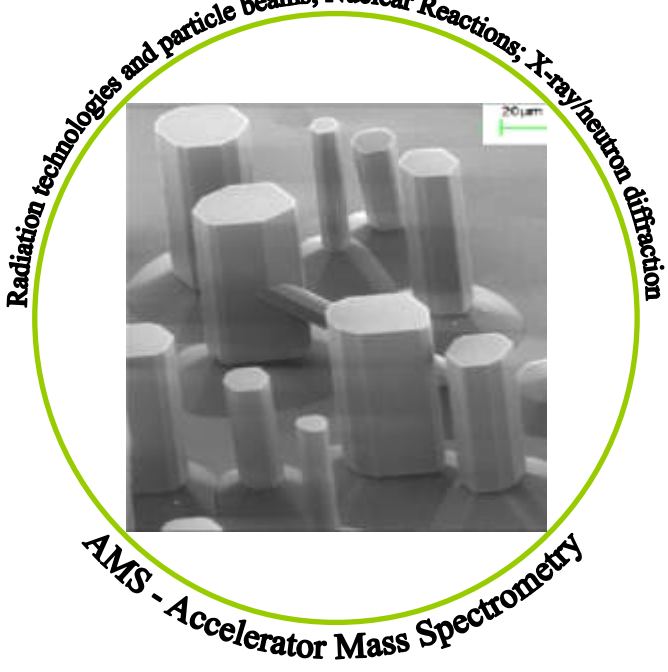
Physics and Accelerators Unit



Physics and Accelerators Unit

E. Alves, F. Margaça, J. Manteigas, L. Botelho

In 2011 the research activities were guaranteed by the research groups installed in the Unit and focused on the current projects using the new possibilities offered by the upgraded laboratories and new infrastructures installed under the re-equipment programme.

The post graduated formation was maintained as an important commitment of the Unit and new graduated students were engaged in the research activities during 2011.

The R&D activity in the Unit was carried out under the responsibility of the following laboratories and groups:

The **Ion Beam Laboratory (IBL),** equipped with a 2.5 MV Van de Graaff accelerator and an ion microprobe end-station, a 3 MV tandem accelerator with a micro-AMS system, and a 210 kV high fluence ion implanter. The laboratory is an open-access facility and the experimental studies cover the fields of Materials Science, Environment, Health, Biomedicine, Atomic and Nuclear Physics (cross-sections measurements). The research topics will appear in the next pages under the headings **Advanced Materials Research Group**, **Materials Characterization with Nuclear Techniques**, **Elemental Characterization and Speciation Group**, **Group of Biomedical Studies and Nuclear Reactions Group**.

The **Condensed Matter Group** investigates hybrid materials and polymers, prepared and/or modified by gamma irradiation, using conventional and neutron scattering techniques. The group maintains and operates the Laboratory of Macromolecular Materials, LM3, that is being upgraded with new facilities for sample preparation and characterization.

The **Radiation Technologies: Processes and Products Group** deepens the applications of ionising radiation using the radiation equipment – Precisa 22, experimental Co-60 facility and a LINAC – linear accelerator (IRIs), and the laboratory of clean rooms (LETAL). These new infrastructures, built under Re-equipment Program, are unique in the country and support the R&D Projects in the fields of Health, Environment, Food and Art.

The **Nuclear Instruments and Methods Group** investigates modelling on neutron and gas discharge physics and development and production of nuclear instrumentation.

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Advanced Materials Research

Eduardo Alves

The Advanced Materials Research Group (GIMA) operates most of the experimental facilities at the Ion Beam Laboratory (IBL). The IBL is equipped with a 2.5 MV Van de Graaff accelerator with a nuclear microprobe and external beam facility; a 3 MV tandem accelerator with a 30 μm lateral resolution Accelerator Mass Spectrometry (AMS) system; a high flux Danfysik S1090 ion implanter.

The group explores and develops ion beam techniques to study advanced materials with high technological impact, e.g. wide band gap semi-conductor nanostructures, oxides and functional materials in collaboration with a long list of other groups. Among the wide band gap materials our major interests are focused on III-nitrides and ZnO. These alloys are the base of an emerging class of optoelectronic devices operating in the visible wavelength range of the electromagnetic spectrum being under intense research worldwide. Our work aims at the optimization of the implantation conditions of magnetic and optically active dopants in these materials. In addition an intense research on the structural properties and Rare Earth doping of GaN/AlN QD layers continued in collaboration with Universities of Aveiro, Grenoble and Strathclyde.

The work in oxides aims at modification of the optical and structural properties of α-Al_2O_3 as well as the study of magnetic doping of ZnO by ion implantation. The potential of these materials for spintronics applications is being investigated with University of Aveiro and the University of Lisboa.

Taking advantage of the versatility of ion beam techniques to study thin films and multilayers, important work continued on the characterisation of magnetic thin films for magnetic spin valves, tunnel junctions, and functional oxynitride coatings, in collaboration with INESC, University of Minho and New University of Lisbon.

The activities under the technology programme of the European Fusion Development Agreement (EFDA), in association with Instituto de Plasmas e Fusão Nuclear (IPFN) was focused on the study of beryllium intermetallics and the study of surface erosion and redeposition processes as well as ^2H retention in JET tiles.

Training and Education continued as a major commitment of the group through the supervision of M.Sc. and Ph.D. thesis.

All the referred activities are funded by projects, either European or National (FCT), in collaboration with other Institutions. Of particular importance are the projects funded by the EC, *"FEMaS-Fusion Energy Material Science"*, EURATOM 7th Framework Programme for Nuclear Research and Training, Grant agreement No 224752-CA, (2008-2011) and *"Support of Public and Industrial Research Using Ion Beam Technology (SPIRIT)"*, Grant agreement No 227012-CP-CSA-Intra (starting date 2009/03/01) and EFDA JET Technology Workprogramme JW11-FT-3.59. The external collaborations allowed a continuous exchange of expertise and mobility of researchers, a key condition to keep the scientific activity of the group at the forefront of research and its international recognition in the field of processing and characterization of advanced materials with ion beams.

Publications (peer reviewed journals): *45*
Proceedings Books*: 12*
Conference and workshop contributions: *3 invited, 19 oral and 27 posters.*
Running projects*: 18*

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Connecting the external proton beam at ITN with silver art objects

V. Corregidor, L.C. Alves, L. Penalva[1], B. Maduro[1], A. J. Candeias[2], J. Mirão[3]

Objectives

The starting point of this work was the detection of kanji characters designed inside an oratory-reliquary, which belongs to the collection of the Museu Nacional de Arte Antiga (MNAA), Lisbon. This object, from the "Vidigueira Treasure", was unquestionably assumed as the result of Indo-Portuguese work. Two other objects, with common origin, a pax "porta-paz" and a missal-bookshelf, are part of this "Vidigueira Treasure".

One of the aims of the work is to perform an elemental compositional analysis of the pieces, using the non-destructive and sensitive capabilities of the micro-external beam setup installed at ITN, and then correlate the data with other coeval silver objects for establishing the provenance of the silver used to produce them.

Results

The first object to be analysed at ITN, was the pax. It was executed in silver partially golden and decorated with silver filigree. The results obtained by PIXE at several points show that an Ag:Cu alloy with Cu contents lower than 10 wt.% was used in the manufacture of the object. The higher Cu concentration (10 wt.%) was found in the interior filigree of the pediment. In all cases, the impurities detected were: Bi, Au, Pb, Hg, Zn, Fe and Ti. The presence and concentration of these impurities give information about the provenance, and so, similar values are expected to be found in the other objects from the Vidigueira Treasure, since the same origin is assumed. The results obtained will contribute to the establishment of a reference composition of the Indo-Portuguese Silver Jewellery. In the golden parts, some of them very deteriorated, only Au, Ag and Cu could be detected, confirming that the mercury gilding technique was not employed in this piece. The analysis and combined information of the spectra simultaneously recorded using both IBA techniques, PIXE and RBS, allowed the determination of Au depth profile in the golden parts. Due to the state of conservation, the distribution of gold on the surface is non-homogeneous and a pure layer of Au could not be detected. Instead, a maximum concentration of Au at the surface, around 85 wt.%, was found in the back side. Gold diffusion lengths in the golden parts were always below 3 µm. The handle of the piece has a snake shape, performed in two parts joined by a gilded frieze. The PIXE elemental maps show the Au, Pb and Hg distribution along the joint (Fig. 1). The Pb and Hg may have been intentionally added during the welding process for reducing the melting point of the Ag:Cu alloy. The top of the main body of the oratory has a silver decorative element, with three small urns. Two of them were also analysed at ITN. Results show not only different Ag:Cu composition ratios for each one, 98:2 wt.% and 90:10 wt.%, but also the presence of different impurities such as Zn and Ni in the latter. Prior to this characterization the pieces were subjected to an exhaustive cleaning process of conservation and restoration and the patinas that were formed over the years have been removed.

Conclusion

It should be noticed that by using the IBA techniques it is also possible studying patinas formed on the surface of silver objects, mostly Ag_2S and AgCl. Although it is not possible to extract direct information on the chemical species formed, the possibility of obtaining 2D distribution of elements such as Cl and S allows inferring the formation of such compounds and define areas with different corrosion extent.

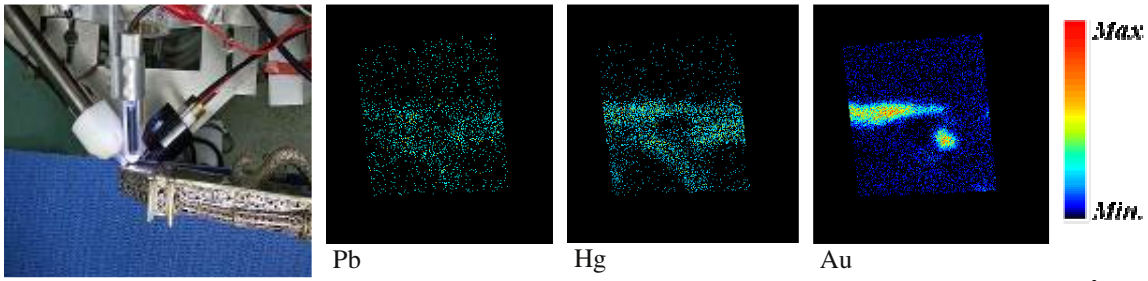

Fig. 1- Pax from the "Vidigueira Treasure" during the measurements at ITN. PIXE elemental maps of Pb, Hg and Au, 800x800 µm^2, from the joint of the handle with snake-shape.

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Defects identification on modern gold coins

V. Corregidor, L.C. Alves, J. Cruz

Commemorative coins, usually made of noble materials such as gold, minted by the Mint Offices of different countries, represent value added goods both for the Mint Officers and for the collectors.
In this sense, manufacture free of defects and good preservation conditions are of utmost importance. But sometimes, even under good preservation conditions coloured spots, often related with corrosion, appear on the surface of the coins. One of these coins, minted in 2006, revealing different types of defects in both faces, was analysed by PIXE at ITN. It was found that most of these defects are related with the presence of silver at the surface (Fig.1). The origin of silver can be related with contamination of the production line, where some silver particles may transfer to the surface of the gold strips. Afterwards, the different processes such as polishing procedures or contact with sulphur-containing contaminants, promote tarninishing of silver tarnish creating silver stains of different colours.

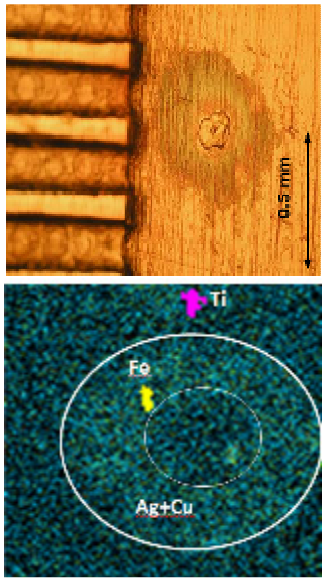

Fig.1 Stereomicroscopy photograph and PIXE compositional scan distribution of a Ag-rich defect with Fe and Ti.

Impurities evolution and patina formation on ancient Portuguese silver coins

V. Corregidor, L.C. Alves, N. Franco, J. Cruz

Ancient silver coins are usually a Ag:Cu alloy. The Cu was intentionally added in order to increase the hardness of the alloy. But other elements can be found in the composition resulting from the raw materials used to mint the coins. The presence of certain elements and its concentration can give information on the origin of the raw material and also on the period in which the coins were minted.
Portuguese silver coins from different centuries (XIX and XX) and provenance were analysed by PIXE. As a general tendency, the presence of impurities and their concentrations have decreased along time (Fig. 1): Ni, Au, Hg or Bi were not detected in the 1 rupia coins from 1912, but they are present in the coin of *6 vinténs* of D. João VI (1816-1826). This tendency is not followed by the Pb concentration when we compare the same *6 vinténs* of D. João VI (1816-1826) and *½ rupia* from 1881.

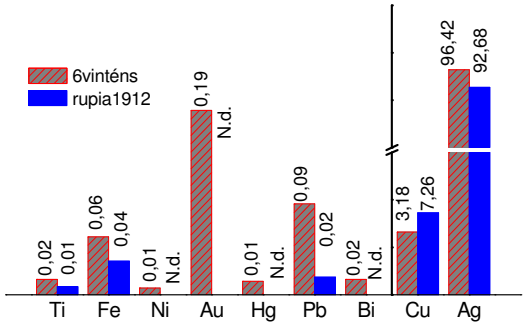

Fig. 1- Comparison of impurities concentration (at.%) found in two coins: 6 vinténs of D. João VI (1816-1826) and 1 rupia coin from 1912.

Characterization of historical tiles

S. Coentro,[1,2] S. Muralha[1,2], M. Larsson[1], M. Vilarigues[1,2], V. Corregidor, L.C. Alves, R.C. da Silva

Historical tiles belonging to Convento de Sta Clara-a-Velha, Coimbra and to Palácio da Pena, Sintra, dated from the late Middle Ages to early Renaissance period but of uncertain provenance, have been characterized by nuclear microprobe analysis, as part of a collaboration project on early Portuguese tile production. Element identification allowed establishing the colour pigments used in the decoration of the tiles and the compositions of the glazes.

[1] DCR/FCT-UNL; [2] VICARTE/FCT-UNL; [3] REQUINTE/FCT-UNL.

Determination of crystal ordering and lattice-site location in ZnO compounds: sublattice-resolved studies via ion channelling

A. Redondo-Cubero, R. Gago[1], A. Hierro[2], M. Vinnichenko[3], J.-M. Chauveau[4], G. Tabares[2], M. Krause[3], A. Kolitch[3], E. Muñoz[2], N. Franco, E. Alves, K. Lorenz

ZnO and MgZnO samples were studied by Rutherford Backscattering Spectrometry under channelling conditions (RBS/C). In ZnO samples, grown in c-plane orientation by pulsed magnetron sputtering, the defect formation was evaluated by RBS/C as a function of the substrate temperature. Interestingly, Zn-related defects are annealed at relatively low temperatures, while a high amount of O interstitials remain even for substrate temperatures up to 550 ºC. These results point to a higher relevance of O-related defects for electronic properties and structure formation of undoped epitaxial ZnO films. In MgZnO samples, grown in a-plane orientation by molecular beam epitaxy, the lattice-site location of Mg was evaluated. Using the asymmetric shadowing effect for both Zn and O sites in RBS/C angular scans, the substitutional behaviour of Mg in Zn-sites was confirmed unambiguously for Mg contents as high as 56 %.

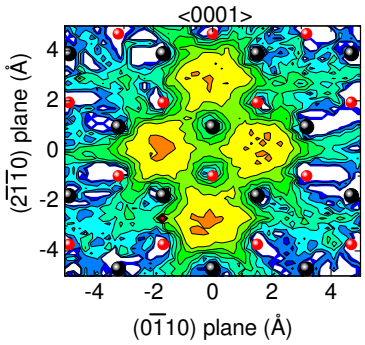

Fig. 1 Flux distribution of channelled particles in the c-axis of a wurtzite crystal (Redondo-Cubero et al., J. Appl. Phys. 110, 113516, 2011).

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Cathodoluminescence of rare earth implanted Ga_2O_3 and GeO_2 nanostructures

E. Nogales[1], P. Hidalgo[1], K. Lorenz, B. Méndez[1], J. Piqueras[1], E. Alves

Rare earth (RE) doped gallium oxide and germanium oxide micro- and nanostructures, mostly nanowires, have been obtained and their morphological and optical properties characterized. Undoped oxide micro- and nanostructures were grown by a thermal evaporation method and were subsequently doped with gadolinium or europium ions by ion implantation. No significant changes in the morphologies of the nanostructures were observed after ion implantation and thermal annealing. The luminescence emission properties have been studied with cathodoluminescence (CL) in a scanning electron microscope (SEM). Both β-Ga_2O_3 and GeO_2 structures implanted with Eu show the characteristic red luminescence peak centred at around 610 nm. Sharpening of the luminescence peaks after thermal annealing is observed in Eu implanted β-Ga_2O_3, which is assigned to the lattice recovery. Gadolinium asimplanted samples do not show rare earth related luminescence. After annealing, optical activation of Gd^{3+} is obtained in both matrices and a sharp ultraviolet peak, centred at around 315 nm, is observed.

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Low temperature implantation damage build-up in GaN with different surface orientations

K. Lorenz, A. Redondo-Cubero, N. Catarino, E. Alves, E. Wendler[1]

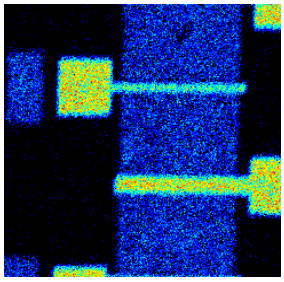
Detailed implantation studies have been performed in GaN epitaxial films grown along different crystal directions (c-plane, a-plane and m-plane material) using *in situ* Rutherford Backscattering Spectrometry /channelling at 15 K. Ar implantation at 15 K allows the study of damage build-up, minimizing thermal as well as chemical effects. Several stages could be distinguished in the damage formation processes. For low ion fluences an almost linear increase of damage with the ion fluence is observed attributed to the formation of point defects in isolated collision cascades (stage I). When the damaged regions produced by single ions start to overlap, vacancies and interstitials can recombine resulting in a plateau-like slow increase of the damage level (stage II). For higher fluences more stable defect complexes form (stage III) and the damage level increases steeply before reaching a second plateau (stage IV). For high fluences (stage V) GaN amorphizes. Up to stage III the damage curve is similar for all three crystal orientations. The saturation in stage IV however, takes place at a much lower level in a-plane material compared to c- and m-plane GaN. Enhanced dynamic annealing effects may be the reason for this pronounced difference or a certain type of defects that are shadowed by the atomic rows of the host material along certain directions.

[1] Institut für Festkörperphysik, Friedrich-Schiller-Universität Jena, Jena, Germany.

Study of ohmic contacts for GaN-based devices

A. Redondo-Cubero, L.C. Alves, M.F. Romero[1,2], M. Peroni[3], C. Lanzieri[3], A. Cetronio[3], R. Gago[4], M.D. Ynsa[5], E. Alves, K. Lorenz, E. Muñoz[1]

The in-depth diffusion and the lateral homogeneity of Au/Ni/Al/Ti ohmic contacts for AlGaN/GaN high electron mobility transistors were analyzed by Rutherford Backscattering Spectrometry (RBS) and Particle Induced X-ray Emission (PIXE). Changing the thickness of the Al barrier layer, different surface morphologies were produced after the rapid thermal annealing. However, using the microprobe beam line at ITN, such morphologies could not be correlated with compositional variations within the resolution limits. Remarkably, RBS does show very large differences in the depth profiles of the metals with the increasing Al thickness. Such behaviour seems to be also linked to the production of ohmic contacts with low resistance.



PIXE map of transmission line models using the signal from the Au(M) line.

[1] ISOM and DIE, ETSI Telecomunicación, Universidad Politécnica de Madrid, Madrid, Spain.
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[3] Selex Sistemi Integrati S.p.A. Via Tiburtina, Roma, Italy.
[4] Instituto de Ciencia de Materiales de Madrid, CSIC, Madrid, Spain.
[5] Centre of Micro-Analysis of Materials, Universidad Autónoma de Madrid, Madrid, Spain.

Composition, structure and morphology of $Al_{1-x}In_xN$ thin films grown on $Al_{1-y}Ga_yN$ templates with different GaN contents

S. Magalhães, K. Lorenz, N. Franco, E. Alves, S. Pereira[1], L. T. Tan[2], R.W. Martin[2], K.P. O'Donnell[2], I. M. Watson[3]

In this work we have studied the composition, structure and morphology of four $Al_{1-x}In_xN$ thin films grown simultaneously on $Al_{1-y}Ga_yN$ (y = 1, 0.93, 0.86, and 0.69) buffer layers by Metal Organic Chemical Vapour Deposition (MOCVD). A nominal InN content of ~16% was chosen to achieve closely lattice matched $Al_{1-x}In_xN$ for the buffer layers with intermediate GaN molar fraction, a small tensile strain for growth on GaN, and compressive strain for the buffer layer with the lowest GaN fraction. Only the film deposited on GaN shows true pseudomorphic growth to the template. For growth on the ternary templates, the film roughness and the pit density increases with decreasing GaN content of the $Al_{1-y}Ga_yN$ template due to the roughening of the growth templates themselves. While the macroscopic crystal quality of these ternary templates is quite homogeneous, the quality of the films varies significantly across the wafers. Results indicate that the structural and morphological quality of the templates does not only influence the structure and morphology of the films but also influence the strain state, the homogeneity and phase purity of the $Al_{1-x}In_xN$ films.

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[3] Institute of Photonics, SUPA, University of Strathclyde, Glasgow G4 0NW, U.K.

Study of CdTe nanorods grown on glass substrates

V. Corregidor, E. Alves, L.C. Alves, N. Sochinskii[1]

Photovoltaic (PV) companies are actively involved in commercializing thin-film PV technologies using CdTe. In order to obtain higher efficiencies values, a new concept is under development. It is based in the use of nano-structured CdTe as the absorber layer. Nanorods of CdTe have already been successfully grown on sapphire substrates, but it is a critical issue the replication on commercial soda-lime glass. For this study, a monolayer of polyvinyl alcohol was deposited on the substrate prior to the deposition of bismuth, which acts as a catalyst material. The CdTe nanorods are grown by pulsed laser deposition using as a target a CdTe ingot previously grown by the Bridgman method. The samples were characterized by Rutherford Backscattering Spectrometry. The results indicate that the CdTe nanorods formation is very dependant on the processing parameters, specially the growth temperature. The composition of the CdTe nanorods is not stoichiometric: they all were found to be Te-rich, with concentration values up to 70 at%. Others catalyst materials such as NiCr or ZAO are under study.

[1] Consorzio C.R.E.O., Via Pile 60, I-67100 L'Aquila, Italy.

Effect of electron irradiation on the structural properties and photoluminescence of bulk HVPE GaN

M.-Y. Xie[1], A. Santos, E. Alves, T. Monteiro[2], N. T. Son[1], J. Rodrigues[2], A. Tolstoguzov[3], A. Usui[4], C. Hemmingson[1], B. Monemar[1], V. Darakchieva

Point defects have a strong effect on optical and electrical properties of materials, and their behaviour during device fabrication. We studied the effect of 2 MeV electron irradiation on the photoluminescence (PL) and structural properties of bulk GaN fabricated by HVPE.

Low-temperature PL reveals strong excitonic emission with well pronounced two-electron transitions and absence of DAP emission for the low electron concentration non-irradiated samples. Pronounced yellow luminescence (YL) is observed from the N-face GaN, while the Ga-face exhibits green luminescence (GL). PL measurements suggest that the GL is due to $V_{Ga}O^{-}$ while the YL is related to $V_{Ga}O^{2-}$. The electron irradiation results in an overall decrease of PL intensity indicating generation of non-radiative defects. In additiona UV band develops at ~ 3 eV upon electron irradiation. The irradiation leads to expansion of the lattice in both *c* and *a* directions, which could be associated with point defects in the Ga sublattice. Elemental analysis by SIMS and PL revealed inhomogeneous distribution of O along the GaN thickness, much higher in the N- side than in the Ga counterpart: different concentrations of the complexes formed by the irradiation induced point defects with O impurities may explain the changes observed in the lattice parameters of the N- and Ga-faces of bulk GaN.

[1] IFM, Linköping University, Sweden.
[2] Departamento de Física and i3N, Universidade de Aveiro, Portugal.
[3] FCT, New University of Lisbon, Portugal.
[4] R&D Division, Furukawa Co. Ltd., Japan..

Origin of n-type conductivity in InN grown by MOCVD

V. Darakchieva[1], M.Y. Xie[1], D. Rogalla[2], H.-W. Becker[2], K. Lorenz, E. Alves, N.P. Barradas, S. Ruffenach[3], O. Briot[3]

The free electron concentrations, structural properties, and hydrogen depth profiles in as-grown and thermally annealed MOVPE InN films were studied. The quality of the MOVPE InN films - – the dislocation and point defect densities, and electrical parameters – was found comparable to state-of-the-art MBE InN. Thermal annealing of the films resulted in lower free electron concentrations and increased electron mobilities. Enhanced H-concentrations were measured in the near-surface regions of the MOVPE InN films, similarly to MBE InN. Annealing lead to reduction in bulk H-concentration(Fig. 1) in correlation with decreasing the free-electron concentration, but does not lead to any change in dislocation densities or strain in the films. The results suggest that hydrogen is a major source for unintentional n-type doping in MOVPE InN films.

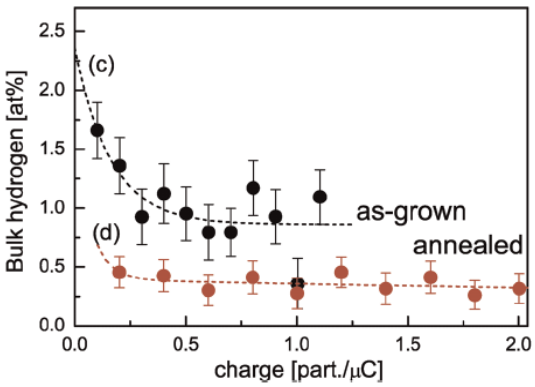


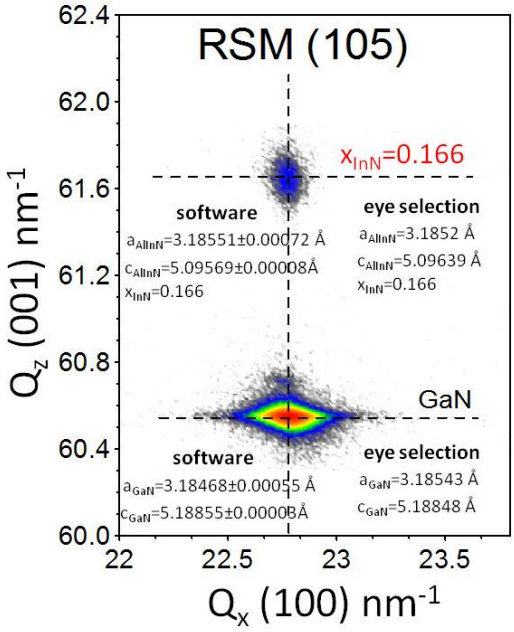
Fig. 1: Bulk hydrogen concentration versus the fluence for the as-grown (c) and annealed (d) MOVPE InN films.

[1] Department of Physics, Chemistry, and Biology, Linkoping University, 581 83 Linkoping, Sweden.
[2] Ruhr-Universitat Bochum, 44780 Bochum, Germany.
[3] Groupe d'Etude des Semiconducteurs, Université Montpellier II, 34095 Montpellier, France.

High accuracy lattice parameter derivation from XRD reciprocal space maps

S. Magalhães, K. Lorenz, N. Franco, E. Alves

Lattice parameters of a crystal are fundamental physical quantities of any material and an accurate measurement of their absolute values is crucial. X-ray diffraction (XRD) reciprocal space maps (RSM) are often used to determine lattice parameters of thin heteroepitaxial films and allow the investigation of strains. The figure shows a RSM of a 100 nm thick $Al_{0.834}In_{0.166}N$ thin film grown pseudomorphically over a GaN buffer layer on a (0001) sapphire substrate. To analyse such a RSM, the lattice parameters of the GaN buffer were measured absolutely using the Bond method and the lattice parameters of the film were then determined relative to the GaN peak. For an automated analysis of such RSM, software was developed which extracts a large number of horizontal and vertical cuts through the RSM, performs peak fitting on these cuts and determines the maximum of each peak in the RSM. The main contribution to the uncertainties in the lattice parameters and compositions was found to be the error in the peak centres. The uncertainties are then estimated by adding in quadrature the uncertainities in the lattice parameters of the GaN buffer with the uncertainty the position of the $Al_{1-x}In_xN$ peak in the RSM. The resulting uncertainty in the InN molar fraction is below 0.1% absolute. The new program allows a more accurate determination of the lattice parameters and estimation of uncertainties.



Crystal quality enhancement of ternary AlGaN alloys with high Al content by PA-MBE growth at low temperature

V. Fellmann[1], P. Jaffrennou[1], D. Sam-Giao[1], B. Gayral[1], E. Bellet-Amalric[1], B. Daudin[1], K. Lorenz, E. Alves

We have studied the influence of III/N flow ratio and growth temperature on the structural and optical properties of high Al-content (50–60%) AlGaN layers grown by plasma-assisted molecular beam epitaxy. From Rutherford Backscattering Spectrometry we established that a flow ratio slightly above 1 produces layers with low amount of structural defects. Under this optimal III/N flow ratio, we found that optimal temperatures for growth of $Al_{0.5}Ga_{0.5}N$ layers with compositional homogeneity correlated with narrow UV photoluminescence are in the low temperature range for growing GaN layers, i.e. 650–680 ºC. We propose that lowering the Ga adatom diffusion on the surface favours random incorporation of both Ga and Al adatoms in wurtzite crystallographic sites leading to the formation of a homogeneous alloy.

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Ion implantation of Cd in AlN

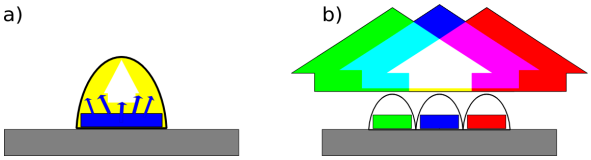
S.M.C. Miranda, N. Franco, E. Alves, K. Lorenz

AlN based semiconductor performance is hampered by the lack of an efficient p-type dopant. In order to shed more light on this topic AlN thin films were implanted with cadmium (a possible alternative to Mg for p-type doping), to fluences of 1×10^{13} at/cm^2 and 8×10^{14} at/cm^2. The implanted samples were annealed at 950 ºC under flowing nitrogen. The implantation damage could be fully removed for the lower fluence, while for the higher fluence the crystal quality was only partially recovered. For the high fluence sample the lattice site location of the ions was studied by Rutherford Backscattering/channelling (RBS/C): Cd ions were found to be incorporated in substitutional Al sites and no diffusion is seen upon thermal annealing. The observed high solubility limit and site stability are prerequisite for using Cd as p-type dopant in AlN.

Solid State Lighting Redefined by Bandgap Engineering Techniques

K. Lorenz, M. Auf der Maur[1], A. Di Carlo[1], K.P. O'Donnell[2]

The design strategy presently employed to obtain 'white' light from semiconductors combines the emission of an InGaN blue or UV light-emitting diode (LED) with that of one or more yellow-orange phosphors. While commercially successful, this approach suffers from energy losses during the absorption and reemission processes; compared to the alternative of combining 'true' red, green and blue (RGB) sources, it is intrinsically inefficient.



Schematics of the two main approaches to achieve white light using LEDs: a) A blue or UV LED is combined with a yellow (or multi-colour) emitting phosphor. b) Combination of three LEDs emitting in the green, blue and red.

The two major roadblocks to the RGB approach are 1) the green gap in the internal quantum efficiency (IQE) of LEDs and 2) the diode droop in the efficiency of LEDs at higher current densities. The physical origin of these effects, in the case of III-nitrides, is generally thought to be a combination of Quantum Confined Stark Effect (QCSE) and Auger Effect (AE). These effects respectively reduce the electron–hole wavefunction overlap of In-rich InGaN quantum wells (QW), and provide a non-radiative shunt for electron–hole recombination, particularly at higher excitation densities. We developed a novel band gap engineering strategy based upon graded QWs that offers solutions to both of the roadblocks mentioned above. Its potential is tested by the results of simulations of green InGaN diodes performed using TiberCAD device modelling suite, which calculates the macroscopic properties of real-world optoelectronic and electronic devices in a multiscale formalism.

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Experimental determination of the bandgap of epitaxial rhombohedral BN

N. Ben Sedrine, M.-Y. Xie,[1] N. Franco, M. Chubarov,[1] A. Henry[1], V. Darakchieva[1]

Boron nitride (BN) has unique physical and chemical properties, such as strong planar covalent bonds but weak interactions between its basal planes, high thermal and chemical stabilities. In order to develop BN as a UV optoelectronic material, it is important to reliably determine the optical band-gap energy, *E*g. All BN forms have a wide bandgap; however, despite extensive experimental and theoretical studies for cubic BN, wurtzite BN and hexagonal (h)-BN, little is known about rhomboedral (r)-BN. We have performed spectroscopic ellipsometry and cathodoluminescence spectroscopy of r-BN films grown by chemical vapour deposition on sapphire substrate. The ellipsometric data modelling of the 400nm-thick BN layer using Cauchy model, reveals the uniaxial nature of the r-BN film with ordinary and extraordinary dielectric responses for direction of light polarization parallel and perpendicular to the sample surface, respectively. We have determined the band-gap energies of r-BN Eg_{ord} = 5.314 eV (233 nm) and $Eg_{extraord}$ = 5.671 eV (218 nm) from the calculated absorption coefficient α for the respective polarizations. Our results indicate that the band gap of r-BN is very close to the values reported previously for h-BN. Additional structural analysis further indicates that previous works might have erroneously assigned the band gap of r-BN to h-BN and calls for new experimental and theoretical works.

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Ageing effects on the wettability behaviourr of laser textured silicon

B. Nunes, A. P. Serro[2,3], V. Oliveira[4,5], M. F. Montemor[5], E. Alves, B. Saramago[2], R. Colaço[1]

We investigate the ageing of acid cleaned femtosecond laser textured <100> Si surfaces. Changes in the surface structure and chemistry were analysed by Rutherford Backscattering Spectrometry (RBS) and X-ray photoelectron spectroscopy (XPS), in order to explain the time evolution of the water contact angles with the laser textured surfaces. It is shown that highly hydrophobic Si surfaces are obtained immediately after laser texturing and cleaning with acid solutions (water contact angle > 120º). However these surfaces are not stable and ageing leads to a decrease of the water contact angle which reaches a value of 80º. XPS analysis of the surfaces shows that the growth of the native oxide layer is most probably responsible for this behaviour.

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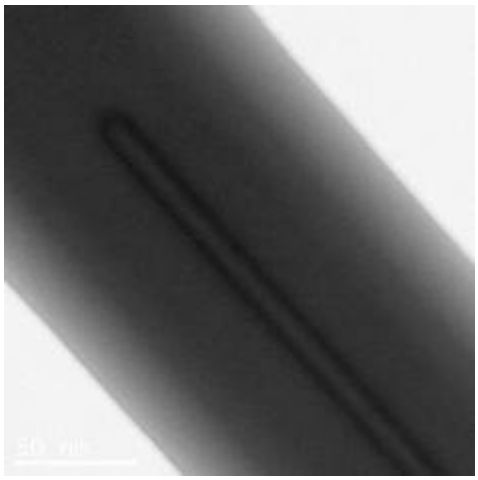
Optical properties of InAsP/InP core shell nanowires grown by molecular beam epitaxy

C. Bouhafs, N. Ben Sedrine, L. Samet[1], D. Lindgren[1], B. Monemar[1], J. C. Harmand[1], R. Chtourou[1], V. Darakchieva

We have studied InAsP/InAs core shell nanowires grown using molecular beam epitaxy (MBE) on InP (111) B substrate. The structural properties have been investigated using transmission electron microscopy (TEM) and scanning electron microscopy (SEM). The crystal structure of the InAsP core is wurtzite without defects and the crystal structure of the InP shell is mainly wurtzite with thin slices of zincblende inserted randomly on the top of the wire as seen in the figure. The diameter of the InAsP is around 20 nm (below Bohr radius of the InAsP) allowing the 2D confinement in the nanowires.
The optical properties have been investigated using micro-photoluminescence (μPL) and photoluminescence as function of temperature (from 7.8 to 180 K) and excitation power density, which confirmed the confined nature of the bound excitons.



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Enhanced dynamic annealing and optical activation of Eu implanted a-plane GaN

N. Catarino, E. Nogales[1], N. Franco, V. Darakchieva, S.M.C. Miranda, B. Méndez[1], E. Alves, J.G. Marques, K. Lorenz

The implantation damage build-up and optical activation of a- and c- GaN epitaxial films was studied upon 300 keV Eu implantation at room temperature. The implantation defects cause an expansion of the lattice normal to the surface, i.e. along the a-direction in a-GaN and along the c-direction in c-GaN. The defect profile is bimodal with a pronounced surface damage peak and a second damage peak deeper in the bulk in both cases. For both surface orientations, the bulk damage saturates for high fluences. Interestingly, the saturation level for a- GaN is nearly three times lower than that for c-plane material suggesting very efficient dynamic annealing and strong resistance to radiation. a-GaN also shows superior damage recovery during post-implant annealing compared to c-GaN. For the lowest fluence, damage in a-GaN was fully removed and strong Eu related red luminescence is observed. Although some residual damage remained after annealing for higher fluences as well as in all c-plane samples, optical activation was achieved in all samples revealing the red emission lines due to the $^5D_0 \rightarrow {}^7F_2$ transition in the Eu^{3+} ion. The present results demonstrate great promise for the use of ion beam processing for a-GaN based electronic devices as well as for the development of radiation tolerant electronics.

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Damage formation and recovery in Fe implanted 6H–SiC

P. Miranda, U. Wahl, N. Catarino, K. Lorenz, J.G. Correia, E. Alves

In order to study the damage formation and Fe lattice site location we have implanted 6H–SiC single crystals with $^{56}Fe^+$ ions with an energy of 150 keV, at fluences of 5×10^{14} Fe^+/cm^2 and 1×10^{16} Fe^+/cm^2 at different temperatures. The samples were subsequently annealed up to 1500 °C in vacuum in order to remove the implantation damage. The results show that the amorphisation is avoided by implanting at temperatures above 250 °C. For the samples implanted at lower temperatures, the amorphous layer regrows epitaxially at 1500 °C. The recrystallization induces the redistribution of the Fe ions in the implanted region with some segregation towards the surface, but the total amount of Fe is conserved. The samples implanted above the critical temperature that avoids amorphisation reveal a high fraction of Fe incorporated into regular sites along the [0001] axis. After the annealing at 1000 °C, a maximum fraction of ~75%, corresponding to a total of 3.8×10^{14} Fe^+/cm^2, was measured in regular sites along the [0001] axis.

Position-sensitive detectors for use in RBS/Channelling studies

P. Miranda, U. Wahl, J.G. Correia, E. Alves, E. Bosne[1], M.R. Silva[2], M. Campbell[3], L. Tlustos[3], X. Ll. Cudie[3]

We have investigated the possibilities for the use of 2-dimensional position-sensitive detectors (PSDs) in RBS/C studies. For that purpose two types of PSDs were tested with 2 MeV ^{4}He particles: a) a 1×1 cm^2 Si diode working with resistive charge division (RCD), b) a 256×256 pixel 1.5×1.5 cm^2 TimePix detector. Both detector types were able to process count rates of ~1 kHz, however, the energy resolution of the RCD PSD (34 keV FWHM) was somewhat superior to the TimePix (55 keV). While a position resolution of 160 μm (standard deviation) was achieved with the RCD PSD, the position resolution of TimePix is superior due to its 55 μm pixel size. However, in both cases the angular resolution of the measurement is dominated by the size of the ^{4}He beam spot, collimated to 0.5 mm. For typical experimental geometries, angular resolutions around σ = 0.12° (RCD PSD) and σ = 0.06° (TimePix) are achieved. The most promising fields of application for both types of detectors are considered to be strain measurements in thin films and lattice location studies of heavy impurities.

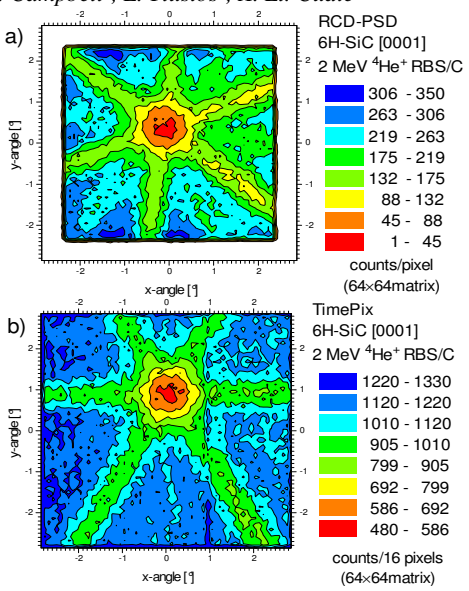

Fig. 1- Blocking patterns of 2 MeV ^{4}He$^+$ backscattered around the [0001] axis of a 6H-SiC crystal, measured with the a) RCD PSD, b) TimePix detector.

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[3]MEDIPIX, CERN, 1211 Geneva 23, Switzerland.

Laser grooved buried contacts (LGBC) on silicon solar cells for low concentration purposes

V. Corregidor, L.C. Alves, J. Wemans[1], G. Sorasio[1]

At a certain solar concentration level the efficiency of solar cells decreases due to power dissipation. One of the main factors responsible for this behaviour is the front metal grid, which should be optimized as well as the grid shading factor. For silicon solar cells working at low concentration levels (up to 10 times), the LGB contacts are an attractive solution for the production of low-cost concentrator cells able to handle the large current densities produced under concentration conditions. Silicon cells with LGB contacts, manufactured by NaREC and bought by the WS Energia, were characterized by PIXE. When the edge of a cell is analysed the Cu-rich buried contact can be clearly observed (Fig. 1). It is shown as well that the copper metallization also contains some Ni. On the other hand, Ag is only in the front surface with an irregular pattern. It is crucial to obtain a homogeneous distribution of the elements along the contacts since even under low concentration conditions, ohmic losses at the grid metal-semiconductor interface are critical to the efficiency of the cell.

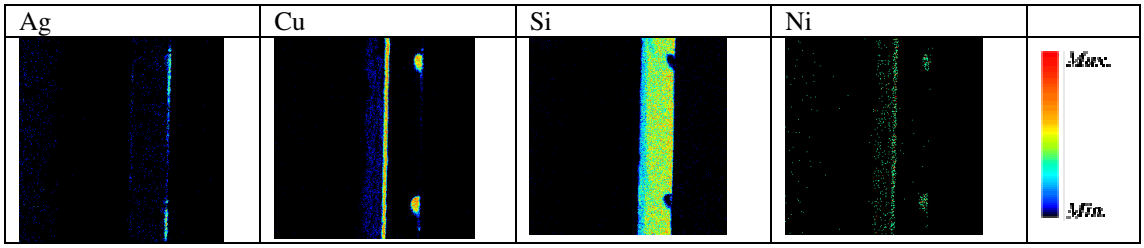

Fig. 1- 530x530 μm^2 PIXE maps. Elemental distribution of Ag, Si, Cu and Ni in the edge of the cell. Two fingers of the front metallization grid are shown.

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Generation of defects on CdZnTe (CZT) detectors due to metallisation

V. Corregidor, E. Alves, Q. Zheng[1], F. Dierre[1], J. Crocco[1], H. Bensalah[1], E. Diéguez[1]

The influence of deposition methods and type of metal (Au, Pt and Ru) contacts on the defects generated at the metal/semiconductor interface has been investigated by means of photoluminescence (PL) at low temperature, current-voltage characteristics, RBS and gamma response. Among the metals studied, Au and Pt have the best I-V characteristic curves and gamma response. In all cases there is a tellurium oxide layer between the substrate and the contact, but in the case of the thermal evaporation method this layer is very thin. In the case of the electroless method there is a wide ($750x10^{15}$ at/cm^2) interface with a strong mixing of Au, TeO_2 and CZT. This inter-diffusion mechanism is clearly observed in the A-center region of the PL spectra and in the increase of the (A-X) intensity line. The gold contacts deposited by electroless method showed the best results in terms of gamma response and I-V ohmic behaviour.

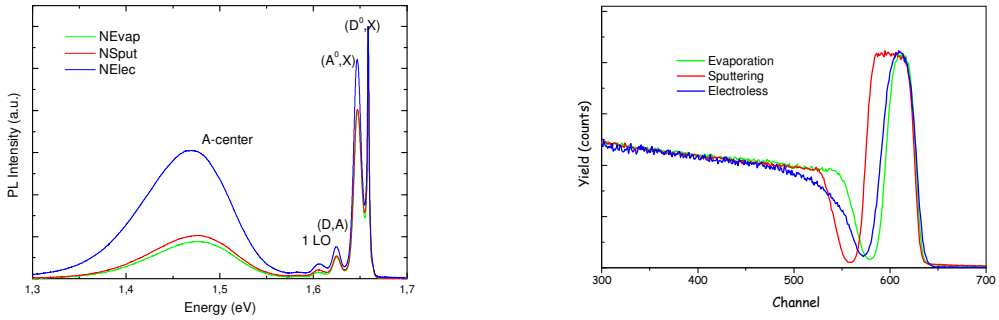

Fig. 1- The PL and the RBS spectra recorded for Au contacts deposited by different methods (thermal evaporation, sputtering and electroless).

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Formation of transition metal nitrides by N^+ ion implantation

A.R.G. Costa[2], M.M. Cruz[1,2], M. Godinho[1, 2], N. Franco, R.C. da Silva

Seeking for magnetic metal nitrides, iron, cobalt and nickel plates of purity 99.9% were implanted at room temperature with 50 keV N^+ ions to fluences of 2×10^{17} cm^{-2} and 5×10^{17} cm^{-2}, in order to study the influence of implanted nitrogen concentration in the production of nitride phases. Rutherford Backscattering Spectrometry with 2 MeV He^+ ions along with the RUMP code allowed calculating the effective implanted fluences as ~1.9×10^{17} cm^{-2} and ~3.5×10^{17} cm^{-2} respectively. The presence of nitride phases Fe_2N and Ni_3N was detected by XRD. Conversion Electron Mossbauer Spectrometry was also used with the iron samples, showing the presence of Fe_2N and also different $Fe_{3-\delta}N$ phases after annealing at 250 ºC. Iron and cobalt single crystals with different orientations were implanted at room temperature with 5×10^{17} cm^{-2}, 50 keV N^+ ions$^+$, under 10º incidence angles to avoid channelling effects. The effective fluence was evaluated as ~3.4×10^{17} cm^{-2} for cobalt and (110) Fe crystals, and ~3.0×10^{17} cm^{-2} for the (100) and (111) Fe crystals. Phase identification in both cobalt and iron crystals by XRD showed that Co_2N forms in *m*-cobalt but not in *c*-cobalt, while Fe_2N was identified for all Fe crystal orientations.

[1] Dep. Física, FCUL; [2]CFMC, UL.

Characterization of neutron irradiated Be samples

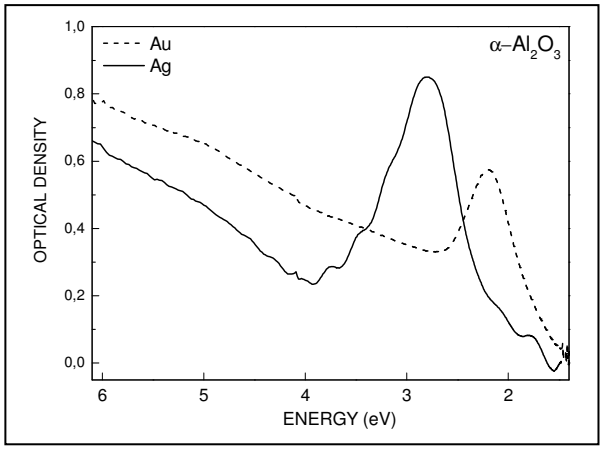
L.C. Alves , E. Alves, N. Catarino

Related with the study of advanced fusion reactor materials, four sets of Be pebbles and two different composition Ti beryllide samples, all of them subjected to a high neutron flux irradiation, were analysed with Ion Beam Analytical techniques using a Nuclear Microprobe. The main objectives were to determine possible structural changes after irradiation and extent of surface oxidation. Complementarily, gamma spectra were accumulated once the samples presented a low level of induced radiation. Comparison on the oxidation behaviour of the sets of pebbles provided by the same manufacturer but with different diameter size (0.5 mm and 1 mm) was also established as well as the dependence with irradiation temperature (425 ºC, 525 ºC and 750 ºC). Surface oxidation extent showed no relevant dependence on pebble size although within one of the sets the 'typical' behaviour was difficult to established, with some of the pebbles presenting heavy N contamination of unknown origin.

Surface morphology, thermal and electrical conductivity of α-Al_2O_3 single crystals implanted with Au and Ag ions

B. Savoini [1], M. Tardío [1], R. Ramírez[1] C. Marque, E. Alves

α-Al_2O_3 single crystals with *c*, *m* and *r* orientations, ion implantation at room temperature, with Au or Ag ions, energy of 160 keV and fluences ranging from 5×10^{15} to 1×10^{17}ions/cm^2. The presence of metal precipitates was explored by AFM and monitored by the variation of the intensity of the extinction bands associated with Mie scattering from Au and Ag colloids located at about 2.2 eV (563 nm) and 2.8 eV (443 nm), respectively (Fig.). The results show a dependence on the surface roughness with the ion fluence and the implantation orientation. The *I-V* characteristic of the electrical contacts reveals an ohmic behaviour, independent of the ions implanted and the crystallographic orientation. Measurements of the electrical conductivity at different temperatures in the investigated range predominantly suggest a band conduction mechanism thermally activated, with energies of about 0.09 eV and 0.07 eV, in samples implanted with Au or Ag ions, respectively. We conclude that the enhancement in conductivity observed in the implanted regions is related to the intrinsic defects created by the implantation, rather than to the implanted Au and Ag ions.



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TiN_x coated polycarbonate for bio-electrode applications

P. Pedrosa[1,245], E. Alves, N.P. Barradas, P. Fiedler[3], J. Haueisen[3] F. Vaz[4], C. Fonseca[1,2,5]

Titanium nitride (TiN_x) thin films were deposited by PVD, in a wide range of compositions ($0 < x < 0.99$), on polycarbonate (PC) substrates, aiming at studying their potential application as bio-electrodes. The electrochemical study of the TiN_x films, performed in an isotonic sodium chloride solution, proved the very good chemical stability of all films in salt solution conditions. On the other hand, the electrochemical noise analysis showed that the electrical noise generated at the stoichiometric TiN/electrolyte interface is of the same magnitude as that generated by the traditional Ag/AgCl electrodes. The lower impedance values of the films will facilitate bio-signal transfer in the presence of a sweat layer.

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[2] SEG-CEMUC –Universidade de Coimbra, Portugal.
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The width of an RBS spectrum revisited: influence of multiple scattering

N.P. Barradas

P. Bauer, E. Steinbauer, and J.P. Biersack calculated with Monte Carlo the RBS spectrum of 100 keV protons backscattered from a 100 nm thick Au foil (P. Bauer et al. NIM B64 (1992) 711). They stated in the caption of Figure 2 that a fairly large area, located to the left of the single scattering signal, "corresponds to the contribution due to multiple scattering". This led to the widespread belief amongst code developers that "multiple scattering manifests itself mostly as an additional contribution to the energy spread and as small low energy tails on signal edges arising from non-Gaussian wings in the energy distribution. (…) The small tails have not, so far, been modelled analytically." (E. Rauhala et al. NIM B244 (2006) 436. A new analytical calculation is now presented, showing that an extra yield due to multiple scattering indeed exists, but it is much smaller than the hatched area in Figure 2 of Bauer et al. That area is not an additional contribution of multiple scattering, it is simply the width of the low energy edge of the single scattering signal, which is broader due to the extra energy spread coming from multiple scattering.

The electronic transport mechanism in indium molybdenum oxide thin films RF sputtered at room temperature

E. Elamurugu[1], P. Shanmugam[1], G.Gonçalves[1], R. Martins, [1] E. Fortunato[1], N. Franco, E. Alves

Indium molybdenum oxide (IMO) thin films were radio-frequency (RF) sputtered at room temperature (RT) and studied as a function of base pressure (BP). The crystallinity of the films is decreased with the increase in BP. A maximum mobility (μ) of 49.6 cm^2 V^{-1} s^{-1} was obtained from the IMO films deposited at RT without any post-annealing treatment. The electronic behaviour of the deposited films was investigated by temperature dependent (100-550K) Hall measurements. Study on the scattering mechanisms based on the experimental data and theoretical models show that the ionized scattering centres are dominating. The films possess wide work function (4.91 eV) and high transmittance (> 70 %) over visible-near infrared (NIR) range. The results obtained, especially the high work function and NIR transmittance, are promising that the deposited IMO films may be useful in the applications such as solar cells.

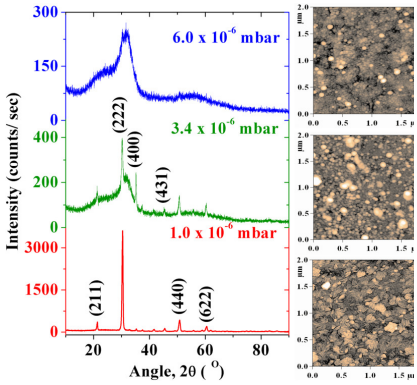

Fig. 1- XRD patterns of IMO films as a function of base pressure; AFM microstructures are shown next to the corresponding XRD

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Structural characterization of FeSiBCuP samples melt quenched under high gravity

R.M.S. Martins, I. Takeuchi [1], Y. Furuya [2], N. Schell [3]

Considerable attention is devoted to Fe-based magnetic nanocrystalline alloys, which show excellent soft magnetic properties. However, an efficient control of their average grain size is required for a significant decrease of the effective magnetic anisotropy. At Hirosaki University in Japan, FeSiBCuP nanocrystalline soft magnetic alloys were prepared by ultra high G quenching technique. Here, we report the experiments carried out at the High Energy Materials Science beam line HEMS at PETRA III, DESY, which offers excellent research possibilities to investigate fine structural details. The α-Fe phase was found to be the primary phase present in the studied samples. Fe_2B was also noticeably in all samples. X-ray diffraction patterns obtained for samples with Fe 83.3 at% and with Fe 85.3 at%, processed under the same gravity conditions (5000 G), showed that the formation of Fe_3B compounds is much more pronounced for the sample with higher Fe content. The study of samples with Fe 87.3 at% processed under 4000 G and 5000 G showed that higher gravity conditions promote the development of Fe_3B compounds.

[1] University of Maryland, USA,[2] Hirosaki University, Japan,[3] Helmholtz-Zentrum Geesthacht, Germany.

Study of new colouration processes in glasses

M. Ventura[1,2], A. Ruivo[2,3], A.P. Matos[2], L.C. Alves, R.C. da Silva

As part of a collaboration research on new processes of colouring glasses, industrial float glasses were colour doped with noble metal ions of Cu, Ag, and/or Au. The coloured glasses had both their faces characterized by RBS and PIXE, seeking at understanding the role of Sn in the final depth distribution and aggregation state of the noble metal ions in the glass matrix, in correlation with the observed colours. The results obtained so far show that in the case of Cu-doping by spray pyrolysis onto hot glass surfaces, Cu concentrates at the surface reaching to higher concentrations in the Sn-bearing faces, diffusing inwards and developing only a red tint upon annealing. In contrast, the glasses doped by dipping in mixtures of molten Cu compounds display a strong ruby red colour and correspondingly intense SPR optical band, along with much stronger Cu Kα intensities in their XRF spectra, but significantly lower RBS yields that extend to low energies. This is a clear indication of extensive diffusion with formation of a large number of Cu nanoparticles, evenly dispersed along several micrometres from the surface of the glass matrix. As the effect is much more marked in the Sn-bearing face Sn does have a role in the colouring process. In the cases of Ag and Au colouring by thermal reduction of a multi-layered polyelectrolyte dip coatings doped with the noble metal ions, the RBS and PIXE analysis indicate generically similar trends: Ag and Au tend to concentrate more in the Sn-bearing faces.

[1] DCR/FCT-UNL; [2] VICARTE/FCT-UNL; [3] REQUINTE/FCT-UNL.

Limited angle STIM-tomography

A.C. Marques, D. Beasley, L.C. Alves, R.C. da Silva

Building on work initiated in 2010, preparing the setup and software for ion beam tomography, morphological characterization of SiC grains (~30 µm in diameter) immobilized in formvar fibers was performed by STIM tomography in off-axis geometry. The data gathered allowed morphological reconstruction and showed it will allow determining 3D density distribution. Despite the improvements made on the existing setup and procedures, there remain positioning and alignment problems with the rotation axis: some degree of misalignment seems inevitable. Two types of solution were devised to mitigate these problems: *i*) assembly of a positioning stage with 5+1 degrees of freedom is being implemented, and *ii*) pre-processing software with alignment algorithm was developed and tested. Aiming at better reconstruction an algorithm allowing the efficient removal of background noise was also implemented. These were integrated in a common graphical user interface, *TomoAlign*. Use of the BFP-based *Tomo3D* software shows that a reasonable reconstruction is achievable after suitable data pre-processing of digital alignment (cf. Fig.1).

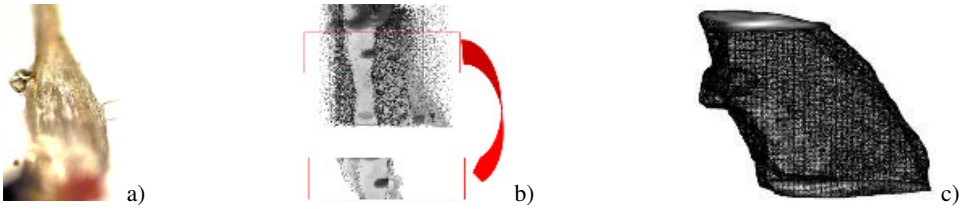

Fig.1 a) Microscope image of the STIM-T analyzed formvar fiber with SiC grains; b) projections acquired over 180º rotation range in steps of 10° (top), and one section cropped after pre-processing (bottom). By BFP the 3D image in (c) was obtained. Besides alignment corrections pre-processing included the removal of noise and of spikes.

Synchrotron radiation-based micro-computed tomography applied to study embryonic dinosaur fossils

R.M.S. Martins, F. Beckmann [2], R. Castanhinha [1,3], O. Mateus [1,4], R. Araújo [1,5], P.K. Pranzas [2]

One of the most relevant specimens of the Museum of Lourinhã is the nest found in Paimogo, in 1993, with embryos attributed to the theropod dinosaur *Lourinhanosaurus antunesi*. In this nest more than 100 eggs or eggshell concentrations were detected. The embryonic remains were found as isolated skeletal elements in ovo or in close association with eggs. The site is late Jurassic in age, which fills an important temporal and phylogenetic gap of the fossil record regarding theropod embryos. Furthermore, it belongs to a clade widely regarded as ancestors of birds. Several embryonic vertebrae have been studied by Synchrotron Radiation-based Micro-Computed Tomography (SRµCT) at the BW2 beam line. We have obtained high-resolution 3D tomographic datasets using a non-destructive procedure. A photograph showing a dinosaur embryonic vertebra (ML565_Paim55) found in the Paimogo nest is presented in Fig. 1 (a). The specimen was imaged by microtomography in absorption mode with photon energy of 24 keV. Microtomographic slices through the vertebra are shown in Fig. 1 (b)-(c). The exquisite anatomical and histological preservation of the embryonic bones will allow observations of unprecedented precision and detail for theropods. This material allows us to extend in time and to considerably supplement in great detail our knowledge of early ontogeny of carnivorous dinosaurs.

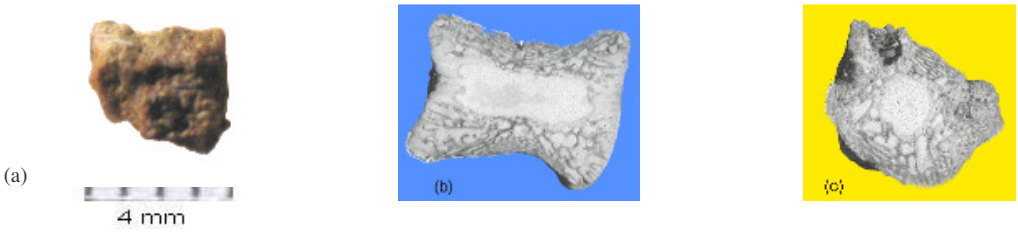

Fig. 1- Embryonic vertebra of *Lourinhanosaurus antunesi* found in the nest recovered from Paimogo site: (a) photograph of the vertebra; (b) longitudinal section of the vertebra; (c) general view of a centrum cross-section of the vertebra.

[1] Museu da Lourinhã, Portugal; [2] Helmholtz-Zentrum Geesthacht (HZG),Germany; [3]IGC, Portugal;
[4] FCT, Universidade Nova de Lisboa, Portugal; [5] HDES, Southern Methodist University, USA.

Hydrogen retention in W-Ta composites proposed for use in first wall applications

R. Mateus[1], M. Dias, J. Lopes[2], J. Rocha, V. Livramento[1,3], J.B. Correia[1,3], P.A. Carvalho[1,4], K. Hanada[5], E. Alves

In opposition to W-Ta alloys, in W-Ta composites the individual phases remain quite separated within the microstructure and the behaviour of the final material under irradiation depend of the properties of pure components. W and Ta plates and W-Ta composites consolidated via spark plasma sintering (SPS) were implanted with energetic He^+ and D^+ ion beams at different temperatures (RT, 200 and 400 ºC). The pre implantation with He^+ induces the blistering of the He enriched surfaces, in particular in the Ta phase. However, the induced retention of D is only significantly increased in W plates while for Ta the increment is much weaker. This behaviour is explained by the lower radiation resistance of W relatively to Ta. As a consequence, hydrogen retention in W-Ta composites is preferentially associated to the W component and to the composite production route. The results also evidence that the hydride phase (Ta_2H) in pure Ta is not stable at higher temperatures. Furthermore, the hydride phase could be avoided in the W-Ta composites by controlling the SPS consolidation temperature and the interdiffusion between the components, which is moderate below 1300 ºC due to mainly diffusion of W into Ta.

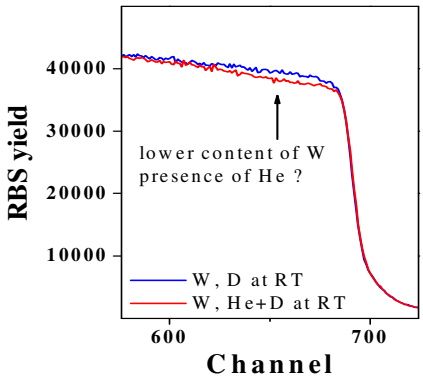

Fig. 1- RBS spectra of pure W implanted with ^2H and $^2H+^4He$.

[1] Instituto de Plasmas e Fusão Nuclear, Instituto Superior Técnico, Lisboa, Portugal; [2] Instituto Superior de Engenharia de Lisboa, Lisboa, Portugal;
[3] Laboratório Nacional de Energia e Geologia, Lisboa, Portugal;
[4] ICEMS, Departamento de Bioengenharia, Instituto Superior Técnico, Lisboa, Portugal;
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Determination of stopping powers of different ions in different systems

N.P. Barradas, E. Alves, Z. Siketić [1], I.B. Radović [1]

The knowledge and amount of stopping power measurements available for heavy ions is much smaller than for for H and He, with negative consequences for the accuracy of calculational schemes such as SRIM. We used a bulk method, previously developed by us and applied successfully to other systems, to determine experimentally the stopping power of He, C and O in Si, GaN and TiO_2. This is particularly useful for heavy ion elastic recoil detection analysis, given that Si is one of the most commonly analysed materials, and C and O are light elements which quantitative determination is ideally made by HI-ERDA. We compare the results with SRIM and MSTAR as well as with other data.

[1] Ruđer Bošković Institute, P.O. Box 180, 10002 Zagreb, Croatia.

Improving the CASSIS code for channelling computer simulations

A. Kling

Ion channelling is very powerful for the study of the real structure of monocrystalline materials (e.g. lattice site of foreign atoms, intrinsic defects, etc.). The development of the CASSIS code started more than a decade ago and continuously yielded extensions to investigate additional channelling phenomena (e.g. the influence of the incident angle on the channelling spectra in catastrophic dechannelling at strained layers interfaces, simulation of extended 3D-defects etc.). Due to the enormous progress in computer technology it was necessary to refurbish parts of the existing code by updating calculation methods using more detailed and/or higher precision algorithms. A long-standing problem was the lack of user-friendliness of the code. This has been resolved by introducing a simple input procedure for the most common channelling problems that is assisted by several automatic procedures (e.g. determination of the crystallographic system and the necessary vectors for the simulation from the input data). The improved speed of the program allows now also the calculation large-dimension 2D-scan plots that may be useful for the determination of useful crystallographic directions in complex crystalline systems not studied so far.

Laboratory operation and development – accelerators data acquisition and motion control automation
N. Catarino, C. Cruz, E. Alves, L.C. Alves, R.C. da Silva

Previously detected functional instabilities in the interplay with the data acquisition software lead to a thorough revision of the goniometers motors automation control code. The causes were identified and the problem solved by changing the execution of critical external calls to synchronous mode operation. This further allowed elimination of delay patches making the code faster. The code sections related to the interaction with the data acquisition software, protections of mutually exclusive operations, and with the switching of control between experimental setups, were also rewritten in optimized form adding to its operational robustness. As part of code revision three important new features were introduced: *i*) an instance was added allowing automatic acquisition of long series of IBA spectra in repetitive characterization experiments of JET related materials; *ii*) an instance was developed allowing the execution of random spectra in RBS/C experiments, by sampling over user defined sets of coordinates, and *iii*) the simple graphical display of angular scan data was enhanced with new functionalities, among which: split/merge of individual scan data sets, vertical and horizontal scale expansion/compression, and selection of sub-scan and call to the viewing window.

Installation, test and implementation of the JET chamber and a new IBA beam line
N. Catarino, C. Cruz, L.C. Alves, *R.C. da Silva, E. Alves*

An experimental chamber dedicated for IBA characterization of JET related materials was received from Sussex University and installed as a removable insert in the PIXE beam line. For this a support base and sliding system were designed and manufactured enabling the alignment of the chamber and its removal when not in use. After successfully testing operational status was achieved by collecting RBS and NRA spectra, under correct measurement of the beam charge, a new stepping motor was fitted to the target loading port to allow automatic acquisition of IBA spectra during repetitive characterization experiments, under remote computer control.

In order to make operational a new multipurpose IBA beam line on the 3 MV tandem accelerator facility, endowing it with RBS/NRA/PIXE in combination with channelling, a new electronic drive system was planned for control and of the goniometer stepping motors and automation of the experiments. Contrary to the proprietary systems developed in-house two decades ago, and still in operation in the 2.5 MV VG accelerator facility, an off-the-shelve commercial solution was chosen, based on two RS 217-3611 stepper motors activation boards and one RS 440-098 programmable control board with built-in control firmware. These Eurocard boards intercommunicate via a 2×32 way-to-1×64 way DIN 41612 connectors and allow computer control through standard RS-232C ports. A control program was developed for testing the new system and preparing its integration in existing automation code.

Installation and optimization of a rapid thermal annealing furnace
S.M.C. Miranda, N. Catarino, E. Alves, K. Lorenz

Rapid thermal annealing (RTA) equipment ("AS-one 100" by Annealsys) was installed and tested for sample processing up to 1500 ºC. It is capable of reaching temperatures up to 1500 ºC in vacuum or gas atmospheric pressure using fast heating and cooling ramps. On-site facilities (power- and water supply) were installed in order to operate the RTA furnace. A gas supply system was projected and assembled which allows for annealing in N_2, Ar, and NH_3/N_2 atmospheres. RTA process operation and annealing recipes were optimized in order to improve functionality and safe handling, obtain reproducible results, and reduce overshooting above the desired anneal temperature. Annealing procedures and parameters were optimized for implant activation in GaN thin films. The adequate heat ramp setting was found to be 30 ºC/s and the highest temperature that could be reached without inducing dissociation of the sample was 1200 ºC in NH_3 containing atmosphere. NH_3 was found to stabilize the surface and reduce the out-diffusion of nitrogen.

Laboratory operation and development
A.R.G. Costa[1,2], R.C. da Silva

A prototype of an acid saw was designed, developed and assembled for cutting metal crystals producing the least damage by mechanical deformation. The cutting action is achieved by an acid soaked fiber wire undergoing a reciprocating movement with a net positive displacement, using two stepping motors (NMB PM55-048), which are controlled by an open-source single-board microcontroller Arduino®, via a driving circuit designed in-house that supplies the necessary power, under control of the microcontroller based firmware. The controlling code was developed in a proprietary ANSI-based C language and uploaded into the microcontroller.

[1] Dep. Física, FCUL; [2] CFMC, UL.

Biomedical Studies

Teresa Pinheiro

The activities of the Biomedical Studies group are the study of biomarkers of exposure and disease.

Research activities carried out are an end product of intense and interactive collaborative work among researchers in Cardiology, Pneumology, Dermatology, Biology, Biochemistry, Chemistry and Environmental Sciences.

Current projects join different groups from three ITN Units, Reactor, UCQR and UFA, which are working in consortium with other national and international research institutes, academia and hospitals.

Major research areas focused:

1) Clinical research targeting chronic diseases;

2) Environmental health problems related with metal exposures;

3) Methodological approaches to assess nanoparticles toxicity.

A variety of scientific and technical skills developed in Biomedical Studies group of ITN, involving proton microscopy, inductively coupled plasma mass spectrometry (ICP-MS), flow cytometry, biochemical methods and cell function evaluation techniques, helped consolidating the scientific niche and launching new areas of research.

Continued funding in the areas of environmental and biomedical sciences during the last years had strengthened existing skills and promoted advanced training of Ph.D. and M.Sc. students.

The main achievements of 2011 are summarised in the following pages.

Research Team

Researchers

T. PINHEIRO, Aux.
L.C. ALVES, Aux.

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R. VELOSO, Post-Doc
C. RAMOS, M.Sc., BI grant
P. FÉLIX, Ph.D. student, ITN/TU Delft

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R. PINHEIRO

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R. CRUZ FERREIRA, M.D., CHLC/EPE, Hospital Sta. Marta
M.D.YNSA, Researcher, CMAM, Univ. Autónoma Madrid, Spain

Endothelial function, inflammation and composition of the atherosclerotic plaque

P. Napoleão[1], C. Ramos, R. Cruz Ferreira[2], M. Mota Carmo[3], M. Selas[2], M.C. Monteiro[4], M.B. Criado[4], A.M. Viegas-Crespo[5], F. Caeiro[5], A. Turkman[6], V. Andreozzi[6], T. Pinheiro

At present, heart disease remains far and away the leading cause of mortality in developed countries. Although some of the traditional risks factors are linked with disease incidents there are no biomarkers for diagnosing coronary artery disease.

The main goals are the identification and characterization of the vulnerable plaque in hopes of identifying morphologic and physiological features that predict plaque rupture in coronary artery disease (CAD). Several molecules, T lymphocytes and endothelial progenitor cells (EPCs) are been studied, which may have relevant roles in endothelial function and in plaque rupture.

Results

Approximately 80 patients with CAD and 50 controls (CTR) were evaluated. Patients with acute myocardial infarction (AMI), sable (SA) and unstable (UA) angina, were enrolled in the study. Angiographic data of coronaries obtained during angioplasty and percutaneous intervention, and plaque biological characterization obtained with virtual histology intravascular ultrasound (VH-IVUS) are being related with clinical findings and circulating indicators, which may be associated with the disease.

Indicators of the endothelial function and plaque activity, such as vascular endothelial growth factor (VEGF), interleukins, oxidized low-density lipoprotein (ox-LDL), and T lymphocyte activation profile (CD69+ and CD25+), were some of the indicators that are being evaluated and detected in blood by flow cytometry and ELISA.

Results: In CAD patients the plaque content of fibrotic and calcified tissue were correlated with VEGF concentration in serum whereas high levels of ox-LDL were associated with larger plaque areas (PA) as depicted in Fig. 1.

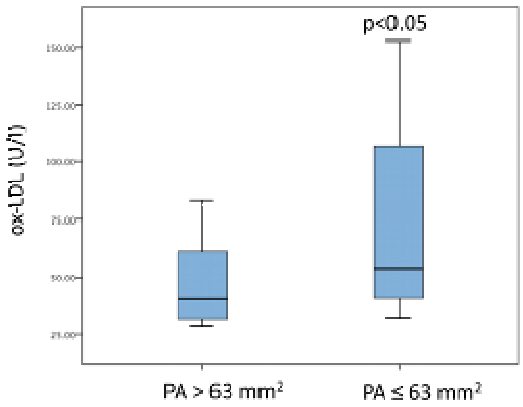

Fig. 1 – Box plots of ox-LDL concentration in two classes (low and high quartiles) of plaque area (PA).

The patients having larger plaque areas and higher levels of ox-LDL in circulation suffered from acute coronary syndromes and the condition was also related to low number of circulating EPCs and high T-cell activation.

CAD patients showed different profiles of T-cells, which express the activation markers CD69 and CD25. In controls T-cells express high CD69 and low CD25 whereas in patients CD69 expression decreased and CD25 augment. In AMI patients CD25- subset expressing high CD69 was most representative and in SA patients the proportion of CD69+ T-cells drastically decreased (Fig. 2). Therefore, the reduced expression of CD69 and activated CD25+ T-lymphocytes may favour immune reactivity of T-cells.

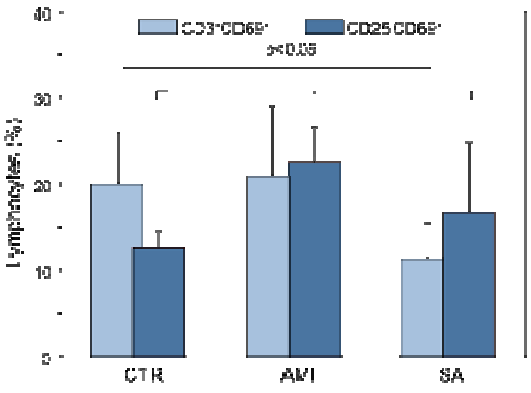

Fig. 2 – T-cells activation profiles in controls and CAD patients.

Conclusions

Circulating markers such as ox-LDL may express plaque vulnerability. The different activation patterns of T-cells in patients and subjects without CAD suggest that T-cell differentiation may condition the immune inflammatory response in these patients. In the overall, changes observed in biochemical indicators EPCs and inflammatory cells were associated to the most adverse disease outcomes.

[1] Instituto Medicina Molecular;
[2] Serv. Cardiologia, Hospital Sta. Marta, CHLC;
[3] Faculdade de Ciências Médicas, UNL;
[4] CESPU, Porto;
[5] CESAM, FC-UL;
[6] CEA, FC-UL.

Metal toxicity:interaction of nanoparticles with cells

M.D. Ynsa[1], A. Picado[2], A. Barreiros[2], L.C. Alves, T. Pinheiro

The key to understanding the toxicity of nanoparticles (NPs) is that their minute size, smaller than cells and cellular organelles, allows them to penetrate these basic biological structures, disrupting their normal function. Synthetic engineered nanomaterials, which has astonishing physical and chemical properties lead to an exponential use of these materials in multiple aspects of daily life. For example, titanium dioxide (TiO_2) NPs possess photocatalyst activity and are used as antibacterial coatings and in sunscreens. Iron oxide with magnetic properties (Fe_3O_4) is used in clinical diagnostic; quantum dots of CdSe are being explored as molecular probes.
The first aims of our study is to evaluate the toxic effect of NPs of similar equivalent spherical diameter (approximately 10-20 nm) and various elemental compositions on human stem cell lines and living organisms, planktonic crustaceans and aquatic plants. Other goal of our studies is to generate experimental set-up for a cytotoxicity screening of NPs toxicity.
The characterization of NPs is done with scanning electron microscopy and nanoparticle tracking analysis. Nuclear microscopy methods are used to identify NPs in living cells and organisms.
So far, exposure tests were performed in vitro and results on viability, biological response and NPs internalization obtained, though not for all models employed. Mesenchymal stem cells were exposed to functionalized NPs of CdSe and Fe_3O_4 and cell culture viability tests carried out. After 48h incubation with some of the NPs types produced, major morphological alterations were observed in cell cytoskeleton and nuclei. For those that were viable the exposure conditions still need to be tuned. Planktonic crustaceans and aquatic plants were exposed to various concentrations of TiO_2. The NPs distributions in plant tissues from root to leaves were inspected. Major deposits were observed at the external wall in roots and leaves as can be observed in the figure below (Fig. 1). However, minor internalization of NPs in plant tissues was observed.

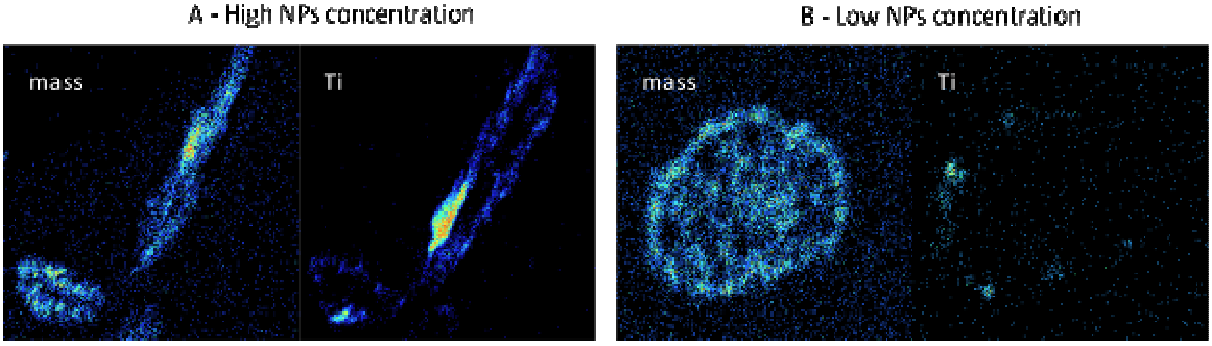

Fig. 1 - Aquatic plant (Lemna) exposed to high and low concentration of NPs of TiO2. Nuclear microscopy images of the mass density and Ti distribution in root sections. NPs deposits were observed at the external wall of roots (Ti maps). A – two sections of roots: longitudinal (centre) and transversal (bottom left) (300x300 μm^2); B – transversal section of the apical root (53x53 μm^2). Rainbow colour scale: minimum content, dark blue and maximum content, red.

[1] CMAM, Univ. Autónoma Madrid; [2] LNEG

Metal bio-availability in water-sediment interfaces – a micro-distribution evaluation.

R. Veloso[1], C. Vale[1], T. Pinheiro

The main objectives are to understand the micro-distribution of trace elements across the interfaces between salt marsh sediments and inhabitant organisms and between sediment and water in natural environment. The micro-distribution of trace elements in sediment profiles were carried out by Proton Microscopy to assess the concentration gradients and infer metal fluxes, especially Mn and Fe, from sediment to the overlying water. Also the elemental profiles between sediments and benthic organisms are being investigated to elucidate whether metals are sorbed on the cellular or tissue wall or uptake by the organism. The balance of these metals at sediment-water interface are being investigated.

[1] INRB-IPIMAR.

Elemental Characterization and Speciation
CEEFI

Miguel A. Reis

The Elemental Characterization and Speciation work line of ITN Ion Beam Laboratory (CEEFI/LFI), carries out R&D work on ion beam based nuclear analytical techniques aiming at elemental composition characterization and instrumental speciation methods. Focusing being on applications to small mass samples (self-supported thin films, micro and nanoparticles) and/or small mass structures (deposited and deep laying thin films, embedded micro and nanostructures).
The main issue being originally, particle induced x-ray emission (PIXE) applied to the characterization of airborne material and its impacts, lead to the installation of an aerosol characterization set-up, which includes a DOAS system (operational since June 2007), a meteorological station (operational since January 2008), on-line in the Portuguese Meteorological Institute Urban Stations Network, and a PM10 and PM2.5 sampling station.

The installation, in 2008, of the High Resolution High Energy PIXE (HRHE-PIXE) set-up at ITN, the world first cryogenic high resolution EDS X-ray microcalorimeter spectrometer (XMS) based PIXE system, lead to very important results showing the advantages of these detectors use for both fundamental research as well as applications.

As a consequence of this a revision of the objectives of the CEEFI main work line was carried out, and the emphasis was shifted towards fundamental, technical and software development for PIXE, as well as frontier applications of the technique.

Within the organics of the Ion Beam Laboratory (LFI), CEEFI is the responsible for the maintenance and improvement of PIXE facilities.

In respect to 2011 developments, focus was put in the quantification of qualitative results developments of 2010 and previous. Besides this, the High Energy component of the HRHE-PIXE system installed in 2008, was promoted and the automation of the HRHE-PIXE positioning system was carried out. Very interesting results were achieved using high beam energy PIXE. In respect to the XMS-PIXE system, comparisons to high resolution data obtained in the Institute Josef Stefan WDS system was initiated but only preliminary results could be achieved so far.

Finally, the quest for quantification lead the team to deepen the relation to a more theoretical work group from the Atomic Physics Centre of the University of Lisbon (CFAUL), which collaboration is looking highly promising and providing the first solid ground results.

Regarding frontier applications, geological samples, nanoparticles, thin films, and chemical or electronic structure environment mapping are presently the main scope of CEEFI work line.

Within this context, during 2011, important results on geological samples analysis, thin films and fundamental developments were published.

Work in the scope of aerosol analysis was reduced to nearly zero in 2011, due to the raise of importance of more fundamental work for the proper quantification of achieved technological developments, but also as a consequence of the present general financial context.

Research Team

Researchers

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P.C. CHAVES, Post. Doc. Fellow under the EU SPIRITproject.

Students

A. TABORDA, Ph.D. Student, FCT grant

Technical Personnel

R. PINHEIRO (30%)

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J.M. MARQUES, Aux. Prof, CFA-UL, FC-UL

Fundamental Developments and Solid State Effects in PIXE

M.A. Reis, P.C. Chaves, A. Taborda, N. Barradas, A. Carvalho[1], L. Carvalho[2], J.P. Marques[2], J.P. Marques[2], J.Dias[3], M.Kavcic[4]

Objectives

Elemental and speciation characterization methodologies for small mass samples, are gaining importance world wide and are a niche were ion beam analysis (IBA) may come to play an important role, with special emphasis to high resolution particle induced X-ray emission (HR-PIXE). During 2011 most of the line activities therefore focused on mostly on objective of carrying out fundamental developments related to the identification of solid state environment effects on emitting ion, which reflect upon the X-ray spectra details. This objectives are the continuation of the main objectives of the previous year of 2010, and correspond to the objectives stated in a sub-task of Key task 15 of the 7th FP project "Support of Public and Industrial Research Using Ion Beam Technology (SPIRIT)", Grant agreement No 227012-CP-CSA-Intra (starting date of 2009/03/01), in which the team plays an important role.

As a consequence of this work, technical details lead to the establishing of an associated objective, namely the application of these developments to geological samples.

Results

During 2011, the Ph.D. program of P.C. Chaves was concluded. The know-how acquired during the Ph.D. was used in the frame of the SPIRIT project task, to install the X-Y positioning system of the High Energy High Resolution PIXE system, end-station of the ITN 3MV tandetron, and to carry out experiments for comparison of the ITN energy dispersive X-ray microcalorimeter spectrometer (XMS) spectra form a Si crystal target, and equivalent ones obtained using the wavelength dispersion system of the Institute Josef Stefan, which has much better resolution but much shorter energy window.

In the context of the ongoing Ph.D. programme of Ana Taborda, a new collaboration was initiated with the Federal University of Rio Grande do Sul, Brazil and a three months permanence there allowed the study of Gd2O3 and Tb4O7 nanoparticles, irradiated at different energies. Fig.1 (right) show the unexpected differences in the M spectra of Tb4O7 nanoparticles obtained if these are dispersed or packed in a pellet. In a three months stay at UFRGS, Ana Taborda also carried out Medium Energy Ion Spectrometry - MEIS analysis of these nanoparticle samples.

Still within the scope of this Ph.D. programme, the collaboration in the frame of the CFAUL was increased, starting with *ab initio* calculation of Si spectrum data, to be compared with data from the XMS-PIXE spectrum and from the Ljubljana WDS spectrum of a Si crystal sample.

Results for both collaborations displayed important problems with no immediate answer. In the case of MEIS, un-explained shifts were identified, while for *ab initio* calculations, experimental data shows to be more problematic than expected in the capacity to reproduce (even in simple cases such as that of Si target).

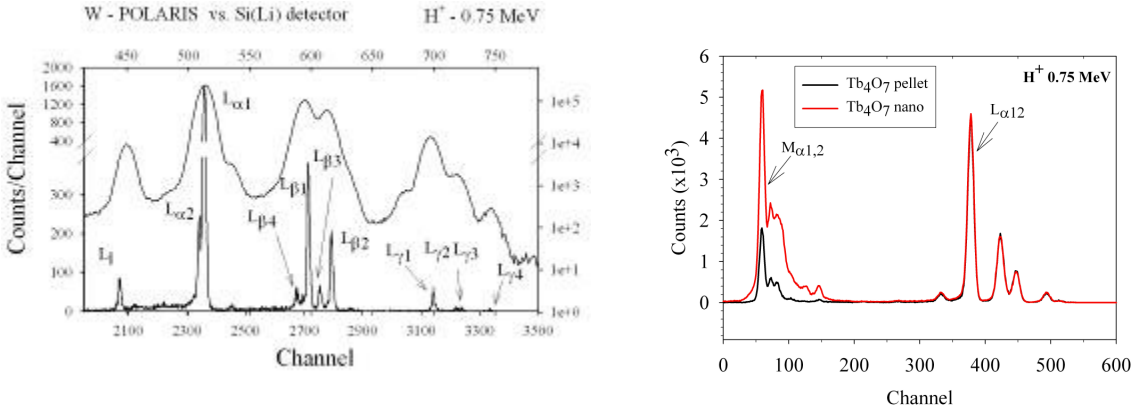

Fig.1 (Left) Comparison of X-ray microcalorimeter spectrometer PIXE system (XMS-PIXE) and Si(Li) spectra of a pure W thick target foil irradiated by a 0.75MeV proton beam provided by the ITN 2.5MV Van de Graaff (Si(Li)) and by the 3.0 MV Tandetron (XMS-PIXE). The clear separation of lines from the XMS-PIXE unit was of major importance for the understanding of results in the scope of P.C: Chaves Ph.D. thesis. (Right) Comparison of the M-shell spectra of Tb4O7 nanoparticles irradiated by a 0.75 MeV protons beam provided by ITN Van de Graaff. and physically disposed in two different ways, namely as a dispersion of nanoparticles in a Nuclepore filter and when packed into a pellet..

[1] CENIMAT, Fac. Ciências e Tecnologia da Univ. Nova de Lisboa, Portugal;
[2] Centro de Física Atómica da Univ. de Lisboa, Fac. Ciências da Univ. de Lisboa, Portugal.; [3] Instituto de Física, Univ. Federal do Rio Grande do Sul, Brazil;
[4] Institute Josef Stefan, Ljubljana, Slovenia.

Materials Characterization with Radioactive Nuclear Techniques

João Guilherme Martins Correia

A **laboratory infrastructure** dedicated to advanced materials research is maintained and developed at ISOLDE-CERN by the Nuclear Solid State Physics group of ITN and CFNUL. ISOLDE is a European Large Scale Facility that produces highly pure elemental and isotopic ion beams of more than 1000 isotopes and isobars of 80 elements, which is a unique feature in the world. In this context nuclear techniques such as Emission Channelling (EC) and Perturbed Angular Correlations (PAC) provide local- atomic-scale information complementing material analysis capabilities at ion beam, x-ray and microscopy laboratories. This infrastructure and related projects are refereed and evaluated each year within the scope of FCT - CERN projects. The scientific work in 2011 was centred in several research subjects approved by the ISOLDE-CERN (INTC) Scientific Committee:

1) IS453 (U.Wahl) "Emission Channelling Lattice Location Experiments with Short-Lived Isotopes". EC experiments study the lattice sites of dopants and impurities in scientifically and technologically relevant semiconductors (e.g., Si, Ge, ZnO, GaN and GaAs). Due to previous years efforts, elements having only suitable short-lived isotopes can now be studied on-line. In 2011, for the first time, Nickel sites were studied using the ^{65}Ni (2.5 h) isotope.

2) IS487 (V. Amaral) "Study of Local Correlations of Magnetic and Multiferroic Compounds". PAC studies a large variety os multiferroic manganites and cromites. By combining PAC data with first principle simulations of charge density distributions on these materials, local phenomena responsible for the coexistence of ferroelectricity, ferromagnetism and ferroelasticity are studied. 2011 was a year of concluding experiments, publications and of new discoveries, as the findings in $CdCr_2S_4$ that the puzzling relaxer behaviour in the paramagnetic (PM) regime is correlated to atomic displacements, where a new dynamic state caused by the presence of simultaneous polar and magnetic clusters is revealed.

3) IS481- addendum approved by INTC on Feb. 2011 (K. Lorenz) "The role of In in III-nitride ternary semiconductors", have combined γ-γ, e-γ and β-γ PAC using the ^{117}Cd/^{117}In, ^{111m}Cd/^{111}Cd, and the ^{115}Cd/^{115}In isotopes. The aim is to study the intrinsic nature of In/Cd defects in $Al_xGa_{1-x}N$ alloys, in order to understand the role of In, in the extraordinary luminescence performance of III-nitride semiconductors and to investigate Cd as a possible p-type dopant.

In early spring two new research proposals have been approved (V.Amaral): *"Radioactive probe studies of coordination modes of heavy metal ions from natural waters to functionalized magnetic nanoparticles"*, and a letter of intent, *"Radioactive Local Probing and Doping on Graphene"*.

2011 will further stay as a reference year due to the donation by the Leipzig University to ITN of their full PAC laboratory, just matching the essential needs of our groups and projects at ISOLDE to run multiple experiments in parallel during beam times. From the three highly efficient 6- detector arrays delivered, we point the new and unique digital spectrometer equipped with 6- $LaBr_3$ detectors and FPGA data acquisition and processing technology. Thanks to this setup, with outstanding energy and time resolution, we report the first ever done PAC measurement onto decay of ^{61}Cu/^{61}Ni, a probe element adequate for nanoscopic scale studies on solid-state and bio-physics materials.

In parallel, the new high-resolution Panmure goniometer, dedicated to on-line experiments with short-lived isotopes, was commissioned on-line at the GHM beam-line, a new arc-furnace has been tested for synthesising and study special compounds *in situ*, and new fast PAD detectors were tested for EC experiments.

Of interdisciplinary nature, these activities integrate and initiate students from different backgrounds and universities, in applied nuclear physics,with shared work between the different environments of ITN and ISOLDE. The work is carried out by students and senior researchers from the universities of Lisbon, Aveiro, Porto, Braga, ISEL as well as from Leuven in Belgium and Bonn in Germany. During 2011 three Ph.D. students defended their thesis; five other Ph.D., two M.Sc. and one Diploma students are performing their work using this infrastructure within the scientific proposals and R&D projects.

Research Team

Researchers

J.G.M. CORREIA, Princ.
U. WAHL, Princ.(75%)
E. ALVES, Princ.(15%)
K. LORENZ, Aux., FCT (15%)

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M.B. BARBOSA, Ph.D. FCT grant
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P. KEßLER, Ph.D. HISKP Bonn grant (e-γ PAC)

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H. HAAS, Emeritus Researcher, ITN
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S. DECOSTER, Post-Doc, IKS Leuven
R. VIANDEN, Prof., HISKP Bonn

IS453 experiment: Emission channelling lattice location studies

U. Wahl, J.G. Correia, E. Alves, L. Pereira[1,2], S. Decoster[1], L. Amorim[1], A. Vantomme[1], D.J. Silva[2], J.P. Araújo[2], M.R. da Silva[3,4], E. Bosne[5] and the ISOLDE collaboration

Objectives

Lattice location of diluted impurity-dopant elements on relevant materials.

Results

1. Mixed Zn and O substitution of Co and Mn in ZnO

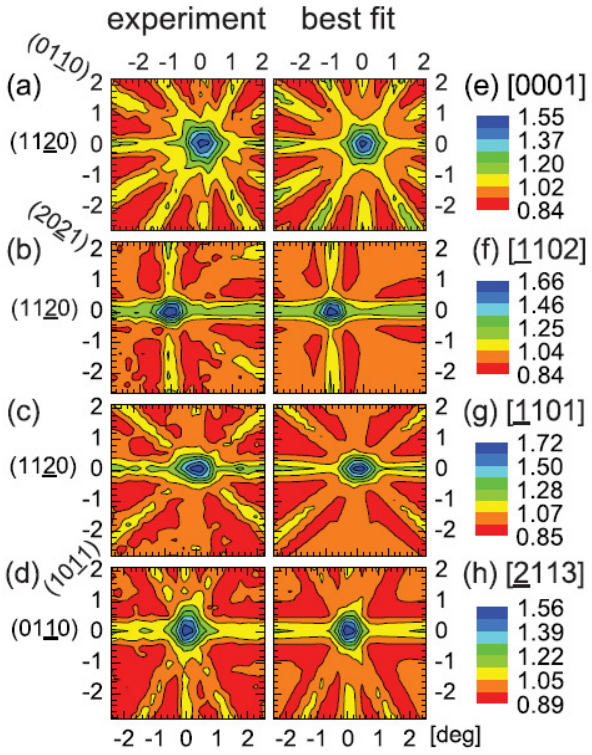

Fig. 1: (a–d) Normalized β^- emission channeling patterns from ^{61}Co in ZnO in the vicinity of the [0001], [-1102], [-1101], and [-2113] directions. (e–h) Corresponding best fits with 82% and 18% of the ^{61}Co atoms on S_{Zn} and S_O sites, respectively.

The physical properties of an impurity atom in a semiconductor are primarily determined by the lattice site it occupies. In general, this occupancy can be correctly predicted based on chemical intuition, but not always. We report on one such exception in the dilute magnetic semiconductors Co- and Mn-doped ZnO, experimentally determining the lattice location of Co and Mn using β^- emission channelling from the decay of radioactive ^{61}Co and ^{56}Mn implanted at the ISOLDE facility at CERN. Surprisingly, in addition to the majority of doping ions substituting for Zn, we found up to 18% (27%) of the Co (Mn) atoms in O sites, which is virtually unaffected by thermal annealing up to 900 °C. As possible reason for the anion site occupancy we tentatively propose that the incorporation might be due to these transition metals assuming 4+ charge states when replacing O atoms under vacancy rich conditions such as those resulting from ion implantation. However, since an anion site configuration had never been considered before for any transition metal in any metal oxide material our experimental results suggest a change in paradigm regarding transition-metal incorporation in ZnO and possibly other oxides and wide-gap semiconductors.

2. Emission channelling with short-lived isotopes

In 2011 we performed three beam times with short-lived radioactive isotopes using our emission channelling on-line setup at the ISOLDE GHM beamline. During the Mg beam time we determined the lattice location of the potential acceptor ^{27}Mg (9.5 min) in GaN and AlN for implantation temperatures from RT to 800 °C. While the majority of Mg was found on substitutional Ga and Al sites, for the first time also a minority fraction on hexagonal interstitial sites could be clearly identified. In both cases the interstitial Mg was converted to substitutional Ga or Al sites at temperatures between 600 °C and 800 °C. During the Mn beam time we used the decay chain ^{61}Mn (4.6 s)→^{61}Fe (6 min)→^{61}Co (1.6 h) to determine the lattice location of Co in n-Si and p^+-Si, where the co-existence of Co on substitutional and tetrahedral interstitial sites was found. As a new radioactive probe we used for the first time ^{65}Ni (2.5 h) to determine the lattice location of Ni in *p*-Si, p^+-Si, n^+-Si, Ge, 3C-SiC, 6H-SiC, diamond, ZnO, GaN, *si*-GaAs, and n^+-GaAs. While the preliminary results show similarities to the previously investigated transition metals Fe, Mn and Co, also clear differences were observed, e.g. in Si the emission channelling effects disappeared already around 600-700 °C, most likely due to long-range diffusion of the Ni probes, which therefore seems to occur, similarly to Mn, at lower temperatures than for Fe or Co. Also Ni in Si showed a greater tendency to occupy bond-centered sites in Si, most likely related to more prominent reactions with double vacancies.

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IS487 – Local distortions in multiferroic AgCrO2 triangular spin lattice (IS487)

A.M.L. Lopes[1], G.N.P. Oliveira[1], T.M. Mendonça[2], J.A. Moreira[2], A. Almeida[2], J.P. Araújo[2], V. S. Amaral[3], J.G. Correia.

The study of the electric-field gradients and magnetic hyperfine fields of the multiferroic $AgCrO_2$ triangular spin lattice is presented (*A.M.L. Lopes et al. PRB84, 014434, 2011).* Perturbed angular correlation measurements performed with $^{111}In/^{111}Cd$ (at the Ag site) at different temperatures, revealed the coexistence of two electric-field gradients, i.e., two distinct local environments (EFGu) and (EFGd) at temperatures below 100 K. The emerging second local environment (d) appears as a distortion of the Cr surroundings resulting in a local symmetry lowering, which emerges much above T_N and concomitantly with the onset of short-range magnetic correlations. The data was correlated with complementary magnetization and complex dielectric permittivity measurements. We give evidence that at $T \approx 100$ K and, concomitantly with the onset of short-range magnetic correlations, a local distortion of the Cr surrounding emerges. We show that when lowering the temperature below 100 K, and still above TN, part of the system loses the local rhomboedral symmetry. Hence, this effect is associated to the coupling of elastic and magnetic degrees of freedom that provide a channel for magnetic frustration release through a lattice distortion. Though we cannot ascertain the detailed nature of the new structure, we argue that the distortions in the $AgCrO_2$ system are not a simple deformation of the equilateral Cr lattice into an in-plane isosceles configuration. Complementary measurements and EFG simulations are still necessary to unveil the details of the distortion aiming the full understanding of these exquisite systems.

[1] CFNUL, Lisbon University, Portugal;
[2] Instituto de Nanociência e Nanotecnologia, Porto University, Portugal;
[3] Department of Physics and CICECO, Aveiro University, Portugal.

IS481 – Cd doping of $Al_xGa_{1-x}N$ alloys via ion implantation studied with perturbed angular correlation

K. Lorenz, S.M. C. Miranda, J.G. Correia, P. Kessler[1], R. Simon[1], R. Vianden[1], K. Johnston[2]

$Al_xGa_{1-x}N$ ternary compound semiconductors, with wide bandgaps ranging from 3.4 eV (GaN) to 6.2 eV (AlN), are promising candidates for ultraviolet light-emitting diodes and laser diodes. However, the production of the required p-type material is still challenging in particular for alloys with high AlN content. As a possible dopant Cd was suggested among other Group II atoms (Be, Mg, and Zn). In this study the incorporation of implanted Cd in $Al_xGa_{1-x}N$ was investigated with the method of the perturbed angular correlation (PAC). Radioactive ^{117}Cd or ^{111m}Cd ions were implanted into thin $Al_xGa_{1-x}N$ films (with $0 \leq x \leq 1$) on sapphire substrate with an energy of 30 keV and fluencies in the range of 10^{11} ions/cm^2. After thermal annealing most of the Cd-probes occupy substitutional lattice sites and almost all implantation damage can be annealed. This results in a distinct frequency in the PAC spectra, which increases almost linearly when the AlN content in the layers increases. The frequency distribution is slightly increased for the ternary compounds compared to the binary alloys due to alloy disorder. In contrast to the formation of an indium nitrogen-vacancy complex observed with the probe ^{111}In on substitutional cation-sites no defects are bound to substitutional Cd impurities.

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LOI132 – Radioactive local probing and doping on graphene

V.S. Amaral[1], T. Trindade[1], J.G. Correia, S-W Hong[2], D. Pribat[2], C. Tenreiro[2], Y. Kadi[3], A. Gottberg[3], K. Johnston[3]

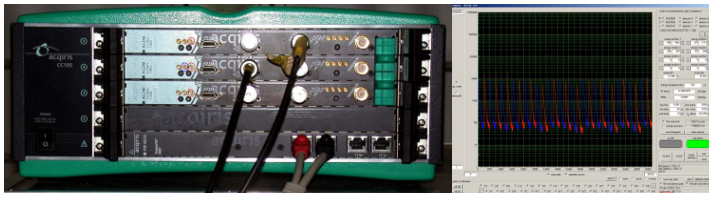
Perturbed Angular Correlations is used to study graphene and graphene-derived structures, to locally investigate properties associated with electron density and interactions, by using a selection of pure isotopes, dopant elements and adatoms at the graphene surface. In 2011 we did specific trial experiments on graphene and pyrolytic graphite standing on water as the carrier support media for the radio-isotope. The aim was to compare different data obtained from different C samples and methods to organize and optimize procedures and to develop dedicated handling equipment. Experiments using the UHV-setup at ISOLDE, ASPIC, looking forward to investigate graphene in ultra clean environments, impurity free, are foreseen for 2012-2013.

[1] CICECO, Dep. of Physics and Chemistry, Aveiro University, Portugal;
[2] Sungkyunkwan University, Seoul, Korea;
[3] ISOLDE-CERN, Geneva, Switzerland.

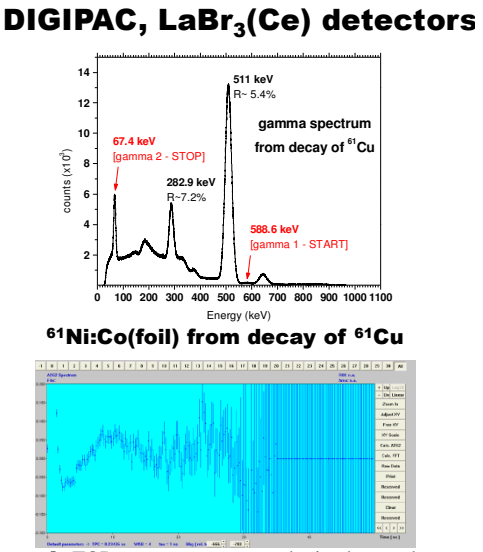
R&D - DIGIPAC and the ^{61}Cu/^{61}Ni New PAC probe isotope

J.G. Correia, M. B. Barbosa, K. Lorenz, T. Butz [1] and M.R. Silva[2]

In Spring 2011 our Laboratory - ITN @ ISOLDE received a donation from the University of Leipzig of three 6-D PAC spectrometers. Two of the traditional type equipped with BaF_2 detectors and one new digital machine (DIGIPAC) equipped with six $LaBr_3$ detectors. The excellent energy resolution of the detectors and the easiness of the data processing and human interface of this machine made possible the first ever done PAC spectrum onto the decay of ^{61}Cu/^{61}Ni. So far only the magnetic interaction has been measured for ^{61}Ni on a Ni foil. We expect having beam by 2012 to measure the quadrupole interaction of ^{61}Ni on materials.



Legend: LEFT DIGIPAC main core, consisting of 3 dual cards integrating each one, two digitizer Aquiris and two FPGAs for digitalizing data and processing
RIGHT data acquisition of 30 time spectra / combinations for each pair of detectors in coincidences γ_1 - γ_2, produced during the ^{61}Cu/^{61}Ni PAC experiment.



Legend: TOP gamma spectrum obtained onto decay of ^{61}Cu ($T_{1/2}$ = 3.3h) with $LaBr_3$ detectors.
BOT - R(t) experimental function where is clearly seen the larmor frequency due to the interaction of the 5/2 spin of the 67.4 keV state on ^{61}Ni with the Ni-host magnetic field.

[1] Nuclear Solid State Physics, University of Leipzig, Germany
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R&D data analysis - TDFIT- Dynamic Interactions in Perturbed Angular Correlations

M.B. Barbosa, J.G. Correia, K. Lorenz, M.R. Silva[1]

Hamiltonians for dynamic quadrupole and magnetic, hyperfine interactions have been implemented on a numeric generator of the perturbed angular correlation (PAC) observable. It rules the change of the population of the m states of the intermediate spin state of the PAC cascade, as due by time dependent operators (transient fields) on the so called "Stochastic Hamiltonean formalism". Systems to study are many, from dynamic correlation's on multiferroic compounds, to the so called “after-effects” in semiconductors and insulators. At the time of this report there are on-going experiments that combine e-γ and γ-γ PAC onto decay of ^{181}Hf/^{181}Ta, implanted and annealed on well characterized samples of (Si) n-GaN, (Mg) p-GaN, (Zn) p-GaN and AlN. The use of e-γ PAC allows to create deep level excitations and ionized states on these materials, which can be further studied as a function of temperature, learning about *point-like* electron dynamics on these materials.

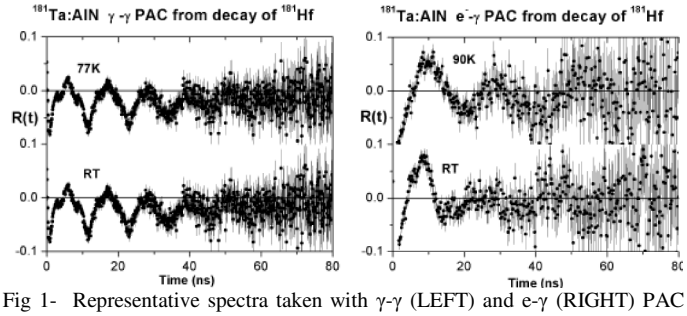

Fig 1- Representative spectra taken with γ-γ (LEFT) and e-γ (RIGHT) PAC where are clearly seen different EFGs and attenuations due to the slow electronic recombination after conversion electron emission. No such effect is observable with γ-γ PAC.

[1] CFNUL, Lisbon University, Portugal.

Nuclear Reactions

Adelaide Pedro de Jesus

This group has been involved in the experimental study of nuclear reactions relevant to nuclear astrophysics and also to ion beam analytical techniques.

The on-going work is related to the development of the AMS line to study reactions relevant to nuclear astrophysics.

Also a new line has been installed in the new Tandem 3MV accelerator, for nuclear reaction studies both for fundamental nuclear physics and nuclear astrophysics and for applied PIGE work.

Research Team

Researchers

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(*) Also members of CFNUL.

Development of a Reference Database for Particle-Induced Gamma ray Emission (PIGE) Spectroscopy – Concerted Project IAEA

F. Lourenço, M. Fonseca, J. Cruz, D. Galaviz, P. Teubig , J. Machado, A.I. Henrique, P. Velho, P. Reis, , H. Luis, J.P. Ribeiro, A.P. Jesus

Objectives

The aim of this work is to contribute to the improvement of data to PIGE analysis (bulk analysis and profiling) by assessment of available data, measurement of new data, evaluation of compiled and measured data, validation of cross section data through benchmark experiments, improvement of our ERYA code.

Results

Following the previous work to establish a methodology for standard-free PIGE analysis [1, 2], the group has joined a Concerted Research Project of the International Atomic Energy Agency, with the above described goals. During this year we have focused in the improvement of ^{23}Na(p,p´γ)^{23}Na reaction data, trying to resolve the discrepancies found in comparison with Caciolli et al [3, 4]. An experiment was run to obtain cross sections of the reactions on ^{23}Na, (p,p´γ): γ – 440 keV and (p,p´γ)+(p,αγ): γ – 1636 and 1634 keV. Fig. 1 show results obtained for ^{23}Na(p,p´γ): γ – 440 keV reaction cross-sections, showing that the mentioned discrepancy with the published Caciolli et al results still persists. This work was presented as a Master Thesis to New University of Lisbon.

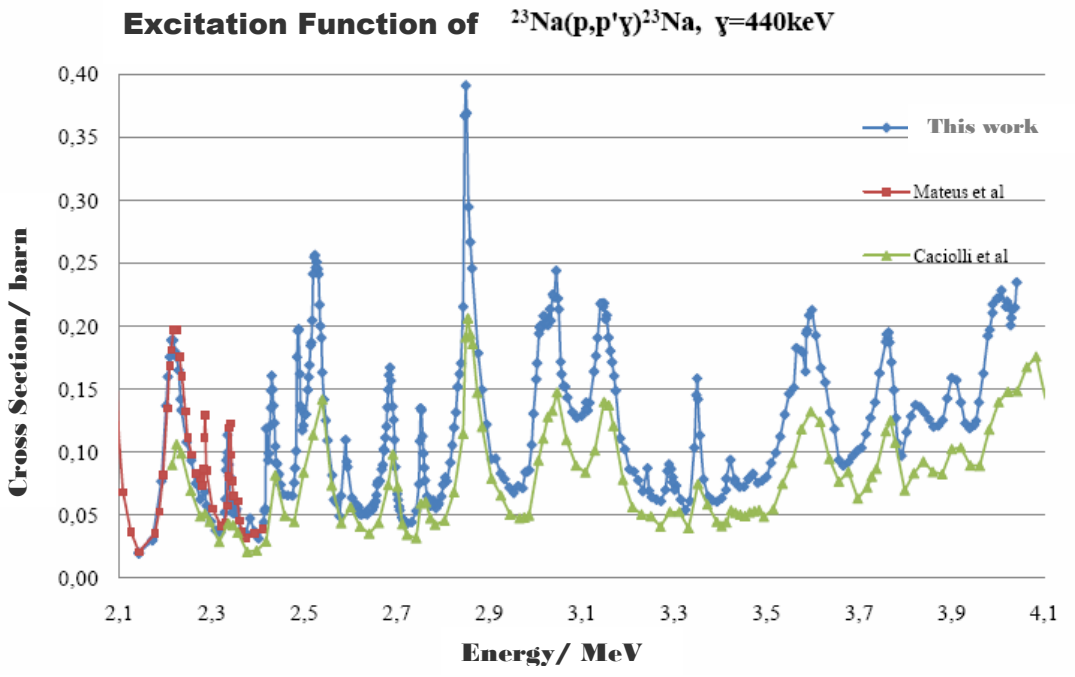

Fig. 1 Cross sections of the ^{23}Na(p,p´γ): γ – 440 keV reaction, compared with previous work.

Future work

This project will continue till 2014 covering the study of other gamma-producing reactions on F, Li, P, including also the development of depth analysis within the ERYA code.

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[2] M. Fonseca, H. Luís, N. Franco, M. A. Reis, P. C. Chaves, A. Taborda, J. Cruz, D. Galaviz, N. Fernandes, P. Vieira, J.P. Ribeiro, A.P. Jesus; Nucl. Inst. and Meth. B 269, 3060-3062 (2011)
[3] R. Mateus, A.P. Jesus, J. Cruz, J.P. Ribeiro, Nucl. Inst. and Meth. B 219 – 220, 307 – 311 (2004)
[4] Caciolli A, Calzolai G, Chiari M, Climent-Font A, Fernández-Jiménez MT, García-López G, Lucarelli F, Nava S. Nucl. Inst. and Meth. B266,1392 – 1396 (2008)

Study of nuclear reactions producing ^{36}Cl by AMS.

H. Luis, J. Cruz, N. Franco, M. Fonseca, D. Galaviz, A.P. Jesus, E. Alves

^{36}Cl is one of several short to medium lived isotopes (as compared to the earth age) whose abundances at the earlier solar system may help to clarify its formation process. There are two generally accepted possible models for the production of this radionuclide: it originated from the ejecta of a nearby supernova (where ^{36}Cl was most probably produced in the s-process by neutron irradiation of ^{35}Cl) and/or it was produced by in-situ irradiation of nebular dust by energetic particles (mostly, p, α, ^{3}He -X-wind irradiation model). In order to contribute to clarify this matter, we started the development of the AMS technique for ^{36}Cl detection and quantification, to measure reactions producing ^{36}Cl.

Samples of pure AgCl were irradiated at the Portuguese National Reactor, choosing irradiation times and sample masses in order to obtain ^{36}Cl to ^{35}Cl ratios in the order of 10^{-9} to 10^{-6} and ^{110}Ag activities under 1 MBq. This procedure had two purposes: measure the cross section of neutron capture by ^{35}Cl normalizing it to the well known cross section of neutron capture by ^{109}Ag, and to obtain ^{36}Cl standards for the AMS measurement of X-wind relevant reactions, testing also the linearity of the measurement process.

In order to obtain the referred neutron capture cross section the gamma-ray activity of ^{110}Ag was measured (Fig.1). The ^{36}Cl to ^{35}Cl ratio was obtained (Fig. 2), employing a micro-AMS system at the ITN's Ion Beam Laboratory.

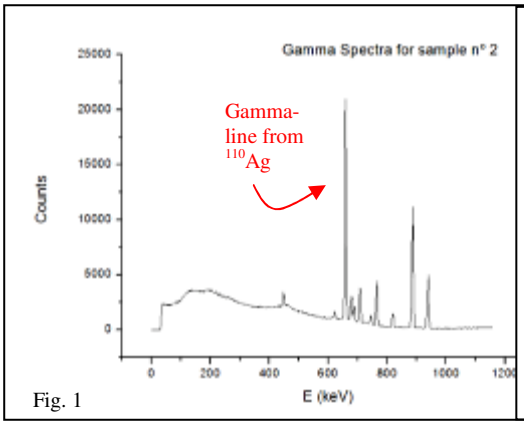

Fig. 1

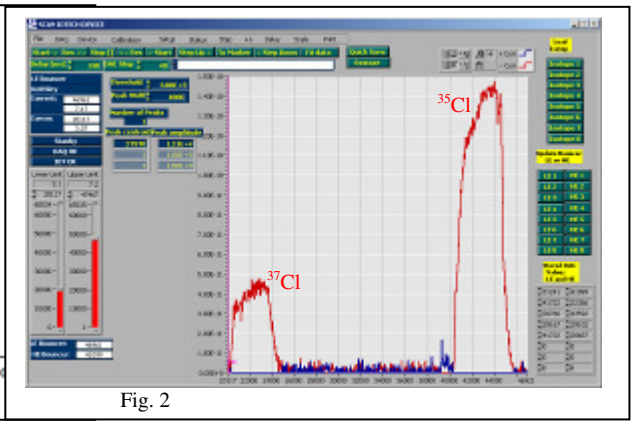

Fig. 2

Determination of (p,n) reaction cross sections of astrophysical interest

P. Teubig , J. Machado, A.I. Henrique, P. Velho, P. Reis, D. Galaviz, M. Zarza, H. Luís, M. Fonseca, F. Lourenço, J. Cruz, A.P. Jesus

Motivated by the work of G. Kiss et al. [1], we aim at measuring (p,n) reactions cross sections in medium-heavy mass nuclei, at energies of astrophysical interest, for channels with negative Q-value, will allow the characterization of the nuclear potentials involved in the process in conditions similar to those of the astrophysical environment.

In particular, we want to take advantage of the PIGE setup in order to measure, via the activation technique, (p,n) reactions for which the resulting product has a short half live (lower than 30 minutes), providing an experimental challenge for this kind of measurements.

The first nucleus we investigated at ITN was ^{128}Te through the reaction ^{128}Te(p,n)^{128}I. This reaction has a Q-value of -2.04 MeV, and the resulting nucleus decays with a half-life of 24.5 minutes. Taking advantage of the PIGE setup, the decay of the ^{128}I was observed with the available HPGe detector. In order to be able to discriminate the prompt background from the measurement, the data was recorded on an event-by-event basis.

At present only one data set, J.P. Blaser et al. [2], contains information close to the astrophysical energy range (between 2 MeV and 4 MeV). In order to proof the principle of the technique, we have performed a measurement covering the energy range between 3.8 MeV and 4.2 MeV. The data is still under analysis, although the signature of the decay of ^{128}I, a photon at 743 keV, was clearly observed. This work was performed in collaboration with the group at the University Complutense of Madrid (Spain), namely L. M. Fraile, J. M. Udías, J. Cal, B. Olaizola, E. Herranz

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[2] J. P. Blaser et al., Helv. Phys. Acta 24, 441 (1951)

Condensed Matter Physics

Fernanda Margaça

The Group's main field of research has been the development and characterisation of materials with new or improved properties. These are prepared and/or modified using mainly gamma irradiation from the ^{60}Co source of UTR.

The studied systems involve the development of new polymeric materials, catalytically actives, and hybrid materials prepared from mixtures of a polymer and different metallic alkoxides.

The preparation and characterisation of the studied materials has been performed in our Laboratory of Macromolecular Materials. The nanoscale structure has been investigated by neutron scattering in collaboration with colleagues at the Laboratoire Léon Brillouin (CEA-CNRS-Saclay) in France, Paul Scherrer Institute, Villigen in Switerzand and Budapest Neutron Centre of KFKI, in Budapest, Hungary.

The Group has also strengthened and widened its scientific collaborations with national research groups during the year of 2011. Namely, important work was carried out in collaboration with the Nuclear Reactions Group (ITN-UFA/ FCT-UNL), that involved the production of an $Ag^{36}Cl$ standard for the AMS technique. Furthermore, thermal analysis has been performed in collaboration with groups from UCRS/ITN, from the Dep. of Ceramic and Glass Engineering of University of Aveiro and from Universidade Nova de Lisboa (FCT-UNL).

During 2011 progress continued in the research and development of hybrid materials, in collaboration with Aveiro University under the project *Hybrid materials for biomedical applications*, with funding from the Foundation for Science and Technology, FCT, contract PTDC/CTM/101115/2008. New applications for these materials are currently being investigated in collaboration with Universidade Nova de Lisboa.

Progress has also been made in 2011 in the project *Preparation of polymeric materials catalytically actives on biodiesel production by vegetable oils methanolysis,* under the FCT contract PTDC/CTM-POL/114579/2009. Work on this project is carried out in collaboration with Universidade Nova de Lisboa.

The *Macromolecular Materials Laboratory*, *M3L*, after being moved in 2010 into a suitable room in the ground floor, in the extension of the UFA main building, was further upgraded during 2011. In fact, it now offers a range of improved possibilities for chemical and physical manipulations of samples as well as for their characterization and simulation.

In 2011 the following equipment was installed in the Macromolecular Materials Laboratory: a *workstation* to perform numerical simulations of the prepared materials, whose software is being installed; a *Universal Testing Machine* for mechanical properties of materials that is now being tested; an *hydraulic press* with temperature control that is ready to operate. Furthermore, the installation of a Positron Annihilation Lifetime Spectrometer (PALS) is underway, which is a sensitive non-destructive tool for studying pores and free-volume holes in materials.

This significant upgrading of the laboratory is a major breakthrough for our group. Now we are reaching the suitable working conditions that will allow us *i)* to extend the characterisation of materials to mechanical properties beyond the thermal analysis and in a short period also to measure the porosity; *ii)* to simulate the molecular structure and dynamics of materials, *iii)* to receive and supervise MSc and Ph.D. students, on a regular basis, to perform experimental as well as simulation work, for their thesis and *iv)* to apply to a wider variety of agencies for funding to carry out research and development in the area of macromolecular materials. Finally, such a laboratory will make us focus on this type of materials and therefore it is the right time to change the name of the group into a more realistic designation as Group of Macromolecular Materials, GM3.

Research Team

Researchers

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A.N. FALCÃO, Princ. (10%)
L.M.M. FERREIRA, Aux.
C.M.M. CRUZ, Aux. (10%)

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ITN BI grant

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J.P. LEAL, Aux.

Study of PDMS conformation in Hybrid Materials prepared from PDMS-TEOS-PrZr precursors by gamma irradiation

J.J.H. Lancastre, N. Fernandes, F.M.A. Margaça, I.M. Miranda Salvado[1], L.M. Ferreira, A.N. Falcão, M.H. Casimiro[2]

Objectives

Investigation of PDMS conformation in hybrid materials of the system PDMS-TEOS-PrZr prepared by gamma irradiation.

Results

All ATR/FT-IR spectra from hybrid materials show bands similar to those found in the spectrum of the irradiated PDMS. Slight differences (marked by the arrows in Fig. 1) were found in the Zr containing samples where an increase in intensity of the base line is observed with increasing Zr content.

This increase in intensity is compatible with the presence of Zr-silicate regions whose FT-IR spectra show broad bands in 600-1300 cm^{-1} (Rayner Jr et al., Microelectron. Eng. 72, 304, 2004). In fact, combining that spectrum with that of irradiated 33PDMS-66TEOS-1PrZr it is possible to reproduce the experimental data by simply applying a scaling factor to the reported Zr-silicate spectrum. This suggests that, at least above certain Zr content, regions of inorganic Zr-silicate oxide are formed.

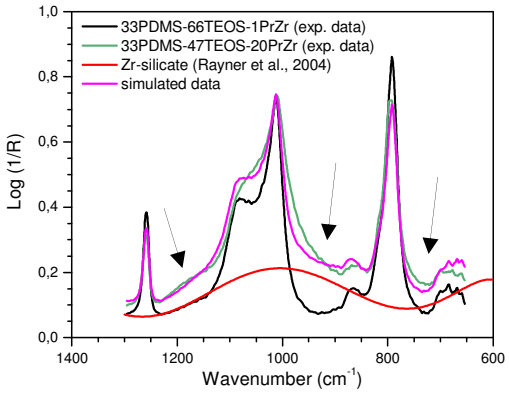


Fig.1 - FT-IR spectrum simulated (magenta line) combining the experimental data from Zr-silicate (red line) with that of irradiated 33PDMS-66TEOS-1PrZr (black line), normalized to the 1012 cm^{-1} peak. The arrows mark the differences existing in the 500-1500 cm^{-1} region.

Fig. 2 shows XRD diffractograms representative of the 33PDMS-(67-x)TEOS-xPrZr system. All diffractograms show two peaks: the fist (2θ ~ 11º) is due to the presence of regions of ordered polymer chains, and the second broader one, (2θ ~ 21º), is associated to the distance between Si atoms in the PDMS backbone.

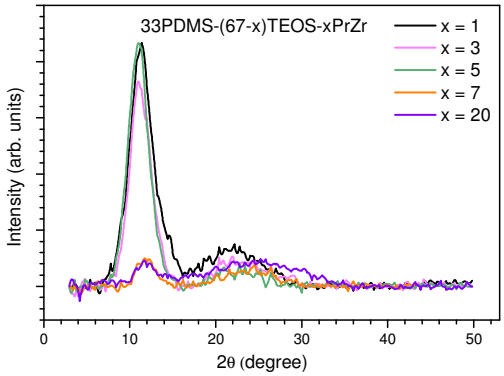


Fig.2 - XRD diffractograms of 33PDMS-(67-x)TEOS-xPrZr (x = 1, 3 5, 7 and 20 wt%) hybrid materials.

The distance between folded chains (*d*), the total peak intensity and the crystallite size (*L*) calculated from XRD results, are shown in Fig. 3 as a function of the PrZr content.

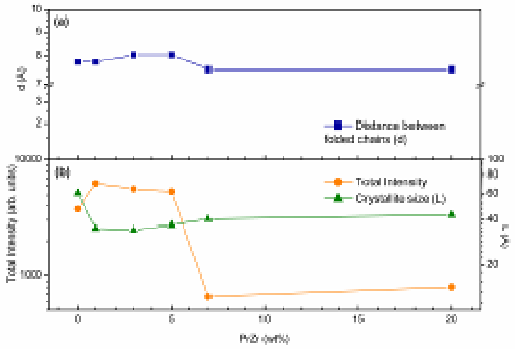


Fig .3- Variation with PrZr content on a semi-log scale of: **(a)** distance between folded PDMS chains (blue); and **(b)** peak total intensity (orange) and crystallite size (green).

The crystallite size is smaller in samples containing Zr than in irradiated PDMS. The distance between folded chains, shows no significant change with increasing Zr. The total peak intensity, associated to the PDMS crystallites, increases with Zr for PrZr ≤ 5 wt% whereas for larger contents it almost disappears. This suggests that Zr, for smaller contents, might be uniformly dispersed in the material probably linked to end positions of the polymer chains. Such a link explains the formation of smaller crystallites than in the reference sample. For larger Zr contents, the saturation of available chain ends for Zr bonding, can lead the excess Zr to bond to PDMS backbone and/or to precipitate preferably on the ordered regions, inhibiting the chain ordering.

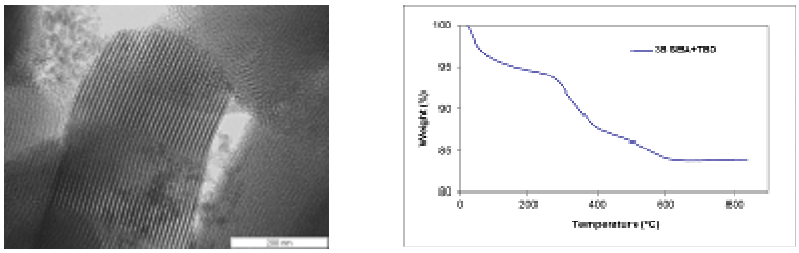
Further work will involve mechanical analysis and bioactivity tests in these materials.

[1] CICECO & Dep. Eng.ª Cerâmica e do Vidro, Universidade de Aveiro, Portugal
[2] REQUIMTE/CQFB, Dep. Química, FCT, Univ. Nova de Lisboa, Portugal

Preparation of polymeric materials catalytically actives on biodiesel production by vegetable oils methanolysis

M.H. Casimiro[1], A.G. Silva[1], A.M. Ramos[1], J.V. Vital[1], L.M. Ferreira

In collaboration with researchers from REQUIMTE-DQ/FCTUNL we have synthesized and characterized solid acid/basic catalysts and acid/basic catalytic composite matrixes: The catalytic materials consist of mesoporous silica MCM-41 and SBA-15 bearing different functional groups. The catalytic composites are made by the organic/inorganic solid base loaded in a polymer matrix. Transesterification catalytic tests in classical batch reactor showed materials with different catalytic activity and stability.



TEM TGA

Fig 1- TEM image and thermogram of solid basic catalyst (mesoporous silica SBA-15 loaded with TBD (1,5,7-triazabicyclo [4,4,0] dec-5-ene)).

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Characterization of PE-*g*-HEMA films prepared by gamma irradiation through nuclear microprobe techniques

L.M. Ferreira, J.P. Leal, P.A. Rodrigues, L.C. Alves, A.N. Falcão, M.H. Gil[1]

PE-*g*-HEMA films with different grafting yields prepared by mutual gamma irradiation method at the ITN ^{60}Co facility have been characterized with ion beam analytical techniques (RBS, STIM and PIXE) using the nuclear microprobe from UFA - Ion Beam Laboratory.

Qualitative analysis showed a random and heterogeneous distribution of contaminant elements, independent of the grafting degree, suggesting the existence of several sources of contamination at different stages of their preparation. Results also suggest that this "phased" contamination occurs simultaneously with mechanisms of agglomeration/entrapment of impurities during the gamma induced copolymerization reaction. Moreover, quantitative data showed that all contaminants found in the copolymeric films are natural contaminants of their reagents of preparation, although at concentrations without toxicological hazard, which points to a low cytotoxic potential.

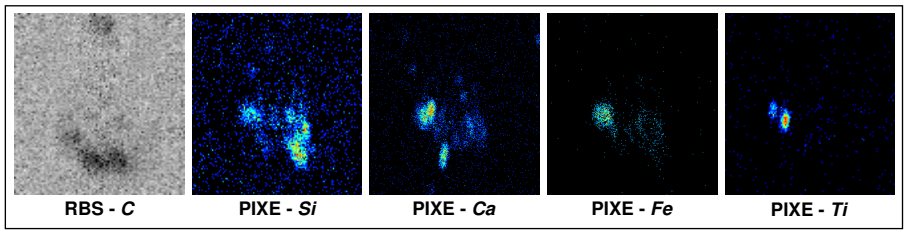

Fig 1- Maps of RBS (Carbon) and PIXE of impurities found in the PE-*g*-HEMA film grafted at 166% (35 hours of irradiation; D_{abs}= 10.5 kGy). Maps dimensions: 100×100 μm^2.

The use of these techniques for materials contamination characterization has worked as a quality benchmark of the experimental development of this materials and, simultaneously, reducing the level of failure of biological tests required by the international standard ISO 10993 - *Biological Evaluation of Medical* Devices, for materials biomedical applications, which are very expensive and time-consuming. The simultaneous use of RBS, STIM and PIXE allow us to know, at a time, "who, where and how" are contaminating our samples.

[1]

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Synthesis and characterization of novel γ-induced porous PHEMA-IL composites

M.H. Casimiro[1], M.C. Corvo[1], E.J. Cabrita[1], A.M. Ramos[1], L.M. Ferreira, F.M.A. Margaça

The synthesis and characterization of novel γ-induced porous PHEMA-Ionic Liquid composites is still being investigating. The resulting composites can be converted into an organogel by addition of DMSO, and converted back to a porous composite by immersion in water. Work in progress involves the use of different ionic liquids and evaluation of PHEMA-IL conductivity.

Fig 1- $BMIPF_6$ (1-butyl 3-methylimidazolium hexafluorophosphate) and HEMA (1:1) before and after gamma irradiation.

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Laboratory of Macromolecular Materials – Lab II (*Physical Characterization*)

L.M. Ferreira, F.M.A. Margaça, N. Fernandes, J. Lancastre, C. Cruz, N. Catarino, M.H. Casimiro[1]

The Laboratory of Macromolecular Materials gained a second room that is devoted to the physical characterization of polymeric and hybrid materials. In this new room, a Universal Mechanical Testing Machine (Zwick 1435) was installed and subsequently automated. It is fully functional to study mechanical properties including flexibility, tensile and compression tests.

Fig 1- Laboratory views showing the main equipment installed during 2011.

As a complement of this equipment it was also installed an hydraulic press (12 ton) with thermal mordents (temperature controlled by programmable water and compressed air mixer). This equipment allows the preparation of polymeric specimens for mechanical testing as well to carry out thermal and compression stress routines in materials.

Fig 2- Laboratory views showing details of the equipment installed in 2011.

Furthermore, a PALS – *Positron Annihilation Lifetime Spectrometer* is in its final stage of installation. Its nuclear instrumentation as well as the software are already assembled, installed and tested. Only the gamma detectors are lacking. These should be acquired and installed next year. This facility will extend our ability to study the material porous dimensions and respective distribution as well the distance between the long chains in polymers and hybrid materials.

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Radiation Technologies: Processes and Products

M. Luísa Botelho

Radiation Technologies: Processes and Products is an interdisciplinary group that uses the holistic approach as the key to conceptualize a research or a service. This interdisciplinarity, using Biology, Chemistry and Physics science, allows the study of a subject from various angles and methods unified by a common goal: the validation of methodologies to understand the subject of study, which core is ionising radiation.

The group *modus operandi* permits a constant connection with Industries, Universities and other Research groups applying its expertise in response to requested services, as a collaborator in a research project or in the transmission of knowledge.

The group activities focus on the delineation, development, validation and application of technologies and processes in various fields, such as Environment, Food and Pharmaceutics. As a fundamental part of the validation studies, Risk Analysis is being applied as a process management tool either in production lines of studied products (*e.g.*: food, devices and pharmaceuticals) or in environmental control (*e.g.* hospital rooms, pharmaceutical industries and buildings energetic certification).

In the scope of ITN mission the group is solicited by the authorities or private industries to undertake a consultant role on sterilization and decontamination procedures mainly applying ionising radiation. The group also develops work with the National and International normalization, standardization and certification bodies (IPQ, CEN and ISO).

The Group's main R&D activities are focused at employing ionising radiation technologies to new processes and applications in Agriculture, Food, Pharmaceutical, Wastewater Treatment, Art and other areas. In order to improve our understanding of the Radiation effects in products integrated methodologies composed by Analytical Methods of Biology, Microbiology, Chemistry and Physics are being used. Molecular Biology new trends based on PCR technique are being developed as a diagnostic tool (*e.g.:* potential pathogenic micro-organisms) and as well as fingerprinting methods to assess the biodiversity profile of environmental samples. On-going R&D in environmental virology is being conducted, namely the inactivation response of enteric viruses (*e.g.* norovirus and adenovirus) to ionising radiation for treatment applications (*e.g.* wastewater, food).

FCT running projects, Pulse Radiolysis (PTDC/QUI-QUI/104229/2008) and RADIART (PTDC/HIS-HEC/101756/2008), are focusing in deepen the ionising radiation effects on liquid/solid matrices. Food Irradiation is also a current group research area. Either in the scope of a "chestnut irradiation" collaboration project (CHESTNUTSRAD - QREN nº 13198/2010) with School of Agriculture of IPB; and as part of an IAEA Coordinate Research Project (CRP D6-RC-1163.2) for development of irradiated food for immune-compromised patients.

Training and "know-how" diffusion are one of the main issues of this Group reflecting in the attainment of academic degrees (Graduation, M.Sc. and Ph.D.) and in the dissemination of obtained results in the scientific community (publications, workshops and conferences).

The financial support of the group is based on projects, sponsored by National (*e.g.* FCT, AdI) and International (*e.g.* IAEA) science foundations and expertise services to Industrial Companies.

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Kinetic study of biorecalcitrant compounds degradation by pulse radiolysis

P.M.P. Santos, A. A. Amílcar[1], R. Melo, S. Cabo Verde, T. Silva, H. Marcos, I. Nunes, J. Madureira, M. Bação, J.P. Leal and M. L. Botelho

Objectives

To use pulse radiolysis technique to study the mechanistic details of the degradation of biorecalcitrant compounds present in industrial (*e.g.* phenolic acids) and municipal discharges (*e.g.* pharmaceuticals), induced by free radicals formed upon radiolysis of aerated water (hydroxyl radical, hydrogen atom, superoxide anion radical and hydrated electron).

Results

Since 2001 the Radiation Technologies: Processes and Products Group has been doing studies on the impact of ionising radiation in the wastewater looking for the advantages and/or disadvantages of the technology application in different kinds of wastewater. These studies covered several fields (*e.g.* chemistry, microbiology and physics). Currently we intended to study the degradation reaction mechanisms of model compounds and predict its radiolysis by-products.

The upgrading of the linear accelerator (LINAC) installed in ITN during 2008 (under FCT Project REEQ/996/BIO/2005) is being performed to work in a pulsed technique. Meanwhile, stationary radiolysis studies with model compounds (*e.g.* gallic acid (GA)) were performed.

Therefore, these integrated studies will be rather useful to assess ionising radiation as a complementary wastewater treatment technology. Concerning the work in progress, several studies have been performed in order to predict a degradation reaction mechanism of GA. This compound is a well known recalcitrant compound present in cork wastewater. Thus, its radiolytic degradation could reduce the high toxicity of these waters.

Standard GA solutions were irradiated in the Co-60 experimental source (Precisa 22) at 1, 9 and 38 kGy at 2 kGy/h. The UV-Vis spectra of non-irradiated and irradiated GA solutions (1 mM) present two absorbance maxima at 269 nm and 212 nm (see Fig. 1).

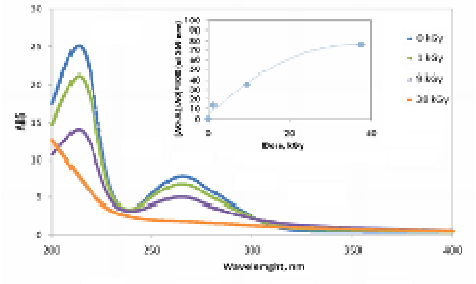

Fig. 1 - UV-Vis spectra of the non-irradiated and irradiated 1 mM GA. Inset: calculated degradation of GA (considering the 269 nm peak) versus irradiation dose.

[1] School of Agriculture, Politechnical Institute of Bragança, IPB, Portugal.

Increasing the radiation dose the absorbance of the peaks overall maximum decrease, which indicate that irradiation effectively degrade GA. The amount of the degradation can be estimated by defining a degradation factor ($(A_0\text{-}A)/A_0\times100$, where A_0 and A are absorbencies of GA solutions at 269 nm prior to and after irradiation, respectively). This factor increase, not linearly, with the dose reaching approximately 76% at 38 kGy (see inset in Fig. 1). However, even at 9 kGy GA degradation reaches almost 35%.

Previous pulse radiolysis studies made in the Department of Chemistry of the Institute of Isotopes (Budapest, Hungary) showed that the hydroxyl radical and hydrogen atom intermediates of water radiolysis react with the solute molecules yielding cyclohexadienyl radicals and generated phenoxyl radicals by loss of water. The GA intermediates formed during reaction with primary water radicals and in the presence of oxygen are transformed to non-aromatic molecules, e.g., to aliphatic carboxylic acids. The Electrospray Ionization Mass Spectrometry (ESI-MS) was used to identify the final by-products of the GA degradation. Based on these results a general reaction sequence for the degradation of GA by gamma radiation is proposed (Fig.2)

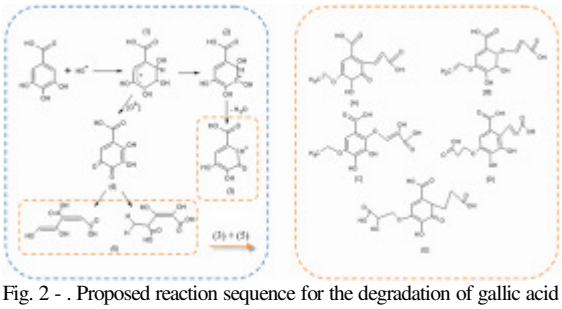

Fig. 2 - . Proposed reaction sequence for the degradation of gallic acid by gamma radiation and some of the possible radiolytic products.

The proposed radiolytic-formed compounds are multi-carboxylic acids and still have the benzenic ring. This proposal is consistent with the fact that the decreasing peak at 212 nm remains present (see Fig. 1) since the smaller radiolytic-formed compounds still have some hydroxyl groups connected to the ring, whereas the one at 269 nm almost disappear (most of the carboxylic groups are broken away from the benzenic ring).

On-going studies focus on the radiolytic degradation mechanisms of other model compounds (*e.g.* acetovanillone and esculetin) by pulse radiolysis.

Published work

Melo, RP; Leal, JP; Botelho, ML. (2011) "Radiolytic degradation mechanism of gallic acid and its end-products" Rapid Communications in mass spectrometry Vol.25 Issue: 1 Pages: 218-222 DOI: 10.1002/rcm.4803.

Application of ionising radiation on the cork wastewater treatment: antioxidant capacity evaluation
J. Madureira, R. Melo, M. L. Botelho

The effects of ionising radiation in cork boiling water are being studied to assess the potential increase of its antioxidant capacity due to hydroxyl radicals' ($OH^{\bullet}$) reactions. Chemical Oxygen Demand (COD) and Total Organic Carbon (TOC) were measured to analyze the gamma radiation effects in terms of organic matter content. Total phenolic and antioxidant capacity by Ferric reducing antioxidant power (FRAP) assay were analyzed to predict the effects of this technology in the antioxidant properties. The samples were irradiated at a Co-60 source at several doses (2, 10, 20, 50 kGy) at two different dose rates (0.4 $kGy.h^{-1}$ and 2.4 $kGy.h^{-1}$). The concentration of oxidable organic matter (expressed in terms of COD) decreases, at both dose rates, with increase of absorbed doses. The TOC content do not decrease due to the breaking of organic molecules in other organic molecules of low molecular weight. At 2.4 $kGy.h^{-1}$ the increase of phenolic compounds is higher (29% to 50 kGy) than at 0.4 $kGy.h^{-1}$ (6% to 50 kGy).). This phenomenon could be explained due to that lower dose rate could not be able to destroy the benzenic ring of the tannins and other phenolics, and only break the bonds and rearrange structures, increasing the concentration of smaller phenolic structures. Concerning the FRAP assay, results show an increase of antioxidant activity with irradiation which could be related with the increase of total phenol concentration connected with $OH^{\bullet}$ radical addition. These results associated to the well-known gamma radiation disinfection effect could be an important issue to the development of advanced oxidation processes as a complementary technology on wastewater treatment processes helping its reuse. These studies are under the scope of the IAEA Coordinate Research Project CRP 1539 "Radiation Treatment of Wastewater for Reuse with Particular Focus on Wastewaters Containing Organic Pollutant".

Food Irradiation: raspberries irradiation
S. Cabo Verde, I. Nunes, P. Santos, A. António[1], T. Silva M. J. Trigo[2] M.L. Botelho

Under the scope of the IAEA Coordinate Research Project CRP D6-RC-1163.2 "Development of Irradiated Foods for Immuno-compromised Patients and Other Potential Target Groups" it was intended to evaluate the irradiation effects on fruit and vegetables and the potential extension of shelf-life, in order to improve the safety and variety of immune-compromised patients diet. The nutritional value of raspberry fruit is widely recognized and is demanded by consumers, especially for protection against several diseases, as well as for general health benefits. Based on that, fresh packed raspberries (*Rubus idaeus*) were irradiated at a Co-60 source at several doses (0.5; 1 and 1.5 kGy) at a dose rate of 2.2 kGy/h with a uniformity of dose of 1.23. Microbiological, physico-chemical and sensorial parameters were assessed after irradiation and during storage time. The characterization of raspberries microbiota point out to an average bioburden value of 10^2 cfu/g and to a diverse microbial population predominantly composed by two morphological types [gram-negative, oxidase-negative rods (34%) and filamentous fungi (41%)]. The inactivation studies on the raspberries mesophilic population indicated a one log reduction of microbial load (95% inactivation efficiency for 1.5 kGy). However after irradiation, the surviving population was mainly constituted by filamentous fungi (79 – 98%), being morphologically identified potential pathogenic/opportunistic fungi such as, *Fusarium* sp. and *Alternaria* sp.. Regarding raspberries physico-chemical properties, irradiation caused a decrease in firmness compared with non-irradiated fruit. Nevertheless, non-irradiated and irradiated fruit presented similar physico-chemical and sensory properties during storage time. Based on the microbiological results, the potential application of raspberries in immune-compromised patient's diet could be questionable.

[1] School of Agriculture, Politechnical Institute of Bragança, Portugal;[2] National Institute for Agricultural Research and Fisheries, Portugal.

Irradiation as a potential conservation treatment for art objects
I. Nunes, T. Silva, S. Cabo Verde, M. L. Botelho

Art biodeterioration is one concerning issue that leads to the necessity of developing new approaches in restoration, preservation, conservation and decontamination areas. Studies were performed in order to evaluate the potentiality of gamma rays as a conservation treatment for cultural assets. Namely, the determination of a minimum dose to attain the decontamination and a maximum dose that preserve the studied art objects. Irradiation of ceramic tiles and parchment samples were performed in the Co-60 experimental source (Precisa 22) at a dose rate of 2.2 kGy/h. Sub-lethal doses (1 – 15 kGy) were applied to verify microbial population inactivation profiles on tiles and parchment samples. For tiles, it was observed a significant microbial population decrease of approximately 25% ($P < 0.05$) for irradiation doses higher than 2 kGy. The results also indicated the prevalence of the filamentous fungi (86%) in the surviving population. For parchment samples it was verified microbial inactivation efficiencies higher than 90% for doses > 4 kGy. The higher dose survivors in parchment belong mainly to the groups of Spore forming gram-positive rods and Filamentous fungi. Concerning irradiation effects on tiles physical appearance it was visible an increase in the ceramic opacity and a darkness of blank pigment with radiation dose. However, a maximum gamma radiation dose of 30 kGy suggested no significant modifications in parchment texture and colour characteristics, based on the evaluated parameters and their conditions. Results indicated the potentiality of gamma radiation in a range of 5-10 kGy as effective for parchment documents decontamination treatment.
These studies are under the scope of the projects RADIART (1 BIC) and MYCHOARCHIVE (1 Ph.D.; 1 McS).

Nuclear Instruments and Methods

João B. Manteigas

The strategy of the group involves activities in the following lines:

1. Modelling of radiation fields, calculation of neutron physic parameters, measurement of neutron cross-sections;
2. Modelling and applications of gas discharges;
3. Development of software for control and data analysis;
4. Design of electronic instrumentation for nuclear applications;
5. Instrumentation and technical assistance;
6. Co-operation with other institutions.

Modelling of radiation fields, calculation of neutron physic parameters

Monte Carlo calculations have been carried in the framework of the n_TOF Collaboration (ITN participation on the n_TOF-Ph2 experiment at CERN).

Measurement of neutron cross-sections

The analysis of the data for cross-section measurement, taken in the TOF spectrometer installed at the CERN, was carried out.

Modelling and application of gas discharges

1. The study of methane conversion by a non-thermal plasma produced by a dielectric barrier discharge system (DBD) to obtain Syngas and other hydrocarbons has continued with (a) study of the influence on rectangular voltage pulses on conversion and selectivity, (b) the study of the electron kinetics in methane/carbon dioxide/helium mixtures and, (c) development of theoretical models to explain the variation of i) the breakdown voltage with rare gas concentration and, ii) the conversion fractions with the specific input energy.
2. The study of a dielectric barrier discharge system for processing of polymers by a non-thermal plasma for industrial applications.
3. The construction of a RF-plasma needle with application in biology and medicine.

Development of software for control and data analysis in nuclear spectrometry

The development of free software has continued with the support of EPICS-based gamma spectrometry equipment on the *PyMCA* software for X-ray analysis.

Instrumentation and technical assistance

1. The main objectives are the development of equipment for ITN groups, fabrication of equipment for specific applications and assistance to industrial companies and scientific institutions as well as technical consulting.
2. The technical assistance takes mainly the forms of specialised consultant engineering advice, installation of nuclear gauges, including calibration maintenance and repair and recharging of gauges with imported radioactive sources.
3. The group started providing maintenance and repairing services of HPGe detectors as well as technical advice in the installation of gamma spectrometry equipment.

Co-operation with other institutions

1. Plasma Physics Centre / Gas Electronics Group, IST;
2. ISEL, Dept. of Automation and Electrotechnical Engineering;
3. Comenius Univ., Dept. of Experimental Physics, Bratislava, Slovakia;
4. Leibniz Institute for Plasma Science and Technology, Greifswald, Germany;
5. Research Institute for Solid State Physics and Optics, Budapest, Hungary;
6. n_TOF collaboration, a consortium of several laboratories in Europe, USA and Japan.

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R.P.F. MENDES, Collaborator (NP, 15%)
A. JANECO, Ph.D. Student (NP)
S. BARROS, Ph.D. Student (IG)

Technical Personnel

T. JESUS
N. INÁCIO
M. CABAÇA

Collaborators

GABRIEL SILVA (10%)

Technical Assistance in the Field of Engineering Applications of Radiation and Radioisotopes

J. Manteigas, J. Neves, N. Pinhão

Objectives

The main objectives are the development of equipment for internal groups, fabrication of equipment for specific applications and assistance to industrial companies and scientific institutions as well as technical consulting.

Results

A summary of the more relevant work carried out is:

(i) Collaboration in corrective and preventive maintenance of the "Ion Beam Laboratory" – TANDEM 3 MV" at the Physics Unit.

(ii) Optimization of the electronic device "Photo-multiplier Divider" for the BaF_2 calorimeter under the project n_TOF-Ph2 experiment at CERN.

(iii) Services in nuclear spectrometry;

(iv) Development and maintenance of electronic equipment to UFA, UPSR, URSN, UCQR and UTR.

Summary of the more relevant Services/Equipment rendered in 2011

Activity	Qty	Client
Electronic Equipment Laboratory equipment for the determination of radioactive element traces by electrodeposition	4	NATS (Qatar)
	1	Dr. Henry Ben (Polónia)
Electronic Equipment Technical Assistance to Nuclear Equipment	21	EMA21 – Portucel/Soporcel (Cacia/Portugal)
	20	EMA21 – Portucel/Soporcel (Cacia/Portugal)
	6	SIDERURGIA NACIONAL (Seixal/Portugal)
	1	ENDRESS+HAUSER (Portugal)
	8	ITN (URSN, UCQR, UPSR) (Portugal)
Prices including TAX (VAT)		**Total Amount: 20 747,66 €**

Participation of ITN in the n_TOF-Ph2 experiment at CERN (5th year)

I. F. Gonçalves, P. Vaz, C. Cruz, J. Neves, C. Carrapiço, R. Sarmento, S. Barros

This project is the continuation of the involvement of ITN in the activities of the n_TOF Collaboration. Since February 2011 the ITN continued the analysis of the data recorded from 2004 till 2010, in collaboration with INFN-Bari and CEA-Saclay, as well as the feasibility studies associated to the construction of the second experimental area. The ITN team participated in several data taken shifts at CERN and is strongly involved in collaboration with Bari and Saclay, in the following areas: Monte Carlo simulation - full and detailed simulation of the geometry of the new experimental area with the usage of Monte Carlo codes MCNPX and GEANT-4; data analysis of the data on neutron capture on U-233 and the analysis of the resonances data on neutron-induced fission on U-236 and Am-241, Am-243 e Cm-245, taken during 2004 using the FIC ("Fast Ionization Chambers") detectors; data analysis of the Fe isotopes (of relevance for innovative technological systems and for Nuclear Astrophysics).

Conversion of methane by a non-thermal plasma using rare-gas/CH_4/O_2 and rare-gas/CH_4/CO_2 mixtures on a dielectric barrier discharge system

J. Branco, N. R. Pinhão, A. Janeco, A. Ferreira, L. Redondo

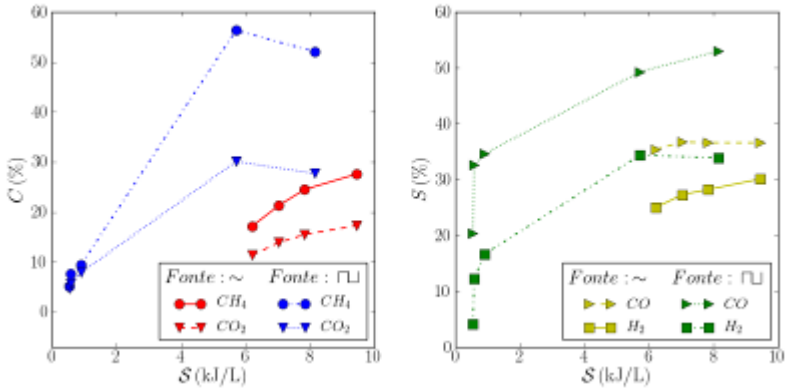

Fig 1- Dependency of conversion, C, for CH_4 and CO_2 and selectivity, S, for CO and H_2 with the specific energy with the voltage pulse shape (sinusoidal or rectangular).

The direct conversion of methane into *Syngas* and other hydrocarbons by a non-thermal plasma is an interesting alternative to the established production process. The study of the conversion of CH_4/CO_2 mixtures in a non-thermal plasma has continued with (i) the study of the influence of the voltage pulse shape of a DBD (dielectric barrier discharge) on conversion and selectivity; (ii) the study of the electron kinetics and, (iii) the development of models for the discharge breakdown. The theoretical model developed is able to explain the results obtained for the breakdown voltage.

Development of software for control and data analysis in nuclear spectrometry

R.P.F. Mendes, N.R. Pinhão

The development of Free Software for gamma and X-ray spectrometry has continued with the extension of the *PyMCA* software (for analysis of X-ray spectra) to support gamma spectrometry and online acquisition from remote equipment based on the EPICS libraries for distributed control of scientific equipment.

***Development of atmospheric non-thermal plasma sources for applications in material and biological* sciences**

N.R. Pinhão, J. Neves

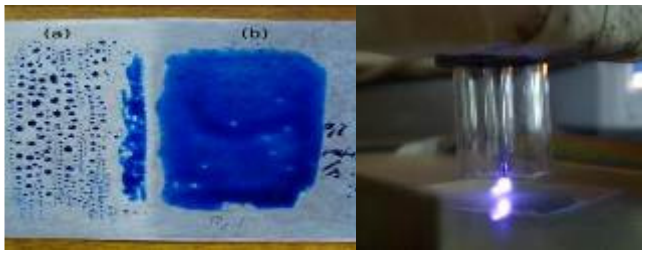

Fig 1 - A - Results on the wettability of a polyvinyl acetate: (a) untreated, (b) treated with a corona discharge. B – Plasma needle discharge at 3 W.

The treatment of surfaces at atmospheric pressure with non-thermal plasmas is an increasingly important field.

The development of two different plasma sources has started: a corona/dielectric barrier discharge system for processing of polymers and a RF-frequency plasma needle for biological and medical applications.