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The effect of wet foam stability on the microstructure and strength of porous ceramics

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ABSTRACT

Wet foam stability is of prime importance in fabricating porous ceramics with the desired microstructure and mechanical properties. In this research, wet foams were fabricated via direct foaming after separately adding an anionic surfactant (TLS) and a cationic surfactant (DTAC) into alumina slurries with a copolymer of isobutylene and maleic anhydride (PIBM) as both the dispersant and the gelling agent. The foam stability was evaluated by a stability analyzer. The bubble size rapidly increased in the wet foam with TLS as the foam stabilizer and many large bubbles appeared within 60 min. The wet foam containing DTAC was very stable. Cationic DTAC increased the hydrophobicity of alumina particles by interacting with the anionic PIBM adsorbed on the particles. The hydrophobically modified particles acted as the foam stabilizer and enhanced the wet foam stability. Furthermore, the fast gelling speed of the slurry containing DTAC also enhanced the wet foam stability. The average cell size of the ceramic with 82.9% porosity from the wet foam with TLS was 188 μm and the compressive strength was 9.7 MPa. The counterparts from the wet foam with DTAC were 54 μm of average cell size and 18.1 MPa of compressive strength. The superior stability of wet foam brought about a smaller cell size and higher strength of the resultant ceramic.

1. Introduction

Porous ceramics possess unique properties including high surface area and low thermal conductivity due to their cellular microstructure compared to dense ceramics. These enable them to have important roles in many industrial applications including molten metal filtration, water purification, catalyst support, high-temperature thermal insulation, and bone tissue replacement depending on their porosity, cell size, and structure [1–3].

Some techniques have been developed to fabricate porous ceramics including sacrificial template, extrusion molding, and direct foaming. Direct foaming is a simple and cost-effective method that has been extensively used to fabricate various porous ceramics such as alumina, silicon carbide, and mullite [4–7]. Wet foam with surfactant as the stabilizer must be rapidly solidified by a gelling system because the

foam is thermodynamically unstable. The gelling system used in direct foaming includes free radical polymerization of acrylamide [5] or methacrylamide [6], nucleophilic polymerization of epoxy resin and amine [8], and spontaneous gelation of PIBM (a copolymer of isobutylene and maleic anhydride) [7,9,10]. On the other hand, particle-stabilized foams have also been widely researched to fabricate porous ceramics [11–14] since ultrastable foams were first reported [15]. The particles used as the foam stabilizer were hydrophobically modified by adjusting the slurry pH value and using short-chain amphoteric molecules such as butyric acid and propyl gallate.

Wet foam stability is of prime importance when one wants to fabricate porous ceramics with the desired microstructure and mechanical properties. However, relatively little attention has been given to characterization of foam stability. Gonzenbach et al. [16] measured average bubble size by optical microscopy, and Sarkar et al. [17] measured the

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volume variation of wet foam to evaluate foam stability. In bubble size measurements, only local points of the wet foam were observed, and many measurements were necessary to obtain reliable data. In volume measurements, macro-level foam variation cannot reveal the micro-level bubble evolution. Furthermore, the impact of foam stability on the microstructure and properties of the resultant porous ceramics has not yet been systemically evaluated.

Turbiscan stability analyzer has been successfully used to analyze the stability of various concentrated colloidal dispersions and emulsions as well as foams [18–20], but it has not yet been used in the wet foam for porous ceramics. The aim of this research was to evaluate the stability of two kinds of wet foams via stability analysis and to correlate the foam stability with the microstructure and mechanical properties of the resultant porous ceramics. The foams were fabricated by adding an anionic surfactant and a cationic surfactant into alumina slurries with PIBM as both the dispersant and the gelling agent, respectively. The porosity, cell size, and compressive strength of the resultant porous alumina ceramics from different wet foams were compared and correlated with the wet foam stability.

2. Materials and methods

2.1. Materials preparation

A commercial alumina powder (AES-11, Sumitomo Chemical, Tokyo, Japan; average particle size 450 nm) was used as the raw material. PIBM (Isobam104, Kuraray, Osaka, Japan; M.W. 55000–65000) was used as both the dispersant and the gelling agent. An anionic surfactant, triethanolamine lauryl sulfate (EMAL TD, Kao Chemical, Tokyo, Japan; noted as TLS), and a cationic surfactant, dodecyltrimethylammonium chloride (QUARTAMIN 24P, Kao Chemical, Tokyo, Japan; noted as DTAC), were used as the foaming agents. Ultrapure water was used throughout the experiments.

Alumina slurries with 40 vol% solids loading and different contents of PIBM (relative to alumina powder) were prepared by milling for one hour in a planetary ball mill. Wet foams were fabricated via direct foaming with a kitchen mixer at 600 rpm for 4 min after adding TLS and DTAC (relative to water in the slurry) into the slurries, respectively. Then the wet foams were cast into plastic molds. After about 10 h' setting, they were demolded and dried at room temperature for 2 days. Finally, porous alumina ceramics were obtained after sintering at 1550 °C for 3 h in air.

2.2. Materials characterization

The interaction between surfactant and PIBM adsorbed on alumina particles was characterized via the zeta potential of alumina particles in a diluted suspension containing 0.2 g/L alumina powder with a zeta potential analyzer (Zetaplus, Brookhaven, NY, USA).

Wet foams with about 65 vol% air from the slurries with 40 vol% solids loading and 0.25 wt% PIBM were employed to evaluate the foam stability by a stability analyzer (Turbiscan Tower, Formulaction, Toulouse, France). After direct foaming, the wet foam was instantly and carefully transferred into a cylindrical vial with 25.5 mm internal diameter, and the height of the wet foam was 45 mm. An 880 nm source was used as the incident light to scan the sample layer-by-layer with 40 μm intervals from the bottom of the vial. The backscattered light (45° from the incident, noted as BS) was measured to reveal the foam stability (Fig. 1). The data acquisition had 3 min intervals and lasted 22 h. In these wet foams, the bubble size (several ten microns) was about 100 times larger than the particle size (~450 nm). The signal variation caused by the particles in the wet foams was neglected. The BS signal at the beginning of the measurement ($t = 0$) was subtracted from those measured with increasing time, and the results were presented as ΔBS (%) profile versus the sample height at different time. Negative ΔBS values indicated an increase in bubble size. The

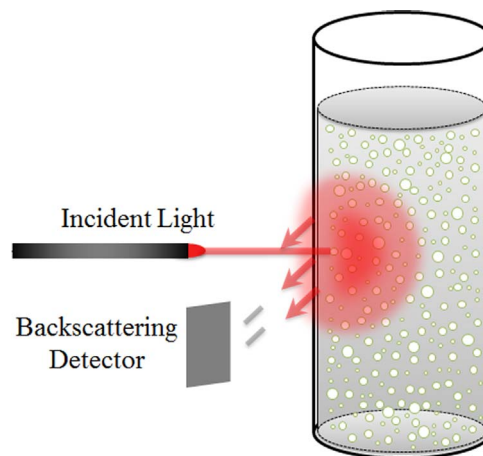


Fig. 1. Schematic illustration of foam stability measurement in Turbiscan stability analyzer.

fluctuation in ΔBS profile indicated heterogeneous bubble sizes in the wet foam. In addition, there is a stability criteria named Turbiscan Stability Index (noted as TSI) [21], which reveals the stability of the sample from a macro level. Lower TSI values indicate more stable wet foam.

The effect of TLS and DTAC on the viscosity and gelation of alumina slurries was characterized via a rheometer (Haake ViscoTester iQ Air, Thermo Electron GmbH, Karlsruhe, Germany) with a parallel plate (20 mm in diameter). Alumina slurries with 40 vol% solids loading and 0.25 wt% PIBM were prepared by ball milling. The TLS and DTAC were respectively added into the slurries and mixed with a glass rod for 10 min followed by degassing to remove air in the slurries. The viscosity of the slurries was determined with a continuous shear rate mode increasing from 1 to 400 1/s. The gelation of the slurries was characterized by measuring the storage modulus of the slurries at 25 °C in oscillatory mode with a frequency of 1.0 Hz and a constant strain of 0.4%.

The relative density and porosity of the porous ceramics were determined via the mass-to-volume ratio and the theoretical density of α -alumina (3.98 g/cm³). Scanning electron microscopy (JSM-6390LV, JEOL, Tokyo, Japan) was used to observe the cell structure of the porous ceramics. The average cell size and size distribution were determined by measuring the diameters of 100 cells in the SEM images. Five cylindrical test pieces with a diameter of 18 mm and a height of 20 mm were drilled out from one porous bulk for compressive strength measurements via a universal testing machine (Instron 5500, Norwood, MA) with a loading speed of 1.0 mm/min.

3. Results and discussion

3.1. Effect of surfactant on zeta potential

Fig. 2 shows the effect of two kinds of surfactants on the zeta potential of alumina particles upon addition of PIBM. In the suspension containing TLS, the zeta potential remained negative when the TLS concentration increased from 0 to 84 ppm. However, in the suspension containing DTAC, the potential signal changed from negative to positive. Taking the suspension with 0.2 wt% PIBM as an example, the zeta potential varied from –23.5 to 11.1 mV with only 10 ppm DTAC addition. The potential variation in the suspension with 0.3 wt% PIBM was similar to that with 0.2 wt% PIBM.

The PIBM used here is an anionic polyelectrolyte and generated carboxyl groups (R-COO⁻) in the alumina suspension adsorbed on alumina particle surface via electrostatic attraction leading to a negative zeta potential of alumina particles. TLS is an anionic surfactant, and therefore the zeta potential remained negative. In contrast, DTAC is a

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