



Contents lists available at www.sciencedirect.com

Journal of the European Ceramic Society

journal homepage: www.elsevier.com/locate/jeurceramsoc



Ultra low-density mullite foams by reaction sintering of thermo-foamed alumina-silica powder dispersions in molten sucrose

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ARTICLE INFO

Article history:

Received 13 September 2016

Received in revised form

11 November 2016

Accepted 11 November 2016

Available online xxx

Keywords:

Foams

Porosity

Strength

Mullite

Insulators

ABSTRACT

Ultra low-density mullite foams are prepared by thermo-foaming followed by reaction sintering of alumina-silica powder dispersions in molten sucrose. The foaming & setting time, foam rise, sintering shrinkage, porosity, cell size and compressive strength are studied as a function of ceramic powder loading, foaming temperature and magnesium nitrate (blowing agent and setting agent) concentration. Phase pure mullite is produced by reaction sintering at 1600 °C. The mullite foams produced without magnesium nitrate have porous struts and cell walls due to improper densification. The magnesium nitrate drastically decreases the foaming & setting time and increases the foam rise and cell interconnectivity. The MgO produced from the magnesium nitrate assists the densification of the mullite as evidenced from the non-porous struts and cell walls at higher magnesium nitrate concentrations. The maximum porosity of 94.92 and 96.28 vol.% achieved without and with magnesium nitrate, respectively, is the highest reported for mullite foams.

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1. Introduction

The importance of mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) foams in the field of high temperature thermal insulation is increasing due to their low thermal conductivity, moderate thermal expansion coefficient, good chemical durability and excellent high temperature mechanical properties [1–4]. These materials also find applications as catalyst support and filter for molten metal [5–8]. The mullite foams are prepared from a colloidal suspension of either mullite powder or its precursors such as alumina and kaolin [5–28]. The reticulated mullite foams are prepared by the polymer foam replication method [5–8]. In this method, the webs of a polyurethane foam template with a suitable pore size are coated with either mullite or its precursors by slurry impregnation method. The slurry coated polymer foam is heat treated for burnout of the polymer foam template and then sintered to produce a ceramic replica of the polymer foam. On the other hand, consolidation of aqueous powder suspensions with starch, wheat flour, and protein is reported for the preparation of mullite foams with cellular structure [9–13]. Gelcasting and freeze casting are also reported for the preparation of mullite foams [14–21]. Fukushima et al. reported

a gelation freezing process for the preparation of macroporous mullite with honeycomb-like pore morphology [22]. The mullite powder suspensions are gelled using gelatin, frozen, dried under vacuum to remove the ice template and then sintered to produce the macroporous mullite. A particle stabilized emulsion-based process has been reported for the preparation of mullite foams by Wang et al. [23]. The particle stabilized emulsions prepared by dispersing octane droplets in aqueous powder suspension cast in a mould are subsequently dried and sintered to produce the mullite foam. Juettner et al. used a process adapted from aerated concrete technology for the preparation of mullite foams from kaolin and aluminium paste [24]. The aqueous kaolin suspension undergoes foaming due to the hydrogen produced by the dissolution of aluminium in water. The aqueous foams were subsequently dried and sintered to produce mullite foams. Kim et al. reported the preparation of microcellular mullite foam from a polysiloxane and alumina using polymer microbeads as a sacrificial pore template [25]. The reaction sintering of silica produced from the polysiloxane with alumina at high temperature produces mullite. Konegger et al. used thermo-foaming of alumina/aluminium powder filled phenyl methyl poly(silsesquioxane) followed by reaction sintering for the preparation of mullite foams [26]. Surfactant assisted foaming kyanite or kaolin suspensions followed by setting using calcium or strontium aluminate cements is used for the preparation of mullite foams [27,28]. The cellular mullite foams produced by most of the reported methods have porosity less than 90 vol.% although the

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gelation freezing route reported by Fukushima et al. and the direct foaming of pre-ceramic polymer filled with alumina/aluminium powder reported by Konegger et al. produces a maximum porosity of 91.5 and 93 vol.%, respectively [24,28].

Recently we have reported a thermo-foaming of powder dispersions in molten sucrose for the preparation of alumina foams [29]. The foaming of the dispersions is due to the bubbles generated by water vapour produced as a result of $-OH$ condensation of sucrose. These bubbles are stabilized by viscosity increase due to the polymerization of sucrose as well as adsorption of alumina particles on the air-molten sucrose interface. The porosity and pore size of the alumina foams are controlled by both foaming temperature and alumina powder to sucrose weight ratio [30]. The advantage of this method is that it does not use any toxic chemical additives or solvent for the preparation of ceramic foam. In the present work, thermo-foaming of alumina-silica powder dispersions in molten sucrose followed by reaction sintering is used to prepare low-density mullite foams. The effect ceramic (alumina + silica) powder to sucrose weight ratio, foaming temperature and magnesium nitrate blowing agent on the foaming & setting characteristics of ceramic powder dispersions in molten sucrose and properties of resultant mullite foams is reported. We achieved a porosity as high as 96.28% by the thermo-foaming method.

2. Experimental

The α -alumina (A16SG, ACC Alcoa, and Kolkata, India) and silica (Alfa-Aesar, USA) powders were of average particles size 0.34 and 2 μm , respectively. The surface area of the alumina and silica powders analyzed using a surface area analyzer (Micromeritics Tristar II, USA) was 10.4 and 2 m^2/g , respectively. The powders were degassed at 200 °C under N_2 atmosphere for 10 h before the analysis. The XRD analysis of silica powder indicates the presence of quartz phase. Analytical reagent grade sucrose, magnesium nitrate [$\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$] and acetone were procured from Merck India Ltd. Mumbai. The sucrose (200 g), alumina powder and silica powder were mixed by planetary ball milling in acetone medium for 2 h at a speed of 200 RPM. The zirconia grinding jar of 500 cm^3 capacity and zirconia grinding media of 10 mm diameter were used. The sucrose to acetone and sucrose to zirconia ball weight ratios were 1:3 and 1:6, respectively. The ceramic (alumina + silica) powder to sucrose weight ratio was varied in the range of 0.8–1.8. The mole ratio of alumina and silica was always maintained at 3:2 as in mullite. The slurry obtained after planetary ball milling was dried in a borosilicate glass tray at 70 °C. The sucrose-alumina-silica mixture thus obtained was heated in an air oven at 185 °C to melt the sucrose. The melt was stirred well with a glass rod to achieve uniform dispersion of alumina and silica particles in molten sucrose. The alumina-silica powder dispersion in molten sucrose was heated in an air oven at temperatures in the range of 120–170 °C for foaming and setting. The solid foam thus obtained was cut into rectangular bodies of 6 cm \times 6 cm \times 4 cm size and heated in an electrically heated furnace in air up to 1600 °C for removal of the organics and reaction sintering. The heating rate used was 30 °C/h up to 600 °C and 120 °C/h from 600 to 1600 °C. A holding time of 2 h was given at 1500 °C and 1600 °C. In order to study the effect of blowing agent, magnesium nitrate was added to the sucrose-alumina-silica mixture before the planetary ball milling. The amount of magnesium nitrate used was in the range of 4–16 wt% of sucrose in the mixture.

During foaming, the foam height was visually observed every 15 min. The time after which there is no further increase in the foam height was measured as the foaming time. After the completion of foaming, the foams were physically inspected at an interval of 1 h. The time required for the soft foam to get transformed into a hard one is determined as the foam setting time. The ratio of the initial

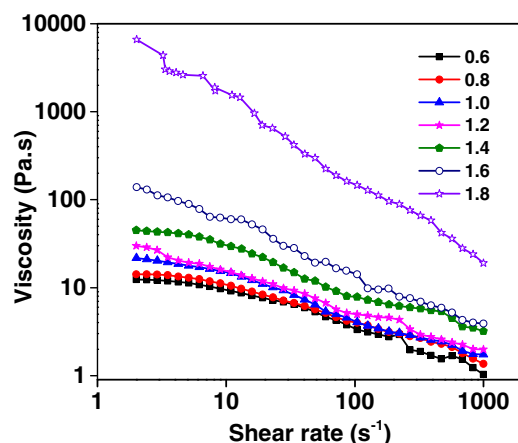


Fig. 1. Effect of $W_{p/s}$ on the viscosity of ceramic powder dispersions in molten sucrose at 140 °C.

height of the powder dispersion to the final height of the solid foam was taken as the foam rise [29,30]. The sintering shrinkage was calculated from the initial and final dimensions of the foam bodies. The porosity of the foam bodies was obtained from the bulk density calculated from their weights and dimensions. Viscosity measurements of the powder dispersion in molten sucrose were carried out at temperatures in the range of 120 °C–150 °C using a Rheometer (Anton Paar MCR 102, Austria). The plate and plate method was used for the viscosity measurements. The zero gap was 0.8 mm. The shear rate used was in the range of 2–1000 s^{-1} . The XRD analysis was carried out using an X-ray diffraction analyzer (X'pert Pro, Philips). The average cell size of the mullite foams was measured on the magnified image of the foam bodies observed using a vision inspection system with a CCD color camera (Vision 300 GL; TESA Technologies, Switzerland). The microstructure of the mullite foam bodies were observed on the fractured surfaces using a scanning electron microscope (SEM; FEI Quanta FEG200, Hillsboro, OR). The compressive strength measurement of the mullite foams was carried out using a Universal Testing Machine (Instron 5984; Instron, Norwood, MA) at a crosshead speed of 0.5 mm/min using samples of 24 mm \times 24 mm \times 12 mm size (ASTM standard C365/C365M-05).

3. Results and discussion

3.1. Effect of ceramic powder to sucrose weight ratio

A homogeneous dispersion of alumina and silica powders in molten sucrose is formed when the sucrose-alumina-silica mixtures heated in a borosilicate glass tray at 185 °C due to the hydrophilic interaction between sucrose hydroxyls with the alumina and silica particle surfaces. The dispersions, in general, show high viscosity with shear thinning flow behavior. The high viscosity is due to the strong intermolecular hydrogen bonding interaction between the sucrose hydroxyls. The viscosity and shear thinning behavior of the dispersions increases slowly with an increase in ceramic powder (alumina + silica) to sucrose weight ratio up to 1.6 and thereafter rapidly. Hereafter ceramic powder to sucrose weight ratio is denoted as $W_{p/s}$. Fig. 1 shows viscosity versus shear rate plot of alumina-silica powder dispersions in molten sucrose measured at 140 °C. The increase in viscosity and shear thinning character with an increase in $W_{p/s}$ is due to an increase in the particle to particle interaction. The remarkably high viscosity observed at $W_{p/s}$ of 1.8 indicates the attainment of maximum powder loading limit.

The dispersions undergo slow foaming when heated in an air oven at 140 °C by the nucleation and growth of bubbles in the viscous dispersions due to the water vapour generated as a result of

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