electrostatic stabilization is accomplished by generating a surface charge on the particles, which causes a shift in pH of the slurry from the isoelectric point (IEP). The IEP of 3 mol% yttria-doped zirconia (Y-TZ) is pH 7.7 [10,11]. However, Y-TZ slurries prepared only with water showed pH values of around 5.4. Such slurries also show low viscosity, which is considered to be due to the positive surface charge of the particles. On the other hand, electrosteric stabilization requires the presence of polyelectrolytes. Previously, we reported the effects of addition of polyacrylic acid on the fluidity of Y-TZ slurries [12].

A number of crushing and milling techniques for the preparation of ceramics slurries have been developed, including ball milling [13], attrition milling [14], ultrasonic mixing [15], etc. In general, ball milling is used for preparation of high-solids slurries. However, the ball milling technique takes a long time to prepare a slurry with good colloidal stability and high fluidity.

Recently, a blade-free planetary mixer was developed for the mixing of ink, paint, or conducting paste. [16] When revolution and rotation are combined, the slurry in the container is mixed by whirling in the direction of the circumference and moving upward and downward along the rotation shaft. This device can be used to prepare homogeneous suspensions, slurries, and pastes in a short time.

There have been several reports of the application of this device for the preparation of ceramics slurries. Fukuoka et al. [17] reported the preparation of a homogeneous SiO_2–TiO_2–BaO sol using planetary mixer in a very short time. In the present study, the fluidity of Y-TZ slurry prepared by planetary mixing was compared with that of a slurry prepared by the conventional ball milling technique.

2. Experimental

The ceramic powder used was high-purity ZrO_2 in partially stabilized form (Y-TZ) (TZ-3YS, Tosoh Co., Ltd., Tokyo, Japan). Average particle size, specific surface area, and density of the Y-TZ were 0.56 μm, 7 $m^2\,g^{-1}$, and

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Table 1
Revolution speed

	Setting values		
	5	7	9
Revolution (rpm)	804	1058	1340

Table 2
Rotation speed

Rotation	Revolution (rpm)		
	5	7	9
7	619	815	1032
8	703	931	1179
9	804	1058	1340

$6.05\ g\ cm^{-3}$, respectively. Slurries were prepared by adding Y-TZ to water. The water used to prepare all of the sample solutions was distilled and purified with a Milli-Q system (Milli-Q Plus, Millipore Corporation, Billerica, MA). No dispersants or other reagents were added to the slurries.

Concentrated slurries were prepared by two methods. Planetary mixing was performed using a planetary mixer (MS-SNB 350N, Matsuo Industry, Osaka, Japan). Tables 1 and 2 show the rotation and revolution rates used in this study, respectively. The rotation rate is dependent on the revolution rate, and the maximum rotation rate is equal to the revolution rate. The mixing time can be set from 10 to 990 s in 10-s intervals. The other method used was conventional ball milling with Y-TZ balls for 1–48 h at room temperature. Furthermore, a slurry was prepared by ball milling after planetary mixing.

The rheological behavior of slurries was evaluated by monitoring the apparent viscosity obtained from the shear rate versus shear stress curves at 25 °C using a controlled stress rheometer (RS-150, Haake, Karlsruhe, Germany) with double gap cylinder geometry (DG-41).

Sedimentation tests were carried out with 60 wt.% slurries transferred to a filled height of 25 ml in graduated cylinders (ca. 1 cm in diameter and ca. 30 cm in height). The origin of the time scale was taken as the slurries were poured into the graduated cylinders. Sedimentation tests were performed for 30 days.

3. Results and discussion

The fluidities of Y-TZ slurries were measured to examine the mixing efficiency of the planetary mixer. Fig. 1 shows the apparent viscosity and temperature changes of 81 wt.% Y-TZ slurries as a function of the revolution rate and rotation rate. The mixing time was 150 s. Apparent viscosity of the slurries decreased with increases in both revolution rate and rotation rate. However, at a revolution rate of 9, the slurry temperature increased by more than 20 °C and a film formed on the surface, similar to the phenomenon observed when milk is heated, i.e. the Ramsden phenomenon. This was considered to be due to evaporation of water from the surface of the slurry due to the increase in temperature. The formation of this film caused heterogeneity in the Y-TZ slurry. Therefore, the optimum conditions in this system were a revolution rate of 7 and rotation rate of 9.

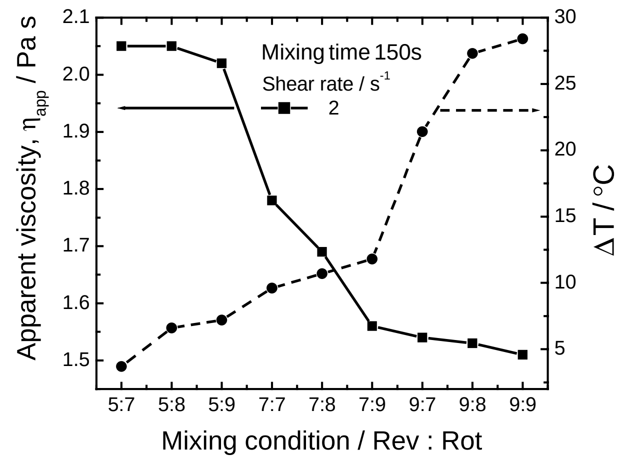

Fig. 1. Apparent viscosity and temperature change for 81 wt.% ZrO_2 slurries as a function of revolution and rotation ratio.

Fig. 2 shows the effects of planetary mixing time on fluidity at the optimum mixing conditions for 81 wt.% Y-TZ slurry. Apparent viscosity decreased to a low value until 150 s, maintained this level until 180 s, and then increased. The slurry temperature also increased with mixing time, and showed an increase of 15 °C after 210 s of mixing. In addition, the slurries with a surface film showed high viscosity.

Fig. 3 shows the apparent viscosities of 81 wt.% Y-TZ slurries prepared by ball milling after planetary mixing or by conventional ball milling only as a function of ball milling time. The apparent viscosity of the slurry prepared by ball milling with planetary mixing was lower than that prepared by conventional ball milling only with all ball milling times examined. The apparent viscosity of the slurry prepared by ball milling for 12 h after planetary mixing for 150 s was

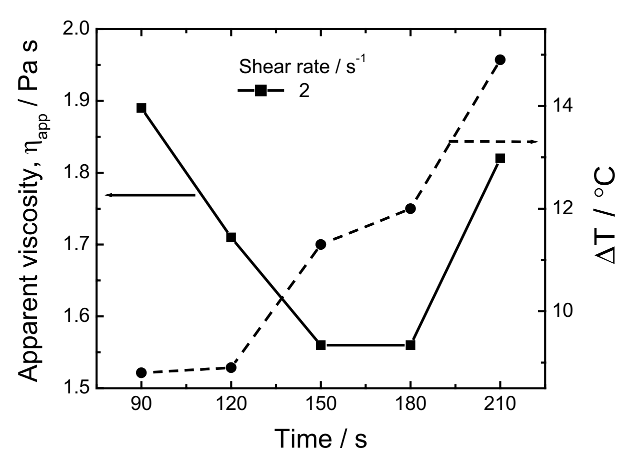

Fig. 2. Apparent viscosity and temperature change for 81 wt.% ZrO_2 slurries as a function of planetary mixing time.

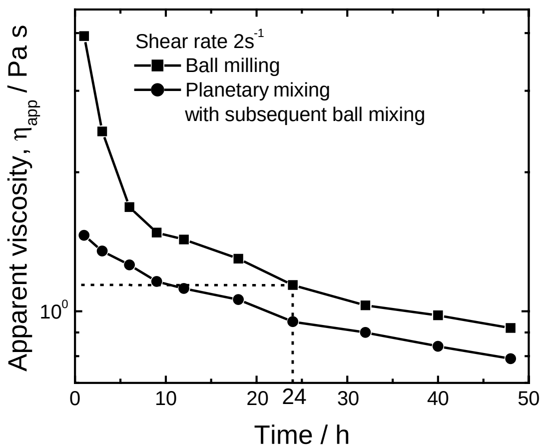

Fig. 3. Apparent viscosity for 81 wt.% ZrO_2 slurries as a function of ball milling time with/without planetary mixing.

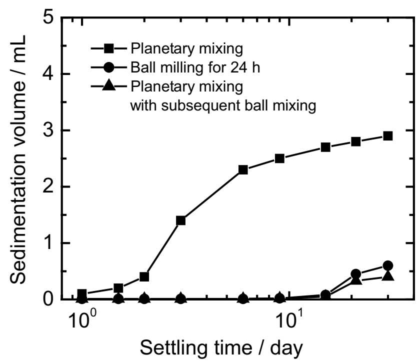

Fig. 4. Sedimentation volume as a function of settling time for 60 wt.% Y-TZ slurries prepared using various techniques.

equivalent to that of the slurry prepared by ball milling only for 24 h.

Fig. 4 shows the results of the sedimentation tests of various slurries. The slurries prepared by ball milling with/without planetary mixing produced little sediment after 30 days. However, the slurry prepared with planetary mixing alone began to produce large amounts of sediment after 4 days, and produced 3 ml of sediment after 30 days. The slurry prepared by mixing alone is considered to contain a portion of the large aggregated particles, suggesting that planetary mixing alone could not break down the aggregated particles.

Finally, Fig. 5 shows the apparent viscosity of the slurry prepared by ball milling with planetary mixing under the optimum conditions, as a function of solids concentration. Slurries retaining low viscosity can be prepared up to 83 wt.% using the method described here.

4. Summary

High-solids loaded Y-TZ slurries could be prepared easily using the blade-free planetary mixer system described here. The characteristics of slurries prepared by planetary mixing

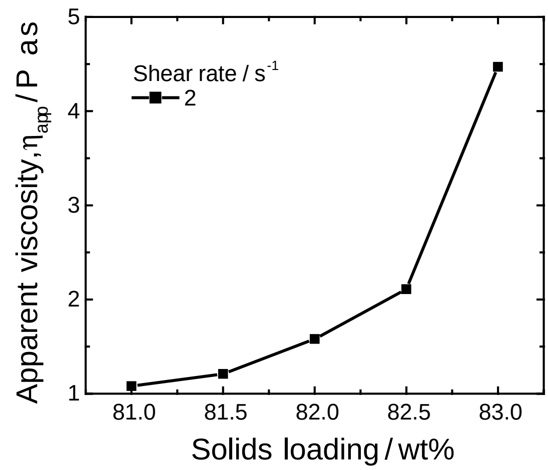

Fig. 5. Apparent viscosity as a function of solids loading for ZrO_2 slurries prepared by ball milling with planetary mixing.

were compared with those prepared by the conventional ball milling method. Planetary mixing allowed the preparation of Y-TZ slurries in less time than conventional ball milling, and the resultant slurries had low viscosity. Ball milling with planetary mixing could be used to prepare Y-TZ slurries in half of the time required by the conventional method. This indicates that planetary mixing can be used to markedly reduce the preparation time for ceramic slurries. When the revolution rate is too fast or planetary mixing time is too long, a film is formed in the surface of the slurry because of the increase in temperature of the slurry. Thus, it is necessary to determine the optimal balance of the revolution rate, rotation rate, and planetary mixing time. Furthermore, a portion of the large aggregate particles could not be crushed by mixing alone. The combination of planetary mixing and subsequent ball milling was useful to enhance the fluidity of slurries keeping processing time to a minimum.

Acknowledgements

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