Condensed Matter Physics

Fernanda Margaça

The Group's main field of research is the development and characterisation of materials with new or improved properties. To this end, radiation is used as a tool to investigate the structure and to induce structural modifications in particular samples. Special polymeric materials have been investigated in collaboration with groups from the Universities of Aveiro and Coimbra, Laboratoire Léon Brillouin (CEA-CNRS-Saclay), KFKI, Budapest, and the Budapest Neutron Centre. The main effort has been made to characterise the hybrid materials and co-polymers prepared by gamma irradiation using the ^{60}Co source of UTR.
The systems studied involved hybrids prepared from mixtures of a polymer (PDMS) and different metallic alkoxides. Characterization of selected samples prepared by gamma irradiation continued, making use of a wide range of techniques, most of them available at ITN.

During 2008 the main work on the development of new copolymers (HEMA grafted on LPDE thin films) suitable for bioapplications was concluded. The sample preparation conditions were correlated to the grafted material structure and the hydration level achieved by the final product, and toxicity studies were finished. Tests of Biocompatibility (haemolysis and tromboresistance) were completed. Work on this system has been summarised in a PhD thesis that was submitted to the University of Lisbon in December 2008.

The Group had also been active in the area of hardware and software instrument development, with emphasis in the design, construction, and testing of systems and components for neutron beam work. Shortage of resources, both human and financial, has been preventing the proper development of the activity in this area. In the last two years the human resources both the staff and the students have been considerably reduced. As concerns the financial resources, the neutron scattering instruments situation became particularly difficult. This type of instruments installed at any reactor source require a set of equipments and components that are generally expensive items, to be able to tailor the neutron beam, to eliminate the undesired background radiation and to collect the data, in a suitable and efficient way. This becomes particularly critical when the source is a low flux reactor.

As concerns the small angle scattering instrument, extensive work had been done to optimise its installation at RPI. However the actual signal to noise ratio was found to be too low to carry out meaningful measurements. During 2007 the reactor core was converted from HEU to LEU which further contributed to lower that ratio value. In 2008 it became clear that no further progress could be made to overcome this problem unless a few technical solutions were implemented. The matter of fact was presented to the Board of the Institute.

Given the considerable funding required and the current financial difficulties of the Institute, the decision was made to temporarily set aside the project of the small angle neutron scattering instrument at the Portuguese research reactor. Characterization of the prepared materials using small angle neutron scattering will continue to be carried out in foreign neutron scattering centres.

Three proposals for small angle scattering measurements have been presented to European neutron scattering centres. Those proposed at the Budapest Neutron Centre in Hungary and at the Laboratoire Léon Brillouin in France have already been approved.

Researchers

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Progress in the characterization of hybrids obtained by γ-irradiation

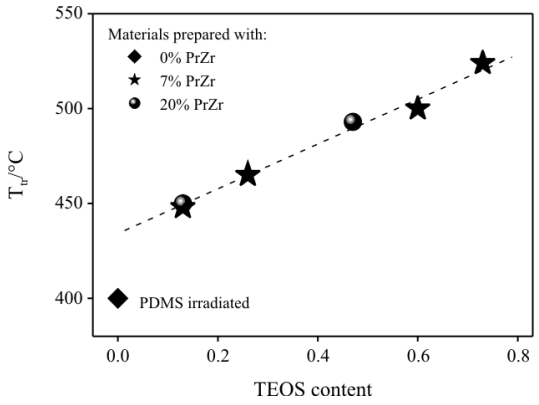
F.M.A. Margaça, L.M. Ferreira, A.N. Falcão and I.M.M. Salvado

Objectives

To characterize organic-inorganic hybrid materials prepared using the gamma-irradiation from the ITN ^{60}Co source from a mixture of composition x polydimethylsiloxane (PDMS) (100-x) Alkoxide, in order to investigate the microstructure and properties.

Results

The alkoxides used as precursors were tetra orthosilicate (TEOS) and Zr-isopropoxide (PrZr). All the obtained materials were monolithic and transparent after drying in air. They have been studied using different techniques. The detailed measurements and results have been presented and published [1-4]. In the following a few of those results are shown.



Figures 1 show the behaviour of the hybrid material rupture temperature, T_{tr}, which is associated to the breaking of the polymer chains into volatile fragments and marks the rupture of the structure. It was found that this temperature increases linearly with the TEOS content in the hybrid materials prepared with PrZr.

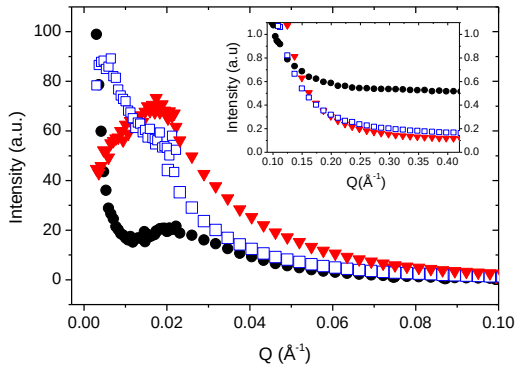


Figure 2 shows Small Angle Neutron Scattering (SANS) data for the 20%PDMS+73%TEOS+7%PrZr sample dried at room temperature (●) and after heat-treatment at 450 ºC (□) and 600 ºC (▼). The insert shows the intensity at large Q before subtracting the incoherent scattering.

The SANS data revealed the mass fractal nature of the inorganic oxide regions structure. It also showed that, although the presence of PrZr is required to obtain a stable hybrid, the increase in its content leads to a less dense mass fractal oxide structure. The slope values of the spectra linear descents revealed that the oxide domains develop via reaction-limited aggregation. The size and the fractal density of the inorganic oxide regions increase with the TEOS content.

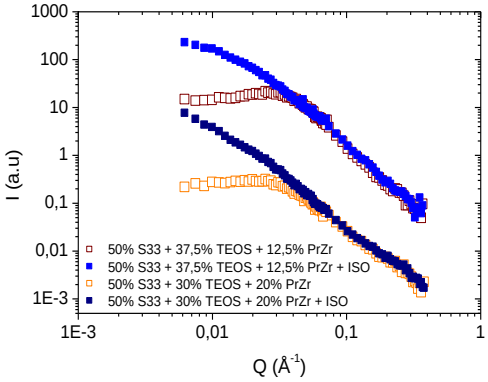


Figure 3 shows the SANS intensity obtained from samples with 50%PDMS and varying contents of TEOS and PrZr prepared with and without further addiction of isopropanol. It was observed that the further addition of isopropanol in the preparation stage causes the inorganic fractal structure to extend over a much larger volume in the samples so prepared. This effect needs further investigation and work is in progress.

Published Work

S.R. Gomes, F.M.A. Margaça, L.M. Ferreira, I.M. Miranda Salvado, J.P. Leal, C. Marques, E. Alves, L.M. Ferreira and A.N. Falcão, Elemental and RBS analysis of hybrid materials prepared by gamma irradiation, Nucl. Inst. Meth. Phys. Res. B, 266 (2008) 288-294.

S.R. Gomes, F.M.A. Margaça, D. Faria Silva, L.M. Ferreira, I.M. Miranda Salvado, A.N. Falcão, Novel Way to control PDMS Cross-linking by Gamma Irradiation, Nucl. Inst. Meth. Phys. Res. B, 266 (2008) 1105-1108.

S.R. Gomes, L.M. Ferreira, F.M.A. Margaça, I.M. Miranda Salvado and A.N. Falcão, Thermal analysis of hybrid materials prepared by gamma irradiation, J. Therm. Anal. Cal., Online First: Oct. 2008, Doi:10.1007/s10973-008-9223-8

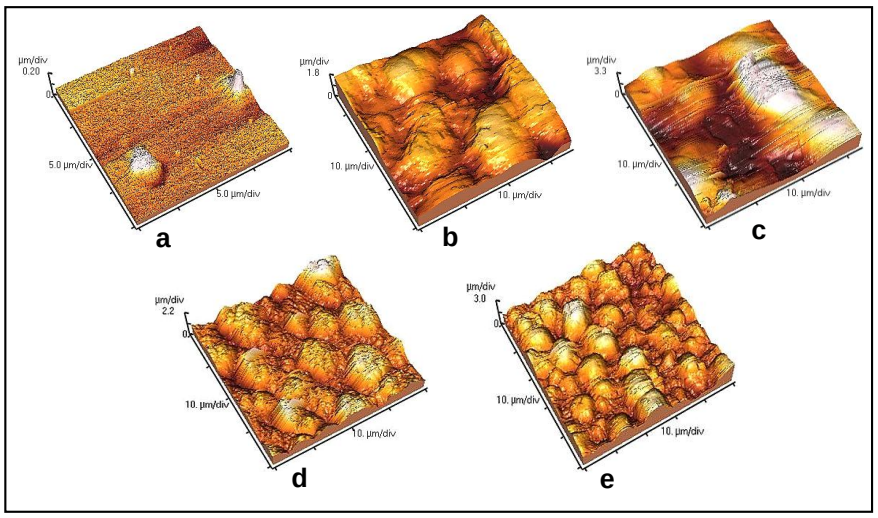
S.R. Gomes, F.M.A. Margaça, I.M. Miranda Salvado, A.N. Falcão, L. Almasy and J. Teixeira, SANS investigation of PDMS hybrid materials prepared by gamma-irradiation, Nucl. Inst. Meth. Phys. Res. B 266 (2008) 5166–5170.

Modification of Polyethylene by Grafting Copolymerization Induced by Gamma Radiation – Its Application in Biomaterials Field

L.M.Ferreira, A.N. Falcão, J.P. Leal, M.H. Gil[1], L.Cerqueira, A. Rodrigues, C. Marques, H. Ferronha[2] and I. Correia[2]

The R&D work on the preparation of a new grafted copolymeric polyethylene based materials by gamma irradiation technique, using the ITN ^{60}Co source, was concluded. The PE-g-HEMA films prepared exhibit a good mechanical behaviour associated with a maximum hemolytic effect of 2%, which, according to ASTM F 756-00 standard, classify them as "nonhemolytic" material and therefore suitable for direct contact with blood. The copolymeric films reveal other good properties such as their porous and rough surface.

These are essential conditions for cell bio-interaction for linkage and/or cell proliferation. Furthermore, these are important characteristics too for physical or chemical immobilization of active biomolecules (enzymes, drugs, tromboresistentes agents, etc).



The work developed in this research project is reported in detail in the PhD thesis of Luís M. Ferreira, which was submitted to University of Lisbon in December 2008. The descriptive documentation process for registration of a National Patent for the new polymeric material is under way.

[1]Univ. Coimbra / Dept. Chemical Engineering.
[2]LNIV (National Laboratory of Veterinarian Research).