Rare earth porous membranes by colloidal processing

S.C. Santos, S.R.H. Mello-Castanho

Nuclear and Energy Research Institute – IPEN/São Paulo, Brazil.

silascs@ipen.br, srmello@ipen.br

ABSTRACT

Rare earth porous membranes (RePM) were manufactured by colloidal processing and impregnation method. Ceramic suspensions based on rare earth concentrated (ReC) with 25vol% were prepared. The rheological behavior of ceramic suspensions as function of solid load and binder concentration was evaluated by flow curves with shearing rate interval from 0 to 1000s^{-1}. A nylon-cotton netting (NC) was selected as template. Impregnated NC templates were subjected to a careful thermal treatment seeing that burning off the organic template could induce sensible residual stress and disrupting the ceramic structure. Sintering at 1600ºC/15h the morphology of RePMs were like NC templates and presented a good handle strength.

Key words: rare earth, rheology, suspension, impregnation method, porous ceramic.

1 . Introduction

In traditional ceramic processing, porous structures have been avoided in ceramic components inasmuch as of their inherently brittle nature, but in the last years, an increasing number of applications that require porous ceramics have appeared. Such applications include gas burners[1], radiant burners[2], filters of molten metals, high-temperature thermal insulation, support for catalytic reactions, etc.

There are many different methods to manufactured ceramic foams, such as replica[3], sacrificial template and gel casting[4]. The first one is considered the cheapest method for producing porous structures. The great flexibility of this method is the fact that it is applicable to any ceramic material that can be appropriately dispersed into a colloidal suspension[5,6,7]. Therefore the knowledge about colloidal processing is also important. This process results in a more uniformed particle packing, better microestrutural control while firing and high mechanical strength of ceramic.

The aim of this work consists in adequating suspensions rheology of ReC to produce porous membranes by replica method using nylon-cotton netting as template.

2. Experimental

The starting material for this study was a rare earth concentrate (ReC) achieved from a rare earth carbonate (Nuclemon - Nuclebrás de Monazita e Associados, Brazil). In our prior work[8] was presented the conditions of ReC powders processing. ReC powders presented the following characteristics: mean particle size (d_{50}) of 1,57 µm, specific surface area of 37.62 $m^2.g^{-1}$, density of 6.83 $g.cm^{-3}$. .

ReC aqueous suspensions were prepared with solids concentration from 25%vol. In order to stabilize the suspensions an ammonium polyacrylate (PAA) was used as polyelectrolyte/dispersant (Duramax D-3005, Rohm and Haas, USA) with concentration at 1wt.% (referred to dry solids). Basicity of the medium was provided by adding tetramethylammonium hydroxide (TMAH), supplied by Aldrich-Chemie (Germany). Carboxymethyl-cellulose (CMC) were added to the suspension (based on suspension weight) for enhance the adhesion of the ceramic suspension on the support material surface. Therefore its effectiveness on the rheological behavior was also evaluated.

Rheological behavior of suspensions was performed with a rheometer (Haake RS600, Thermo Scientific, Germany). The sensor system consisted on a double cone rotor and a stationary plate (DC60/1º). Characterizing the suspensions stability the flow curves were determined in a control rate mode (CR). Measurements were performed by increasing the shear rate from 0 to 1000 s^{-1} in 5 min, maintaining at 1000 s^{-1} for 2 min and returning to 0 in 5 min. Temperature was maintained constant at 25℃ during these experiments. For each CR cycle 200 points were measured. In order to decrease the agglomerates size, the suspensions were stirred for 3 min at high shear rate

(Quimis, Q-252-K18). After that, they were subject to mechanical mixing for 30 min to promote suspensions homogeneity (HeidolpH, mod. RZR1).

Nylon-Cotton nittings (NC) were submerged in the suspension while it fill all the pores. The samples were dried at room temperature for 24 h. The impregnated NCs were subjected to a heat treatment (at 1ºC min^{-1} to 800) in the electric furnace (EDG, EDG 1800), to extract the support material and others additives. Finally the samples were sintered in air atmosphere, at 1600ºC/15h with 1ºC min^{-1} as heating rate.

3. Results and discussion

As the binder (CMC) is added in the suspension, it's observed an increase on the viscosity of the system. This effect is associated to larges molecules of CMC that recover the particles flocculating the system. When the amount of CMC is 0.5wt.% the suspension shows a greater thixotropy (Figure 1). It's should be due to the breaking down of agglomerates and rearrangement of the particles during the shear rate. For impregnation method, this rheological behavior is desired, so that the suspension is fluid enough to enter, fill, coat uniformly the template surface and under static condition, its viscosity is high to remain on the nitting[9] . Figure 1 shows at 1000s-1 the shear stress shifted from 15Pa (0.0wt% of CMC) to 200Pa. As 0.7wt% was added, the increase of shear stress was from 200Pa to 350Pa. It can be associated to many agglomerates formed as result of higher concentration of CMC. Suspensions with this behavior are not desired for impregnation method, thus they neither cover, or fill uniformly the template surface and cavity. Based on these results, the suspension with 0.5% of CMC as selected for replica tests. Comparing the flow behaviors of suspensions to known rheological models, a curve related to 0.0wt% of CMC corresponded to Newnton model, besides 0.5wt% and 0.7wt% curves to Casson Linear model[[10]]

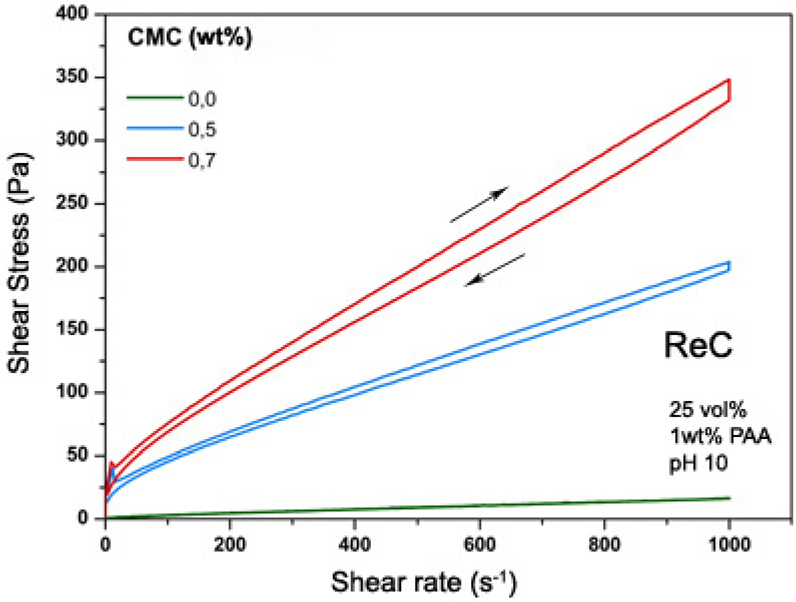

Figure 1 - Flow curves of ReC suspensions as function of CMC concentration

In Figure 2 is shown the ceramic membrane manufactured by impregnation method. The reticulated ceramic maintained the form of the template (nylon-cotton nitting), where can be seen cellular structures with thin struts and interconnected open pores.

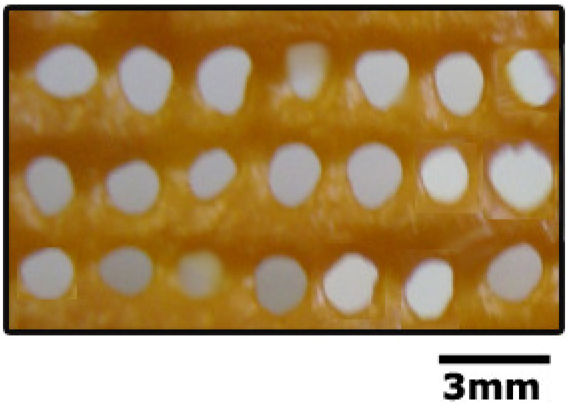

Figure 2 - ReC membrane manufactured by impregnation method

4. Conclusions

Ceramic membranes of ReC was manufactured by impregnation method. The suspension with 25vol.% and 0,5wt.% of CMC exhibited appropriate rheological characteristics for impregnation. It shows the importance of the prior rheological study, where the stability of the suspensions was evaluated by flow curves. Sintered samples at 1600C/15h showed the same morphology of nitting template, without apparent cracked and adequated handle strength.

Acknowledgements

The authors are gratefully acknowledged to the following institutions: The State of São Paulo Research Foundation (FAPESP), The National Council for Scientific and Technological Development (CNPq) and mainly to High Degree People of Improvement Coordination (CAPES) for financial scholarship support of the student Silas Cardoso dos Santos.

References

1 . WOOD, S.; HARRIS, A.T. Porous burners for lean-burn applications. ***Progress in energy and combustion science***., v. 34, p. 667-684, 2008.

2. GÓMEZ, S. Y.; ESCOBAR, J. A.; ALVAREZ, O. A.; RAMBO, C.R.; NOVAES DE OLIVEIRA, A.P.; HOTZA, D. ZrO2 foams for porous radiant burners. ***Journal of Materials Science***.,v, 2009.

3. NORA, M. A. A. M.; HONGB, L. C.; AHMADB, Z. A.; MD AKIL, H. Preparation and characterization of ceramic foam produced via polymeric foam replication method. ***Journal of materials processing technology***. v. 2 0 7, p. 235–239, 2008.

4. TAKAI, C.; TSUKAMOTO, M.; FUJI, M.; TAKAHASHI, M. Control of high solid content yttria slurry with low viscosity for gelcasting. ***Journal of Alloys and Compounds***. v. 408, p. 533–537, 2006.

5 . JIN, L.; MAO, X.; WANG, S.; DONG, M. Optimization of the rheological properties of yttria suspensios. ***Ceramics International***., v. 35, p.925-927, 2009.

6. MEWIS, J.; WAGNER, N. J. Thixotropy. ***Advances in Colloid and Interface Science***., v. 147-148, p. 214-227, 2009.

7 . LANGE, F. F. Shape forming of ceramic powders by manipulating the interparticle pair potential. ***Chemical Engineering Science***., v. 56, p. 3011-3020, 2001.

8 . SANTOS, S. C. Coloidal processing of ceramic components for gas burners. Master Dissertation, Nuclear and Research Institute (IPEN), São Paulo University (USP), São Paulo, 2010.

9. ZHU, X.; JIANG, D.; TAN, S. The control of slurry rheology in the processing of reticulated porous ceramics. ***Materials Research Bulletin***., v. 37, p. 541-553, 2002.

10. BOTELLA, R. M. *Reologia de suspensiones cerámicas*, 2005.