ACTIVITY REPORT 2011

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2011. The gel filing process of the polycarbonate membranes pores.

Preliminary preparation tests have be performed on the filling process of the nanoporous polycarbonate membranes, gel solidification, drying and heat treatments. (2 man x 2 months)

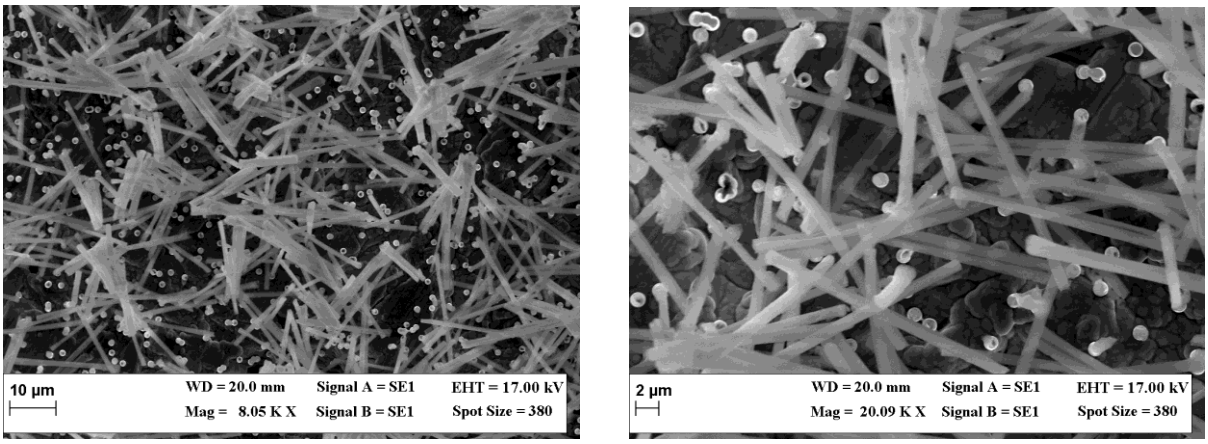

Figure 1. Electron microscopy (SEM) images of the xerogel rods recorded at two different magnifications.

For the preparation of the xerogels rods we used synthesis from liquid solution of organometallic tetraethylorthosilicate (TEOS) precursor corresponding to a chemical reaction implying metal alkoxides and water in an alcoholic solvent. Xerogel microrods have been made by using as template a track etched polycarbonate membrane with a thickness of 30□m, pore diameter of about 1µm and 10^8 cm^{-2} pore density; prior the preparation of the microrods it was covered by sputtering with a thin gold film. This film was thickened by electrochemical deposition of a copper layer. Subsequently, the polycarbonate membrane was clamped in a synthesis vessel with the pores exposed to the liquid gel where was kept for 15 min. and then dried for 48h at room temperature. After the drying process the polymer membrane was dissolved in dichloromethane and the drying was continued for several months. During drying followed by annealing at high temperature, the liquid located within the gel pores is released and we have observed the volume contraction of the rods to about 35% of the initial value which is restricted mostly on the rod length. However a cracking due to the stress arising from the volume contraction was not observed the rods retaining their morphology. It is known that in thin solid films (0.5µm) the stress generated by one dimensional shrinkage is still of low magnitude not to cause cracking, i.e. the energy required to extend a crack is greater than the energy gained from relief of stress near the crack. We

noticed that the surface of the glass-ceramic microrods is rough and not smooth as in the xerogels. It is likely that the liquid was not fully released from the pores during drying and it was released rapidly and energetically during annealing.

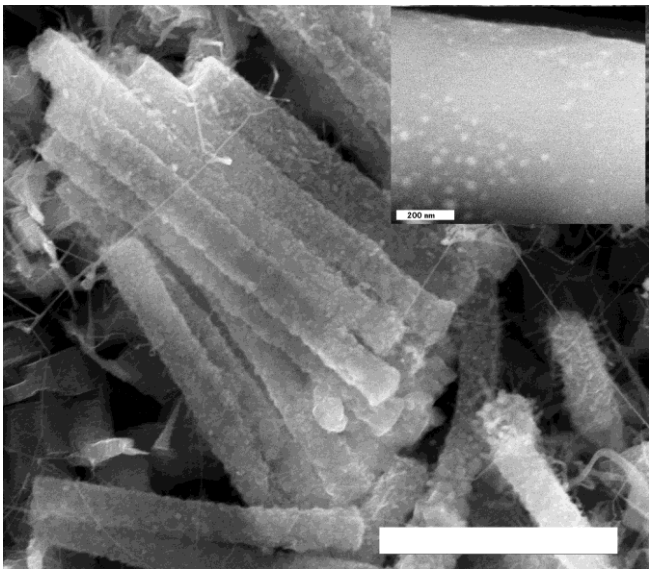

Figure 2. Electron microscopy (SEM) images of the glass-ceramic rods (the white bars correspond to 5 μm): the inset shows nanostructures of about 30-40nm inside such a glass-ceramic microrod.

In conclusion, we described the use of sol-gel chemistry within the pores of a polycarbonate template membrane to prepare luminescent xerogel and oxy-fluoride glass ceramic rods and we have performed their structural and optical characterization. The microrods are obtained after annealing at high temperatures and preserve the morphology of the parent xerogel rods

References:

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[2] Secu, C.E; Polosan, S and Secu, M, Journal of Luminescence, Volume 131, (2011) 1747

[3] M. Nogami, N. Hayakawa, N. Sugioka, Y. Abe, J. Non-Cryst. Solids 197 (1996), 73.