Evaluation of Bubble Content in Aqueous Alumina Slurries

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Entrapped air bubble is one of the main defects in slurry processing of ceramics. For preventing bubble occurrence or removing them efficiently, an improved knowledge of bubble behaviors in slurries is indispensable. In this paper, effects of particle characteristics, solid loading and dispersant concentration on bubble content in aqueous alumina slurries were studied experimentally. The results showed that the characteristics of initial powders, i.e. particle size distribution and shape, significantly affected the bubble content in slurries. The bubble contents were drastically enhanced by smaller and irregular particles with a higher specific surface area. Moreover, the bubble contents were also greatly increased with increasing solid loadings, and this influence was more pronounced for the particles with a higher specific surface area. No noticeable change in bubble content was resulted from varying the dispersant concentration. A new parameter, solid surface area per unit volume of slurry has been introduced to interpret these effects of powders. It is found that the bubble content shows a parabolic increase as a function of the parameter.

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1. Introduction
Slurry processing has been studied extensively for ceramic manufacture owing to its potentials in achieving homogeneous structures and enhanced performances of ceramics.\(^1\),\(^2\) However, entrapped air bubbles in slurries have been recognized to be one of the main sources to cause defects in the final products.\(^3\),\(^4\) Air bubbles are usually introduced during slurry preparation, and difficult to be removed completely in the following de-airing step. Remained bubbles impair the homogeneity of slurries, leaving abnormal pores in green samples and subsequently the final products, and deteriorate the properties of ceramics. Especially for gelcast process, existence of bubbles is more detrimental since the slurry structures tend to be "frozen" during their consolidation to form green compacts.\(^3\),\(^5\),\(^6\) Therefore, fully understanding of bubble behaviors in slurries is crucial for both preventing bubble occurrence and removing them efficiently to produce advanced ceramics with excellent properties. Although a number of de-airing experiments are tried in order to decrease the bubble content in ceramic slurries after preparation, there is still lack of study in bubble behaviors in ceramic suspensions, e.g., which factors affect the bubble content during preparation of the slurries.

In this work, bubble contents in aqueous alumina slurries were experimentally investigated concerning the effects of powder characteristics, solid loading and dispersant concentration. This is a first attempt to give a quantitative analysis of bubble behaviors in ceramic slurries. The main factors that influence the bubble formation, stability and subsequently the bubble content were clarified based on the experimental results.

2. Experimental procedure
Four types of \(\alpha\)-Al\(_2\)O\(_3\) powders were used to prepare slurries, i.e., AL–160SG–1 (99.8% of purity, Showadenko), AKP–20 (99.99% of purity, Sumitomo Chemistry), and AA–1 and AA–05 (99.99% of purity, Sumitomo Chemistry). The particle morphologies were analyzed with scanning electron microscopy (SEM) and the size distributions with X-ray sedimentation. Each type of powder was made into two series of slurries with varying solid loadings and dispersant concentrations, respectively. The powders were firstly mixed with distilled water and a commercial dispersant D–305 (Ammonium polyacrylate, Chukyoyushi), and then ball milled for 24 h in sealed 1000 ml plastic bottles. Slurry was poured into a 200 ml beaker and kept still for 5 min. The samples were then taken from the center of the bulk slurry for bubble content measurements. All the measurements were carried out at 26–28°C. The bubble content was determined by the equation

\[
C_b = \left(1 - \frac{\rho}{\rho_0}\right) \times 100\%
\]

where \(C_b\) is the bubble volume fraction, \(\rho\) the slurry density (measured with pycnometers) and \(\rho_0\) the theoretical slurry density without bubbles (calculated according to the slurry composition).

3. Results and discussion
Figure 1 shows the SEM micrographs of the four types of alumina powders. Their morphologies differ greatly: AL–160SG–1 and AKP–20 powders have irregular shapes, and both AA–05 and AA–1 powders are nearly spherical. The AL–160SG–1 type has the highest B.E.T. face areas due to their larger particle sizes and spherical shape.

Figure 2 shows the particle size distributions of the powders obtained using X-ray sedimentation technique. The AL–160SG–1 powder has the smallest mean particle size; the irregular AKP–20 has a slightly larger one of 0.57 \(\mu\)m. The spherical AA–05 and AA–1 powders have mean particle sizes of 0.67 \(\mu\)m and 1.26 \(\mu\)m, respectively. These results are in good agreement with those of the SEM observations.

The characteristics of all the alumina powders are summarized in Table 1. The AL–160SG–1 type has the highest B.E.T. specific surface area due to the smallest mean particle size and the irregular shape, and the AA-series have low specific surface areas due to their larger particle sizes and spherical shape. The specific surface area reflects influence of both particle size
Fig. 1. SEM photographs of the different types of alumina powders.

Fig. 2. Particle size distributions of the alumina powders obtained by X-ray sedimentation measurement.

Fig. 3. Effect of solid loading on bubble content with a constant dispersant concentration of 0.5 mass%.

Table 1. Summary of Typical Parameters of the Alumina Powders

<table>
<thead>
<tr>
<th>Type</th>
<th>Shape</th>
<th>Mean particle size (μm)</th>
<th>B.E.T. specific surface area (m²/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AL-160SG-1</td>
<td>irregular</td>
<td>0.45</td>
<td>7.0</td>
</tr>
<tr>
<td>AKP-20</td>
<td>irregular</td>
<td>0.57</td>
<td>4.0</td>
</tr>
<tr>
<td>AA-05</td>
<td>spherical</td>
<td>0.67</td>
<td>3.1</td>
</tr>
<tr>
<td>AA-1</td>
<td>spherical</td>
<td>1.26</td>
<td>1.6</td>
</tr>
</tbody>
</table>

Fig. 4 shows the effect of dispersant concentration on bubble content in the slurries. The variation in dispersant concentration seems to have little influence on the bubble content for all types of the powders.

By combining the results given in Fig. 3 and Fig. 4, we conclude that powder characteristics and particle concentration (solid loading) are the main factors affecting the bubble contents in the slurries. Based on the findings, we introduce a new parameter $A_i$, solid surface area per unit volume of the slurry, to interpret the effects of powders on the bubble content. $A_i$ is defined by the equation

$$A_i = \rho_p C_p A,$$

where $\rho_p$ is the solid powder density, $C_p$ is the solid concentration, and $A$ is the solid surface area per unit volume of the slurry.
A relation is fitted very well with a parabolic curve. Clearly, the relation is fitted very well with a parabolic curve. Consequently, bubbles at the surface of the slurry are also stabilized because of the decreased gas diffusion and drainage in the bubble film. The discrete points are experimental data and the line is a least-squares fit using parabola function, where $C_0$, $C_1$ and $C_2$ are constants.

Figure 5 shows the bubble content as a function of $A_i$. The relation is fitted very well with a parabolic curve. Clearly, the parameter $A_i$ dominates the bubble stability, i.e., the powder characteristics (particle size distribution and shape that determine the specific surface area) and solid loading dominate the bubble stability. The relationship is of great significance for ceramics processing as predicting the presence of bubbles entirely on the value of this parameter for all the four types of alumina slurries.

Furthermore, the stability of bubbles in the slurries has been examined by measuring the bubble contents in the slurries kept still in air with prolonged time. The result for the AL–160SG–1 slurry is demonstrated in Fig. 6. The absolute errors of the measured results are less than 0.01 vol.%. The fact that the bubble content has little change indicates that the bubbles formed during the slurry preparation are relatively stable and unlikely escape naturally. Therefore, control of bubble formation in the initial stage of slurry preparation is fundamental and crucial besides the subsequent de-airing procedures.

4. Summary

Bubble contents in aqueous alumina slurries prepared with different types of alumina powders have been studied in this work. It is found that powder characteristics and solid loading greatly affect the bubble contents, whereas dispersant has negligible influence. The bubble contents are significantly enhanced using powders with higher specific surface area as well as increasing the solid loading. A new parameter, solid surface area per unit volume of slurry, has been successfully introduced to interpret the effects of particle characteristics and particle concentration on the bubble content in the slurries. The bubble content increases parabolically with increasing the value of this parameter for all the four types of alumina slurries studied.

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References