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Controlling particle size distribution for preparing multi-layer structure compacts

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Abstract

Ceramic compacts which have a self-arrangement characteristic according to surface properties in moulding process were made. Two types of SiO_2 powder, of which particle sizes are different from each other in average, went through the surface treatment through stearic acid and ABDM (alkyl benzyl dimethyl ammonium chloride) to have hydrophobic and hydrophilic features, respectively. This process is to induce diverse movements to several directions by surface properties in a slip. In gypsum mould, it is shown that different kinds of particles have relatively different movements in the process of squeeze casting to make compacts, that is, particles with hydrophobic features on the surface tend to move to the surface of the gypsum and particles with hydrophilic features on its surface show a tendency to be distributed evenly in the slip. Finally, on the surface of the compact are mainly hydrophobic particles, and hydrophilic particles come to exist mainly in the inside of the compact, and this demonstrates the fact that by modifying surface properties the particles distribution can be controlled artificially, and this study observed the fact through based on the experimental results above.

Keywords: Slip casting; Surfaces modification; Particle size distribution

1. Introduction

The slip casting is a method of moulding by injecting slip mixed with powder and water into a porous mould, and this is being utilized frequently to produce functional gradient materials (FGMs) due to its characteristic of the control of particles distribution in casting. For example, ceramic filters, which were made through several and separate processes, have been currently attempted to be made in a single process by using the slip casting to make FGMs, by some researchers.[1–4] There have been several ways to control particles arrangements: particles sedimentation to make FGMs is a basic method by which particles in the slip arrange by gravity.[5] López-esteban et al. tried to concentrate zirconia on the surface of stainless steel by using the differences between sedimentation levels of particles in the slip, and in such an experiment, two types of particles with different sizes formed vertical concentration gradients.[6] This study aims to present that such a way is useful to produce FGMs due to the fact that surface properties of particles control the formation and detailed structures of compacts in casting.

To make slips, each particle was hydrophobic or hydrophilic-coated on the surface, and then the slip made was injected into moulds. Experiments of this study was carried out based on the supposition that during a casting process, particles with each feature would show different movements from those of other features and by using such characteristics compacts which have an intended arrangement of particles could be made.

2. Experimental

2.1. Raw materials

As starting materials, two types of SiO_2 powder, made by KOJUNDO Chemical Co. in Japan, which has an average particle size different from each other was used, and for fine samples the powder of $d_{50} = 0.8\ \mu m$ was used and for coarse samples, the powder of $d_{50} = 4.0\ \mu m$.

2.2. Surface modification of samples

Samples were added with 3 wt% of stearic acid and stirred by the ball mill method for 60-minute for their hydrophobic features. Contact angles of powder were measured using the

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Table 1
Composition of three type of slips (unit: wt%).

	None treatment		Hydrophobic		Hydrophilic		Additive	
	$d_{50} = 0.8$ μm	$d_{50} = 4.0$ μm	$d_{50} = 0.8$ μm	$d_{50} = 4.0$ μm	$d_{50} = 0.8$ μm	$d_{50} = 4.0$ μm	Talc	Sodium silicate
Slip 1	48	48	–	–	–	–	3	1
Slip 2	–	–	48	–	–	48	3	1
Slip 3	–	–	–	48	48	–	3	1

method of double-sided tape (CAHN, Model DCA 315, USA) to check the hydrophobic degree and the angles ranged from 80 to 85°. As for hydrophilic treatment on the surface of particles, ABDM (alkyl benzyl dimethyl ammonium chloride) which has a very strong hydrophilic atomic group as cation-active surfactant was employed. Designated samples were made to aqueous slurry in controlled pH 12, and then stirred for 24 h after adding ABDM of 5 mg/L. The contact angle of this sample is close to 0.

2.3. Slip preparation and casting

As shown in Table 1, three types of slips, which are classified according to surface properties, were made by using each sample treated differently from others. Slip 1 used the powder with no surface treatment, slip 2 used powder samples with hydrophilic ($d_{50} = 4.0$ μm) and hydrophobic ($d_{50} = 0.8$ μm) ones, and slip 3 used powder samples with hydrophilic ($d_{50} = 0.8$ μm) and hydrophobic ($d_{50} = 4.0$ μm) ones.

Each slip was composed hydrophilic and hydrophobic ones (48%:48%), and as additives, talc of 3 wt% with the size of −325 mesh and sodium silicate of 1 wt%. The specific gravity of slips was adjusted as 1.7–1.8 by using sodium silicate to secure proper conditions for casting. Each slip was matured for 24 h, enough for particles to be fully mixed with water, and then it was injected into gypsum moulds for casting. This is to apply not only the fundamental driving force of particles with different sizes in a slip, by which such particles are easily separated and then move themselves in the casting process, but mobility of water occurred when the gypsum makes a prompt absorption of water. The diameter of the cylinder-type mould was 25 mm and, the green body after de-moulding had the diameter of about 23 mm and the thickness of about 5 mm. Fig. 1 shows the schematic diagram and the experimental route of the slip casting process.

3. Results and discussion

3.1. Effect of particle size and particle segregation

The colloidal approach is very important in casting powder compacts immediately from slurry. Conventional casting such as slip casting and tape casting is able to directly utilize colloid particles and new ways are easily applied into injection process in such castings.[7]

The slip 1 with particles of no surface treatment was applied for casting in gypsum mould. When the gypsum mould absorbs water, smaller particles are gathered first in both sides of the gypsum mould, that is, at the top and the bottom, following water. As shown in Fig. 2, larger particles are put in the part of the middle height while smaller ones accumulate at the top/bottom. At the bottom, the sizes of particles which should be distributed in the top and middle parts are also observed, and it is thought that is because of the sedimentation of larger particles by gravity. Although particles should be distributed in order of their sizes (weights), the phenomenon above demonstrates clearly that the gravity in the experiment plays just a secondary role for particle segregation, based on the results that smaller particles accumulate preferentially at the top and the bottom by characteristics of gypsum mould and that those smaller particles obstruct the sedimentation of larger ones, which is called clogging effects. Fig. 2, however, shows insignificant differences between pictures of different heights because the absorptive power of the gypsum mould is the only driving force to cause such a situa-

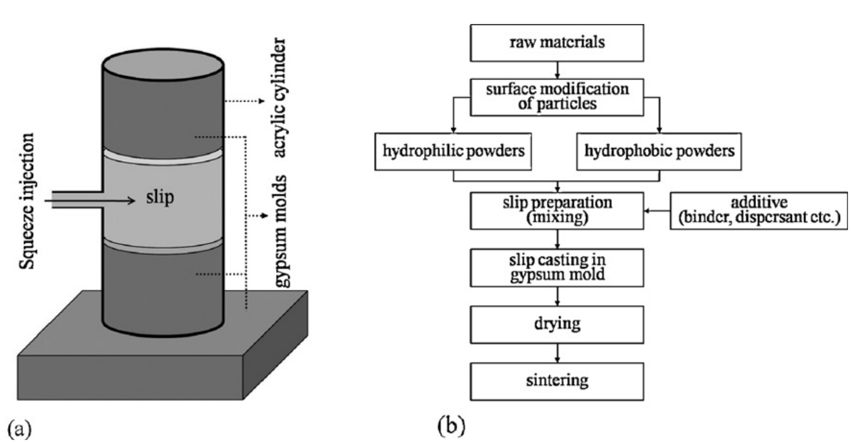

Fig. 1. Schematic diagram and experimental route of the slip casting process: (a) squeeze slip casting in gypsum mould and (b) experimental route.

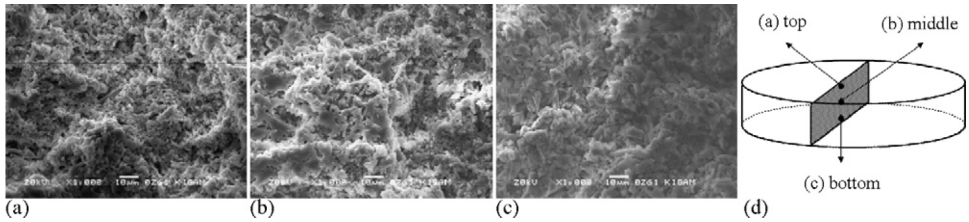

Fig. 2. Position of particles in compact by the slip 1: (a) top (smaller particles), (b) middle (lager particles), (c) bottom (mixed with smaller and larger particles) and (d) position of particles.

tion in this experiment, where particles do not have any surface treatment.

3.2. *Effect of particle surface treatment*

The relations between surface properties of particles and their arrangements are examined in cases of the casting process using the slip 2 and slip 3 which have surface treatment.

Fig. 3 exhibits SEM micrographs of compacts being utilized the slip 2. Fig. 3a is composed of relatively smaller particles, comparing to Fig. 3b and c being mixed with mainly smaller and less larger ones. That shows a state that fine particles of 0.8 um with hydrophobic surfaces are distributed mainly at the top side while particles of 4.0 um with hydrophilic surfaces are located in the middle part.

Such phenomena is more clearly observed than that in Fig. 2, and that is because hydrophobic and hydrophilic particles come to be segregated easily by forces of hydrophobic particles to tend to move out the interface of liquid as well as by absorption mobility of the gypsum mould.

Also there are more less bigger particles at the bottom than those in Fig. 2 although hydrophilic bigger ones are shown together with hydrophobic smaller ones. From the results, it can be thought that the surface properties take ahead sedimentation by gravity and the clogging effect for arranging particles.

As shown in Fig. 4, the slip 3 exhibits most obviously effects of the surface treatment on particle arrangements in case of making compacts. Larger particles come to move to the top and bottom, interfaces of the gypsum mould due to the fact that they are hydrophobic so that they show a strong tendency to move out the liquid despite their large sizes. Naturally, smaller particles with hydrophilic properties are located in the relatively middle part, but by the water flowing caused by segmentation by gravity and the absorptive power of gypsum, some of smaller ones become placed with larger particles at the bottom layer.

3.3. *Mechanism of controlling particle size distribution in the slip casting*

In case of slip casting, particle size distributions can be affected by various conditions, that is, the particle size distribution in a slip is able to be adjusted by a method based on surface properties of particles, differences of the momentum for acceleration and the velocity of particles and the ratios for relative sizes of particles. In this study, we explore a mechanism in which particle arrangements develop by the differences of surface properties, based on the results drawn from experiments. In Fig. 5a, particles with hydrophobic surfaces have a strong tendency to move into gas–liquid or solid–liquid interface, so they go to the interfaces, that is, the surfaces of gypsum, for forming surface layers as shown in Fig. 5c.

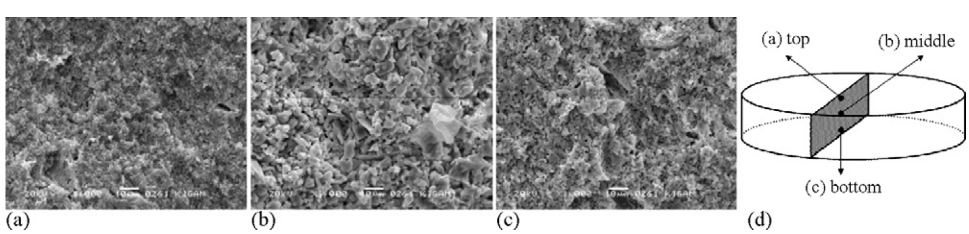

Fig. 3. Position of particles in compact by the slip 2: (a) top (smaller particles), (b) middle (lager particles), (c) bottom (mainly smaller particles and less bigger ones) and (d) position of particles.

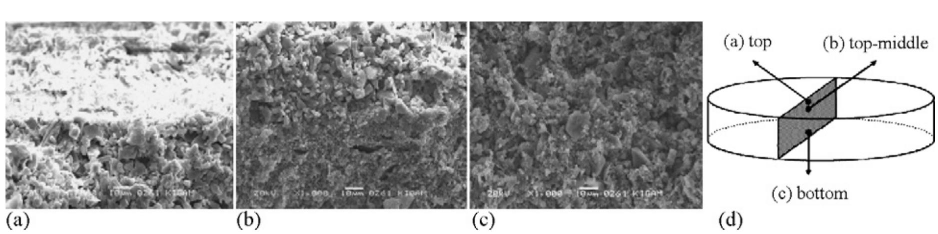

Fig. 4. Position of particles in compact by the slip 3: (a) top (larger particles, (b) top–middle (mixed with lager and smaller particles), (c) bottom (mainly lager particles and less smaller ones) and (d) position of particles.

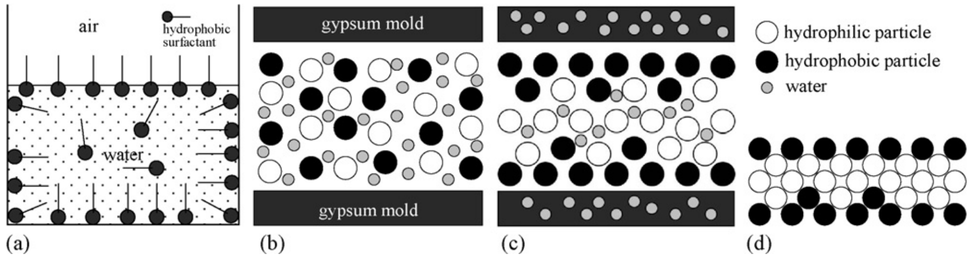

Fig. 5. Mechanism of controlling particle size distribution by surface properties in the casting process: (a) model on hydrophobic surfactant arrangement in gas–liquid and solid–liquid interface, (b) initial stage: random particle mixture (random arrangement), (c) middle stage: particles moved by surface property (1st self-arrangement) and (d) final stage: particle alignment completed (final self-arrangement).

In addition, particles with hydrophilic surfaces tend to be distributed evenly in the slip, and particles which have few specific properties of surfaces show movements driven mainly by sizes of particles, and movements to interfaces or distribution of those particles are seldom observed. Consequently, in case of slip casting, particle arrangements in compacts are affected preferentially by differences of surface properties, followed by the segmentation by gravity or the clogging effects (Figs. 3 and 4).

It is expected to be able to produce compacts of which particle arrangements are controlled according to surface properties of particles as shown in Fig. 5d, by using such a mechanism. The very driving force of the mechanism can be the differences of surface properties of particles and the mobility by fluid flowing occurring when gypsum makes fast absorption of water.

4. Conclusion

This presents possibilities of artificial arrangements of particles by using their surface properties and identifies its mechanism by making compacts with multi-layers. It can be thought that in case of slip casting, surface properties of particles are applied more preferentially than the segmentation by gravity or the clogging effects do for arranging particles in compacts. Under such a mechanism, the slip casting method by using gypsum is able to construct much stronger momentum by surface properties of particles and the mobility caused by fluid flowing occurring by the gypsum to absorb water fast. It is possible to make multi-layers casting by surface properties when applying this mechanism.

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