11th International Conference on Advanced Materials
Rio de Janeiro Brazil
September 20 - 25
ICAM2009

Mullite nanostructured synthesis in thin film-like shape by employing liquid crystal as template

A. S. Pascoli(1)* and E. Y. Kawachi(1)

(1) EAM-M, Instituto Tecnológico de Aeronáutica, Brasil, e-mail: dripascoli@ita.br.
* Corresponding author.

Abstract – Mullite was obtained from sol-gel process and the nanostructuration was performed by means of a liquid crystal system. Tetraethylorthosilicate (TEOS) and aluminum nitrate nonahydrated (ANN) were used as mullite precursors, besides non-ionic surfactant Renex® and $HNO_{3(aq)}$ in the following molar ratios: 1.0, 5.6, 1.3, and 51.2. XRD results have shown orthorhombic mullite formation at 1200ºC indicating good homogeneity of precursors. Conversely, SAXS results have shown the negative influence of TEOS and ANN inclusion in the liquid crystal microstructure yielding a destructuring that might have been caused by both non-effective removal of ethanol formed during TEOS hydrolysis and the drying temperature.

Mullite ($3Al_2O_3.2SiO_2$) is an extremely versatile ceramic material due to its properties such as: high resistance of thermal variation, chemical attacks and wearing; low thermal expansion coefficient and dielectric constant and good mechanical resistance. The most stable phase of mullite is the orthorhombic, which is normally obtained at temperatures higher than 1200ºC, depending on the homogeneity of precursors reagents.

The purpose of this work was the mullite nanostructured synthesis. TEOS and ANN were used as precursors of silica and aluminum respecting the mullite stechiometry. In addition to the precursors, non-ionic surfactant Renex®100 and aqueous solution of nitric acid (HNO_3 1.15 mol L^{-1}) were used keeping the 1:1 mass ratio for liquid crystal obtainment[1]. The purge of ethanol generated during TEOS hydrolysis was performed by using a drier connected to a vacuum system during 1 hour in order to try and maintain the liquid-crystal phase integrity.

After drying at 30ºC the sample in a gel-like form was calcinated at 1200ºC for 2 hours yielding a porous solid. The sample diffractogram depicted in Figure 1 shows mullite formation. In the same figure the splitting of the located at ~26ºC is shown in detail characterizing orthorhombic mullite phase comprising 60% of Al_2O_3 in a molar basis[2]. This result shows that the liquid crystal structure does not interfere in mullite formation. In contrary, it actually might have contributed for a greater precursors homogenization and thus in orthorhombic mullite formation at a relatively low temperature.

Figure 2 depicts the SAXS patterns for the samples made of Renex®100/$HNO_{3(aq)}$ and Renex®100/$HNO_{3(aq)}$/TEOS/ANN. Those results show that the binary system forms a hexagonal mesophase. The inclusion of TEOS and ANN destabilize the system though. A possible deficiency of removal of the ethanol formed during the TEOS hydrolysis along with the drying temperature at 30ºC might have played a determinant role in destructuring the liquid crystal system.

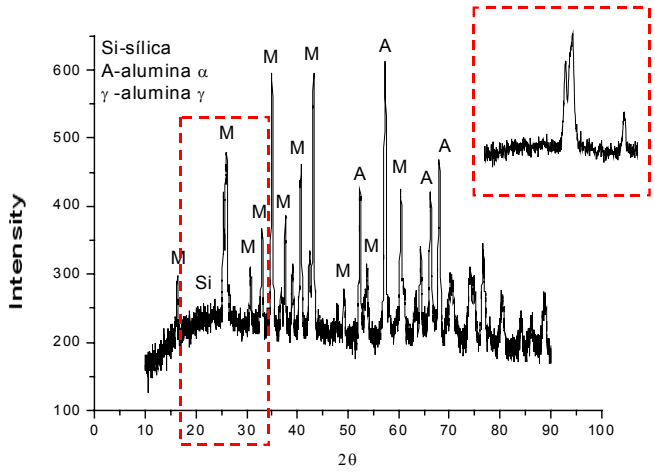

Figure 1: XRD pattern of R®100/$HNO_{3(aq)}$/TEOS/ANN, after calcination at 1200°C for 2 hours.

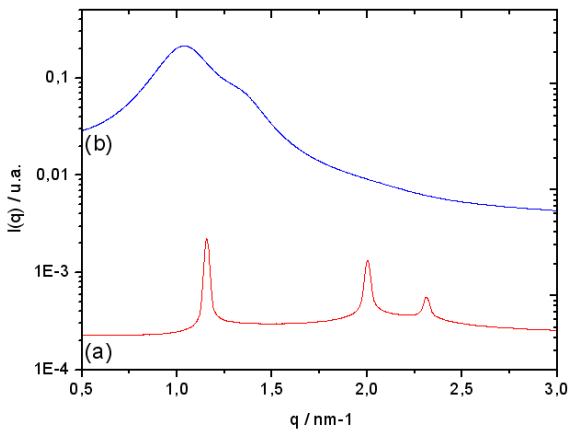

Figure 2: SAXS patterns of (a) Renex®100/H_2O and (b) Renex®100/$HNO_{3(aq)}$/TEOS/ANN.

[1] G.S. Attard, J.C. Glyde, C.G. Goltner; Nature, 378, 366-367, 1995.
[2] C.C. Osawa, C.A. Bertran; J. Braz. Chem. Soc., 16 (2), 251-258, 2005.