J Mater Sci (2009) 44:4507–4509
DOI 10.1007/s10853-009-3693-3

LETTER

Preparation of γ-alumina ceramic foams employing hydrophilated polyester polyurethane sponges

Angela B. Sifontes · Marianis Urbina · Frank Fajardo · Luis Melo · Marta Mediavilla · Nereida Carrión · Joaquín L. Brito

Received: 14 May 2009 / Accepted: 15 June 2009 / Published online: 30 June 2009

Single phase γ-Al_2O_3 ceramic foams with high surface area (179 m^2/g) and porosity (67%) were prepared by the polyurethane sponge replica method from slurries containing γ-Al_2O_3.

Ceramic foams are highly porous brittle materials with large voids (cells) [1]. These cellular ceramics are special classes of porous materials comprised of cells with size ranging from a few microns to a few millimetres, where the cells can be surrounded by ceramic walls or contain solid material at only cell edges (struts), thus creating an interconnected structure (open cell foam) [1–3]. Ceramics foams are finding increasing applications as catalytic supports, ceramic membranes, sensors, filters, thermal and acoustic insulators, due to their properties such as low density, low thermal conductivity, high temperature stability, and high resistance to chemical attack [4]. Due to the high calcination temperatures employed in the conventional preparation of alumina foams, α-Al_2O_3 is always obtained [5]. For catalytic applications, post-treatments such as washcoating of the α-Al_2O_3 monoliths with suspensions of γ-Al_2O_3 or sol–gel deposition procedures can be employed in order to obtain materials with higher surface areas [6]. However, when the foam is coated with γ-alumina or any hydrated form of alumina, the maximum useful temperature is limited to approximately 700 °C to avoid loss of surface area as the result of the transformation to more crystalline forms of alumina [7]. To our best knowledge, there has been only one, very recent report on the preparation of single phase γ-Al_2O_3 ceramic foam [8]. In the present work, the preparation of single phase γ-alumina foams of high surface area employing the polyurethane sponge replica method is reported, which consists of dipping the polymeric foam into a slurry containing the ceramic precursors, followed by a thermal treatment that leads to the burning out of the sponge and to the pressureless sintering at elevated temperatures of the ceramic skeleton [9]. It is expected that the meso/macroporous characteristics of the ceramic foams will parallel those of the templating organic material.

A commercial polymeric sponge (ACE Hardware Corp.), corresponding to hydrophilated polyester polyurethane with pore density of 122 ppi, was tested as template for the preparation of ceramic foams. γ-Alumina which has 283 m^2/g BET area, 0.53 cm^3/g pore volume, and mean pore radius of 7.6 nm, was prepared in this laboratory following the method reported by Xu et al. [10], and was used in the present study for preparing the slurries employed in the impregnation process of polyurethane sponge. Slurries were prepared using the following procedure: 21.8 g of the synthesized alumina was mixed with 24 mL of deionized water and 5 mL of concentrated HCl. The viscosity of the slurry prepared with γ-alumina at pH 3.5 was 103×10^{-4} Pa s. The alumina coating was applied by repeated immersion-drying cycles of the substrate (PU sponge) in the alumina slurries. A minimum of four immersion-drying cycles were

---

Electronic supplementary material The online version of this article (doi:10.1007/s10853-009-3693-3) contains supplementary material, which is available to authorized users.

A. B. Sifontes · M. Urbina · F. Fajardo · L. Melo (✉) ·
M. Mediavilla
Facultad de Ingeniería, Universidad Central de Venezuela,
P.O. Box 48.057, Caracas 1041-A, Venezuela
e-mail: meloher@gmail.com

N. Carrión
Centro de Química Analítica, Facultad de Ciencias, Universidad
Central de Venezuela, P.O. Box 47102, Caracas 1041-A,
Venezuela

L. Melo · J. L. Brito
Centro de Química, Instituto Venezolano de Investigaciones
Científicas, P.O. Box 20632, Caracas 1020-A, Venezuela

required in order to attain self supporting ceramic cylinders that could withstand the calcination treatments without collapsing. Finally, the coated cylinder was calcined at 600 °C for 6 h.

Different analysis techniques were used to characterize the ceramic samples: X-ray diffraction patterns were collected using a Siemens D-5005 diffractometer, employing CuKα radiation in the 2θ range between 5° and 70°. The operating voltage and current were 40 kV and 45 mA. Photographs of some samples were taken with a digital camera (Casio, Japan). Scanning Electron Microscopy micrographs were obtained out with a Hitachi S-2400 instrument. The textural properties of the ceramic samples were characterized by N_2 adsorption porosimetry (Micromeritics, ASAP 2010).

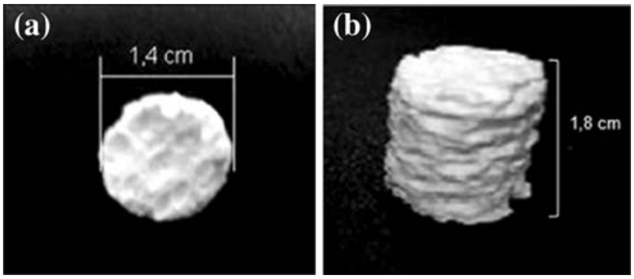


Fig. 1 Photographs of γ-alumina foams prepared **a** top view; **b** side view

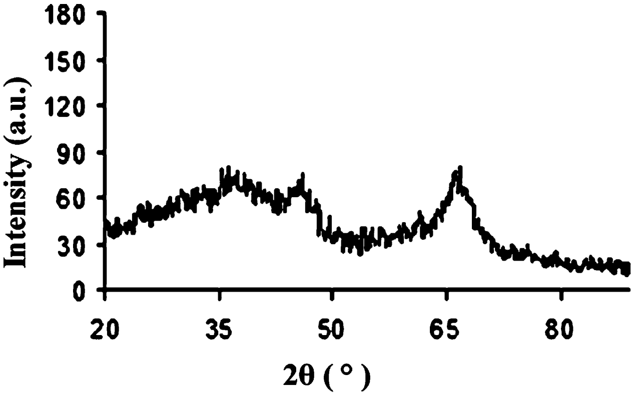


Fig. 2 XRD patterns of γ-alumina ceramic foam prepared from synthesized alumina

Calcined alumina foams (Fig. 1) showed mechanical strength (resistance to breakup/crushing) of the order of 315 kPa. The average porosity of the alumina foams was close to 67%.

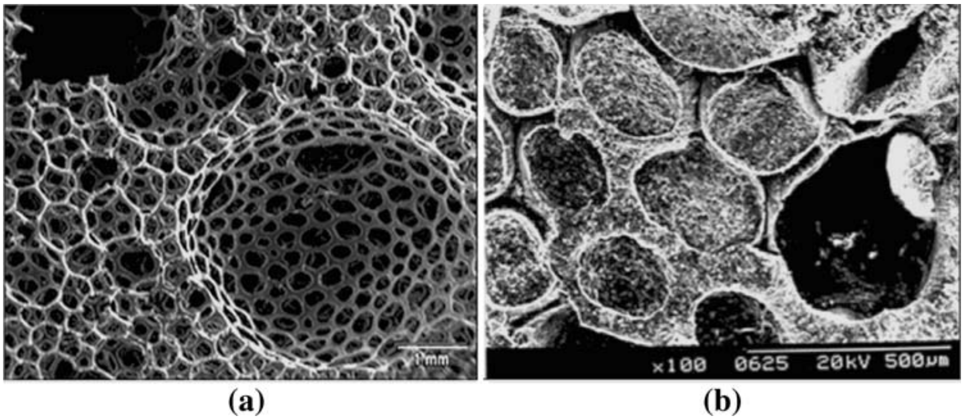
While these mechanical strength values are not particularly high if compared with data reported for α-alumina foams, it must be taken into account that the present ceramic materials are calcined at relatively low temperatures in order to keep the alumina in the γ-phase.

The initial alumina keeps their crystalline properties after preparation of the ceramic structures as shown by the XRD patterns corresponding to the γ-Al_2O_3 phase (Fig. 2) [10].

The results of textural characterization of the prepared γ-alumina foam reports the BET surface area of 179 m^2/g, total pore volume (obtained from the N_2 physisorption isotherm at 0.99 relative pressure) of 0.48 cm^3g^{-1}, and mean pore radius (from BJH method) of 9.8 nm. The N_2 physisorption isotherms (Fig. S1) of the ceramic correspond to type IV of the BET classification, characteristic of mesoporous materials [11].

Figure 3 presents SEM micrographs of PU sponge (Fig. 3a) and of the alumina foam (Fig. 3b) where it can be appreciated the presence of regular shaped macropores with some smooth surfaces and channels. The diameters of the macropores in Fig. 3b (typically between 200 and 400 µm) agree with the more frequent cell size of the polymeric foam in Fig. 3a. The combined results of the present study demonstrate the feasibility of producing γ-Al_2O_3 ceramic foams with high surface areas and porosities, showing relatively good mechanical strength, which could have potential interest for applications in adsorption and catalysis.

Fig. 3 SEM micrographs of **a** polyurethane sponge, **b** γ-alumina ceramic foam



In summary, the synthesis method employed in the present work allowed to obtain γ-Al_2O_3 ceramic foams of high surface area (179 m^2/g) and porosity (67%). The mechanical strength of the ceramic foams seems to depend on the composition of the slurry and the characteristics of the polymeric sponge used as template. The phase composition and structural properties of the alumina employed were not changed after its use in the preparation of the alumina foams.

Acknowledgement The authors would like to thank CDCH – UCV (PG: 08-00-6075-2005) for supporting this work.

References

1. Rocha RM, Moura EAB, Bressiani AHA, Bressiani JC (2008) J Mater Sci 43:4466. doi:10.1007/s10853-008-2654-6
2. Wang C, Wang J, Park CB, Kim YW (2007) J Mater Sci 42:2854. doi:10.1007/s10853-006-0229-y
3. Yu J, Sun X, Li Q, Li X (2007) J Mater Sci 42:8215. doi: 10.1007/s10853-007-1696-5
4. Ávila P, Montes M, Miro EE (2005) Chem Eng J 109:11
5. Han YS, Li JB, Chen YJ, Wei QM (2002) J Mater Process Technol 128:313
6. Twigg MV, Richardson JT (2002) Chem Eng Res Des 80:183
7. Han YS, Li JB, Chen YJ (2003) Mater Res Bull 38:373
8. Zhang Y, Zhao CY, Liang H, Liu Y (2009) Catal Lett 127:339
9. Acchar W, Ramalho EG, Souza FBM, Torquato WL, Rodrigues VP, Innocentini MDM (2008) J Mater Sci 43:6556. doi:10.1007/s10853-008-2585-2
10. Xu BJ, Xiao TC, Yan ZF, Sun X, Sloan J, González-Cortés SL, Alshahrani F, Green MLH (2006) Microporous Mesoporous Mater 91:293
11. Gregg SJ, Sing KSW (1982) Adsorption, surface area and porosity, 2nd edn. Academic Press, London, pp 111, 287