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 special samples. Special polymeric materials are currently investigated in collaboration with groups from the Universities of Aveiro and Coimbra, the University of Sophia, Bulgaria, Laboratoire Léon Brillouin (CEA-CNRS-Saclay), KFKI, Budapest, and the Budapest Neutron Centre. During 2006 the main effort was put into the processing and characterization of hybrid materials and co-polymers prepared by gamma irradiation using the ^{60}Co source of UTR.

The systems studied were: (i) hybrids prepared from mixtures of a polymer (PDMS) and various alkoxides, with emphasis in the thermal characterization of various samples prepared using irradiation methods; (ii) development of new copolymers (HEMA grafted on LPDE thin films) suitable for bioapplications. Here, sample preparation conditions were correlated to the grafted material structure and the hydration level achieved by the final product, and preliminary toxicity studies were conducted. Biocompatibility tests (haemolysis and tromboresistance) are being carried out. Work on these systems is resumed in a M.Sc. thesis completed in 2006 and a Ph.D. thesis to be defended in 2007.

The Group is also 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.

Installation and alignment of the various components and equipments of EPA was resumed in 2006. Detailed test work of the facility has shown that an enhanced signal to noise ratio requires further improvement of the shielding and the installation of a 20 cm long Be filter. The design of a liquid N_2 cryostat to cool this filter is under way. The acquisition of a new detector assembly for the two-axis neutron diffractometer DIDE with 8 linear position-sensitive ^{3}He counters to replace the old "banana" detector with improved count-rate and equivalent angular resolution, awaits a funding decision. A rotating chopper time-of-flight diffractometer, ETV, is operational. Development of the Converging Multi-channel Collimator (CMC) based on an original concept was temporarily interrupted due to the shortage of manpower especially technicians. The design of a prototype having been completed in 2004, it is intended to proceed with the construction and testing of the device during the coming year.

Collaboration with other research groups and a policy of open access for external users to facilities operated by the Group including students are placed high in the ranking of priorities. One student resumed the work for an MSc degree during 2006.

The Group's work is supported by funds from FCT, IAEA, ITN and income from services.

F. Carvalho, senior researcher and founder of the group, has reached retirement age in January 2006. Although he continues to contribute to current activities the long due implementation of a plan of selected recruitment of scientific personnel has become more pressing.

Researchers
F.G. CARVALHO, Senior Researcher [1]
F.M.A. MARGAÇA, Princ. Researcher
A.N. FALCÃO, Princ. Researcher
L.M.M. FERREIRA, Aux. Researcher
C.M.M. CRUZ, Aux. Researcher (20%)
J.S. NEVES, Aux. Researcher (20%)

Students
M. CARRAPIÇO, B.I. Grantee, POCTI [2]
SUSANA GOMES, B.I. Grantee, POCTI

Technical Personnel
D. SILVA, Laboratory Assistant

Collaborators
I.M.M. SALVADO, Dep. of Glass and Ceramics Engineering, UIMC, University of Aveiro
M.H. GIL, Dep. of Chemical Engineering, Faculty of Sciences and Technology, Coimbra University

[1] retired
[2] left in March

Thermal analysis of PDMS-TEOS-PrZr Hybrid Materials Prepared by γ Irradiation

F.M.A. Margaça, S.R. Gomes, A.N. Falcão, L.M. Ferreira and I.M.M. Salvado[1]

Objectives

The purpose is to investigate the thermal behaviour of hybrid materials prepared by gamma irradiation of the mixture of the precursors PDMS, TEOS and PrZr.

Results

Hybrid materials have been prepared by γ-irradiation of a mixture of polydimethilsiloxane, PDMS, and the alko-xides, ALC, tetraethylortosilicate, TEOS, and zirconium propoxide, PrZr. This is a new method [1] of prepa-ration of hybrids which are currently obtained via the sol-gel process. The sol-gel hybrids have been widely used in different fields, ranging from medical uses to electronics. The applications are a consequence of the final properties of the materials which are known to depend strongly on the processing conditions. The thermal stability of the prepared materials has been investigated by Differential Scanning Calorimetry and Thermal Gravimetry Analysis.

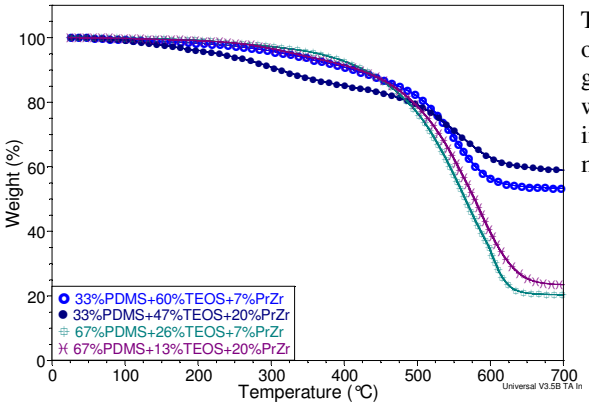

Fig.1: Thermograms of hybrid samples.

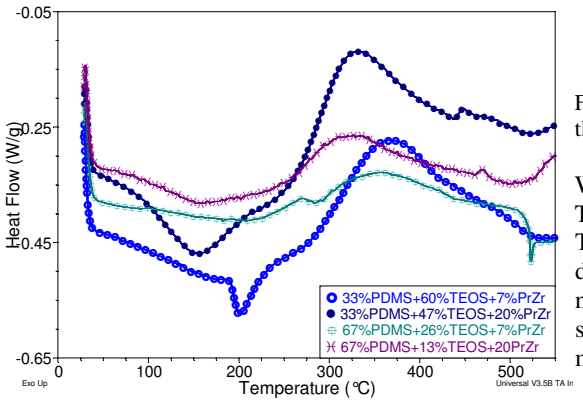

Fig.2: DSC plots of hybrid samples.

DSC and TGA curves show that the thermal behaviour depends on the relative organic/inorganic content. In fact, two distinct behaviours are found depending on whether PDMS/ALK > 1 or < 1, with ALK = alkoxide.

The endothermic peak in the DSC curves is also observed in purely inorganic gels prepared from TEOS and PrZr by sol-gel, as shown in Fig. 3.

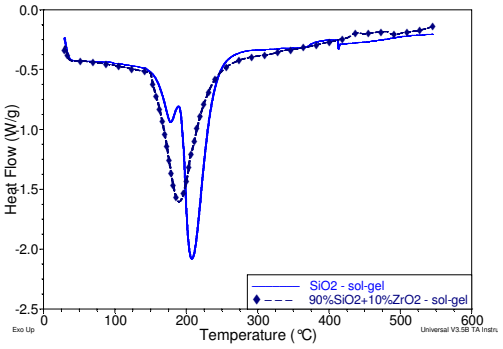

Fig. 3: DSC plot for SiO_2 and 90%SiO_2+10%ZrO_2 materials prepared by sol-gel, without any polymer.

This provides evidence of the presence of inorganic oxide regions in the hybrid materials prepared by gamma irradiation of the precursors. Furthermore it was found that the temperature of the exothermic peak in DSC curves depends on the ratio of the number of molecules TEOS/PrZr.

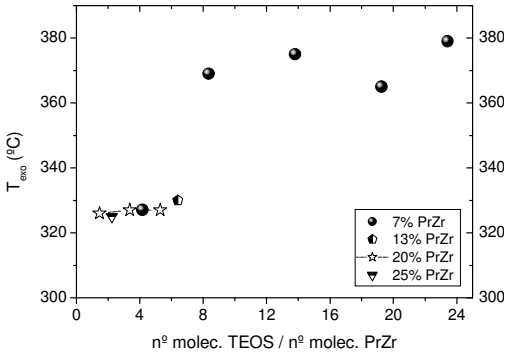

Fig. 4: Variation of the DSC exothermic peak temperature with the ratio no. molecules TEOS/PrZr.

When the number of molecules TEOS/PrZr < 8, T_{exo} ~ 325 ºC. For a higher ratio value T_{exo} > 360ºC. This indicates that the inorganic oxide regions can develop into two structures, depending on the relative number of molecules TEOS/PrZr. A more stable structure develops whenever the ratio of the number of molecules TEOS/PrZr > 8.

Published work

S.R. Gomes, F.M.A. Margaça, I.M. Miranda Salvado, L.M. Ferreira, A.N. Falcão, *Nucl. Instr. & Meth. B* 248 (2006) 291-296.

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Elemental and Topographic Characterization of LDPE Based Copolymeric Films Obtained by Gamma Irradiation

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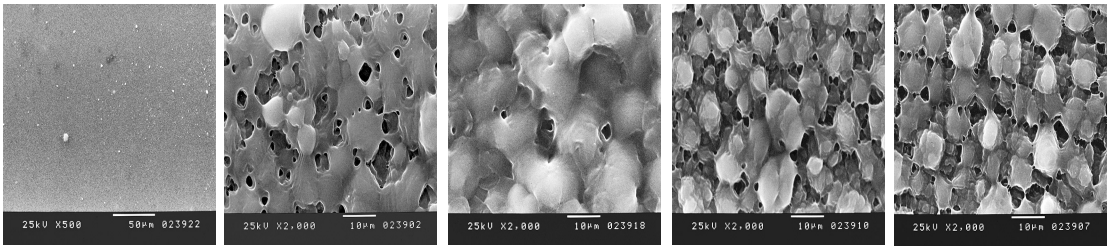
The selection of materials for bioapplications requires proper considerations of mechanical, physical and biological properties, and very rarely a single natural or synthetic material presents all the adequate properties. In our studies, using 2-hydroxyethyl methacrylate (HEMA) as grafting monomer, we have optimized the preparation of new grafted copolymeric Low Density Polyethylene (LDPE) based films by means of ^{60}Co gamma irradiation. The films so developed show, at a macroscopic scale, a homogeneous graft distribution (independent of the grafting degree) reaching hydration levels up to 95%. Their characterization was done using Nuclear Microprobe (NM), Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM). These techniques proved to be very important for the elemental and topographic characterization of the film. Elemental analysis performed by NM using PIXE (Proton Induced X-ray Emission), allowed evaluation of the toxicological risk in the possible uses of the new films. Trace contamination of elements heavier than Si (Ca, Cl, Fe, K, P, S; Si and Zn) were found, but their concentrations do not pose toxicological risk. AFM and SEM techniques revealed the topography and 3D porous structure of the polyHEMA grafted surface that grows over the LDPE matrix film. The observed porous distribution configuration and their dimensions (Ø = [0.405µm - 5.517µm] assure oxygen permeability. Morphological analysis was important to inform about polyHEMA grafted chain scission processes, which seems to occur with increasing radiation dose above a certain threshold. Further work involves biocompatibility tests (haemolysis and tromboresistance studies) of the prepared films as well as the application of the developed technique to pre-configured polyethylene based supports.

Figure: SEM micrographs of LDPE film (500×) and PE-g-HEMA copolymeric films (2000×).



LDPE film matrix (non irradiated)	PE-g-HEMA film (25h; 237%)	PE-g-HEMA film (30h; 403%)	PE-g-HEMA film (35h; 166%)	PE-g-HEMA film (40h; 163%)

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Neutron Spectrometers at the Portuguese Research Reactor

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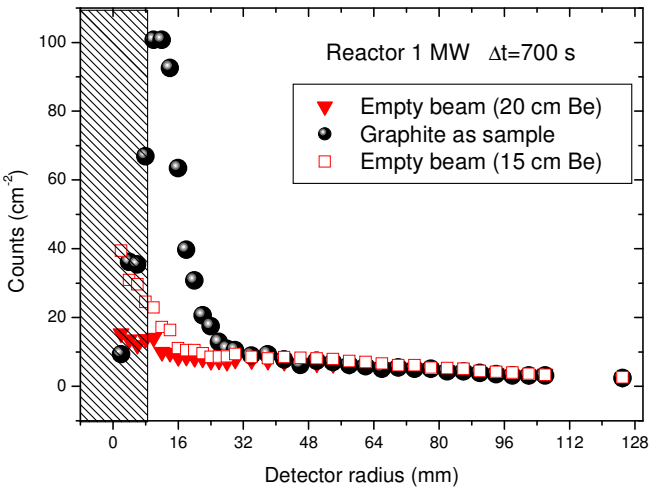

Installation of the out-of-pile components of the EPA instrument was resumed. A software program for data handling was developed. Test work progressed as concerns the measurement of signal-to-noise ratios under different shielding configurations and instrument operating conditions. The need to use a cooled beryllium filter to reduce the noise was confirmed. The influence of the Be filter in the background associated with the beam is shown in the figure for two different filter lengths. The difference is observed in the region nearer to the beam stop (\\\\) that corresponds to the lower Q region of the measurement. The cooling of the filter will improve the useful intensity by a factor of 3, keeping the background unaltered. Further improvement on the detector shielding is required.