Advanced Materials Research

Eduardo Alves

The Advanced Materials Research Group (GIMA) is responsible for running the Ion Beams Laboratory (IBL). The laboratory is equipped with a 2.5 MV Van de Graaff accelerator, an ion microprobe presently being fitted with an external beam facility, a recently installed 3 MV tandem accelerator with an Accelerator Mass Spectrometry (AMS) line, and a 210 kV high fluence Ion Implanter.

The work carried out during the last two decades allowed the group to achieve a large expertise in the field of applications of ion beams to Materials Science. The group activities are focused on the processing and characterisation of advanced materials using ion beam based techniques. Several national and international collaborations allow a continuous exchange of expertise and mobility of researchers, an important condition to keep the scientific activity in the group at the forefront of research in its field.

The current research activities of the group were focused in two kinds of materials: wide band gap semiconductors, and nanostructures and insulators. Wide bandgap semiconductors are under intense research all over the world due to the possibility of developing optoelectronic devices working in the visible wavelength range of the electromagnetic spectrum. Our work aims at the optimization of the implantation conditions of optically active dopants. Other relevant research work is being carried out in quantum well structures and quantum dots. An intense study of the structural properties of GaN/InGaN structures is under way in collaboration with the Universities of Aveiro and Strathclyde. Structural and optical studies of Ge and GaN quantum dots are a new field of research in collaboration with the Universities of Aveiro and Grenoble.

The work in insulators is a continuation of ongoing projects or bilateral collaborations. Of these particular interest resides in the modification of the optical and electrical properties of α-Al_2O_3 and laser materials (KTP and RTP), and also of the magnetic and electrical properties of MgO and rutile doped with ions of magnetic transition metals by high fluence implantation. Besides this and due to the potential of ion beam techniques to study thin films and multilayers, important collaboration work continued in the characterisation of magnetic thin films and layers for magnetic spin valves, diluted semiconductors and tunnel junctions, as well as in the field of protective oxynitride coatings.

The activity in the technology programme of the European Fusion Development Agreement (EFDA), in association with the Centro de Fusão Nuclear of the Instituto Superior Técnico continued with studies on the oxidation behaviour of beryllides, and on the characterization of the new Eurofer (ODS) steel.

Integrated in these research activities the group has also been strongly involved in training graduate and undergraduate students, through the supervision of M.Sc. and Ph.D. thesis.

All these activities were financially supported by a large number of projects, both European and National (FCT), either in collaboration with other Institutions or lead by members of the group.

The scientific activity of the group in 2006 was materialized in:

Publications (peer review journals): 91
Conference and workshop contributions: 9 invited talks, 13 oral and 34 posters.
Running projects: 12

Researchers(*)

E. ALVES, Principal researcher (Coordinator)
R.C. DA SILVA, Principal researcher
L.C. ALVES, Auxiliary researcher (75%)
U. WAHL, Principal researcher
N. BARRADAS, Principal researcher (10%)
A.R. RAMOS, Auxiliary researcher (10%)
A. KLING, Auxiliary Researcher (10%)
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C.P. MARQUES, Ph.D. student, FCT grant
E. RITA, Ph.D. student, FCT grant
J. VAZ Pinto, Ph.D. student, FCT grant
M. VILARIGUES, Ph. D. student***
N. FRANCO, Ph.D. student, FCT grant**
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(**) High Temperature Materials Lab.
(***) Technician, Dep. Cons. & Rest., UNL

Structural analysis of nitride quantum structures

K. Lorenz, N. Franco, S. Magalhães, N. Barradas, E. Alves, I.M. Watson[1], R.W. Martin[1], K.P. O'Donnell[1], B. Amstatt[2], B. Daudin[2]

An issue of fundamental importance in semiconductor physics continues to be the measurement of strains and crystal quality within quantum structures. These include strained-layer structures incorporating wide-gap group III nitride semiconductors which have an impressive variety of realized and proposed applications in (opto-) electronic devices, such as lasers, LEDs, transistors, sensors etc.

Rutherford backscattering/channelling spectrometry (RBS/C) and X-ray diffraction (XRD) were used to study the composition and structural properties of AlInN/GaN bilayers and AlN/GaN superlattice structures.

The ternary AlInN is attracting much research interest due to the possibility of growing AlInN lattice-matched on GaN for InN concentrations around 17-18 %, promising low misfit dislocation densities. Furthermore, AlInN alloys are candidates for opto-electronic devices covering an extremely wide spectral region from deep UV to IR since the band gap can range from 6.2 eV (AlN) to 0.7 eV (InN).

Near-lattice-matched $Al_{1-x}In_xN$ layers were grown by metal organic chemical vapour deposition (MOCVD) on micrometer thick GaN buffer layers on sapphire at different growth temperatures between 760 and 840 ºC. As expected, with increasing growth temperature the amount of InN introduced into the layer decreases and the crystalline quality is improved.

Discrepancies between composition determination by XRD and RBS indicate a deviation of this alloy from Vegard's law as it was also predicted by density functional theory calculations. A tentative correction to Vegard's law reconciles the XRD and RBS/C measurements.

Steering effects in the interface influence the angular yield from the GaN-layer in RBS/C and cause failure of the conventional means of assessing strain by RBS/C. We have shown for the first time that Monte Carlo simulations are viable as a routine tool to correct channeling results for such steering effects, leading to an excellent agreement of RBS/C and XRD strain measurements. The in plane strain in high crystalline quality AlInN-on-GaN films was found to change from tensile to compressive as the InN fraction increases from 13 to 19 %. Lattice matching is predicted to occur at 17.1(9) %.

Self-assembled GaN quantum dots in AlN multilayers consisting of 150 periods were investigated. Using RBS in grazing incidence geometry the first eight bilayers can be clearly separated, and it is possible to draw information on layer thickness, composition and quantum dot properties.

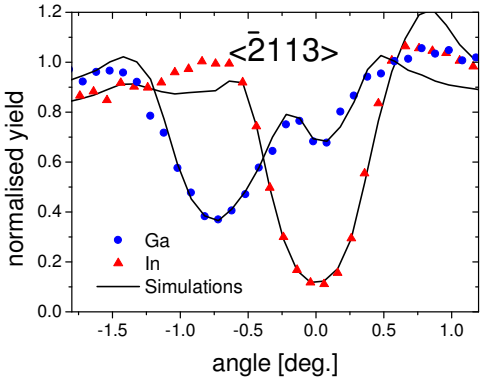

Fig. 1: Ga and In RBS/C angular scans across the $<\bar{2}113>$ axis of a AlInN/GaN bilayer with 13 % InN content. The second dip in the Ga-scan with a minimum at the same position as the In-scan is due to steering effects in the interface. The effect is well reproduced by Monte Carlo simulations, which allow the correct determination of strain in the layer.

Angular scans across the <0001> axis show a low minimum yield of 8 % indicating a perfect stacking of the layers along the growth direction. Further investigation will focus on the determination of strain in the multi-layers and aim to explain the influence of strain in the process of vertical self assembly of the GaN quantum dots.

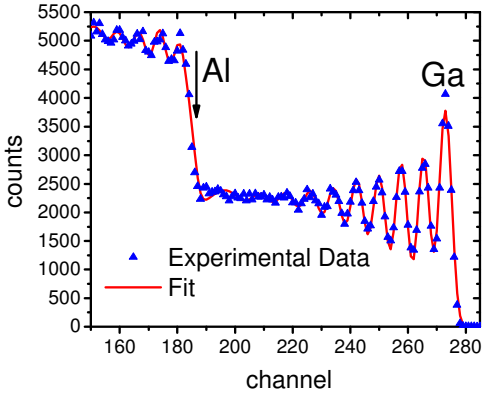

Fig. 2: RBS spectra and fit of a GaN-QD/AlN superlattice taken with the sample tilted by 86º in order to improve the depth resolution. The NDF simulation program was modified in order to describe cylindrical quantum dots.

[1] University of Strathclyde, Glasgow, U.K.
[2]CEA/CNRS, Grenoble, France

Rare earth doping of III-nitride alloys by ion implantation

K. Lorenz, E. Alves, T. Monteiro[1], A. Cruz[1], M. Peres[1], A.J. Neves[1], M.J. Soares[1], I.S. Roqan[2], K. Wang[2], R.W. Martin[2], C. Trager-Cowan[2], K.P. O'Donnell[2]

Rare earth (RE) doped nitrides are widely studied with respect to potential applications in electroluminescent devices. While GaN is the most studied nitride host for RE implantation, AlN and the ternary alloys $Al_xGa_{1-x}N$ are also promising host candidates. Their wider band gap allows exploitation of higher lying RE levels and a lower thermal quenching of the luminescence is expected. Furthermore, those Al containing alloys are more resistant to implantation damage than GaN.

$Al_xGa_{1-x}N$ ($0 \le x \le 1$) epilayers grown by HVPE or MOCVD were implanted with Eu and Tm. Rutherford backscattering/channelling (RBS/C) was used to study structural properties and the lattice site location of the implanted ions. Optical studies were performed using photoluminescence (PL) and cathodoluminescence (CL) spectroscopy. $Al_xGa_{1-x}N$ with $x \ge 0.08$ was shown to be more resistant to implantation damage than GaN and no surface amorphisation occurs. Furthermore, the samples are stable at annealing temperatures well above 1000 ºC while GaN at these temperatures requires a special protection of the surface, for example by a thin AlN cap layer. The RE are found slightly displaced from the substitutional cation sites with stronger displacements for higher Al concentrations. The near substitutional fraction and PL characteristics of Eu implanted AlN are influenced by the annealing ambient and temperature.

The PL intensity at room temperature of Eu implanted $Al_xGa_{1-x}N$ increases strongly when the Al content is increased from 0 to 30% and drops steeply when the Al content is further increased.

The possibility to exploit higher lying RE levels in AlGaN as compared to GaN was shown for the blue lines of Tm.

[1] Dep. de Física, Universidade de Aveiro, Aveiro, Portugal
[2] Department of Physics, SUPA, University of Strathclyde, Glasgow, G4 0NG, U.K

Optical and structural study of Ge/Si quantum dots on Si(100) surface covered with a silicon oxide layer

A. Fonseca, E. Alves, J.P. Leitão[1], N.A. Sobolev[1], M.C. Carmo[1], A.I. Nikiforov[2]

Si/Ge low-dimensional structures attract the attention of the scientific community due to their potential to develop new electronic and optoelectronic devices. Among the different ways to produce Ge islands on Si substrates, a technique was recently developed that is based on the Wollmer-Weber growth mode, which relies on the growth of the Ge islands on top of a SiO_2 interlayer. Through this mode, we may obtain smaller Ge islands with extremely high density.

The formation of Ge quantum dots (QDs) grown on an ultrathin interlayer of SiO_2 on top of a Si(001) substrate was investigated, as a function of the thicknesses of the SiO_2 interlayer (0.5, 0.75 or 1 monolayer) and of the Ge layer (0.3, 0.6 or 0.9 nm). The structural characterization was performed by Rutherford backscattering spectroscopy (RBS). Photoluminescence (PL) was used to characterize the optical behaviour of all samples. Hydrogen treatment was performed in order to passivate non-radiative recombination channels, thus enhancing the PL intensity. The results suggest the formation of Ge nanoislands for the sample with 1 ML of SiO_2 and 0.9 nm of Ge, and exclude their formation for samples with lower SiO_2 and Ge layer thicknesses. We also observe an influence of the SiO_2 interlayer thickness in the QDs formation.

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Implantation of nanoporous GaN with Eu ions

S. Magalhães, K. Lorenz, E. Alves, M. Peres[1], T.Monteiro[1], S. Tripathy[2]

Nanoporous GaN samples were implanted with 150 keV Eu^+ ions with a fluence of 5×10^{15} cm^{-2}. Channeling and x-ray diffraction (XRD) measurements indicate a high crystalline quality. After implantation the optical activity of the GaN is quenched by the implantation damage. The <0001> aligned spectrum reveal broad damage distribution over the entire porous layer responsible for an expansion along the c-axis with a parameter increase of 0.3% as indicated by XRD. Photoluminescence due to above band gap excitation at low temperature indicates the presence of two regions with different optical properties after annealing. We observe a transparent zone with UV and yellow-green broad emissions and a pale brown region where the UV emission is absent. In both regions the intraionic 4f ^{6} Eu^{3+} emissions are present. XRD and channeling confirm the good crystalline quality of the samples after the annealing and the incorporation of Eu into near substitutional Ga sites.

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[2] Institute of Materials Research and Engineering, 117602, Singapore

Effect of annealing and ion implantation on AlN/GaN quantum dot heterostructures

S. Magalhães, K. Lorenz, E. Alves, B. Amstatt[1], B. Daudin[1]

The incorporation of quantum dots in III-nitride heterostructures aims at exploiting novel optical and electronic properties due to quantum confinement effects. The development of devices may include processing steps like high temperature annealing or ion implantation which can alter the structural and optical properties of the heterostructures. Ion implantation presents an alternative to incorporate dopants into heterostructures but the implantation damage can deteriorate the structural and optical properties of the samples.
In this work multilayers consisting of up to six GaN quantum dot layers separated by AlN buffer layers grown by molecular beam epitaxy were studied. Eu ions were implanted into the heterostructures with 120 keV to a fluence of 1×10^{15} cm^{-2} (see figure). Structural properties and the effects of thermal and ion beam processing were analysed using X-ray diffraction (XRD), X-ray reflection (XRR) and Rutherford backscattering and channelling spectrometry (RBS/C). High resolution RBS with grazing beam incidence, of as grown samples enabled the individual GaN-dot layers to be resolved and XRD reveals the existence of a second peak corresponding to a region in AlN which is deformed by the strain caused by the GaN quantum dots. Annealing performed in nitrogen atmosphere for 20 min at temperatures between 1000 ºC and 1200 ºC produced slight changes of the multilayer period with high temperature annealing as shown by XRR. For 1200 ºC annealing the broadening of the XRD profile indicates the deterioration of the crystal mainly due to surface degradation. The implantation damage in the AlN/GaN quantum dot heterostructure will be compared and discussed with respect to the results found in thick GaN and AlN layers.

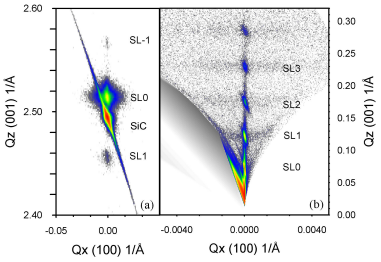


Fig.: (a) (002) Reciprocal Space Map (RSM) of the as grown sample. The SiC and superlattice peaks are indicated as SiC, SL0, SL-1 and SL1, respectively. (b) corresponding XRR RSM.

[1] CEA/CNRS GROUP, "nanophysique et semiconducteurs", Dépt. de recherche fondamentale sur la matière condensée

Study of InGaN/GaN single quantum wells grown by metal-organic chemical vapour deposition

E. Alves, N.P. Barradas, S. Pereira[1], A. Kholkin[1], I.M. Watson[2]

Strong interest has recently developed in the transfer of excitations from InGaN quantum wells into other luminescent media. Previous work specifically relevant to the current investigation involves non-radiative energy transfer from near-surface InGaN quantum wells to either colloidal semiconductor quantum dots or light emitting polymers. In this context, the accurate measurement of the fundamental structural parameters in SQW and MQWs such as the well compositions and thickness, and in particular cap layer thicknesses are fundamental from a technological and scientific point of view. However, accurately measuring these parameters in ultra-thin (~2 nm), buried epitaxial layers is extremely difficult, particularly for SQWs with low (<0.05%) InN mole fraction.
Here we report detailed structural characterization results of InGaN/GaN MQWs and SQWs obtained from grazing incidence Rutherford backscattering (RBS) analysis. Careful simulation of the RBS spectra could provide precise estimations of individual well/barrier compositions, thickness and the extent of In/Ga intermixing. Moreover, in the case of low InN content SQWs grown with different GaN cap thickessess (nominal values of 5, 7.5 and 15 nm) the cap layer thickness could be determined with a good precision.

[1] CICECO
[2] University of Strathclyde, Glasgow, U.K

Ni induced growth of crystalline silicon nanowires

A.R. Ramos, E. Alves, I. Ferreira[1], L. Pereira[1], P. Vilarinho[2], E. Fortunato[1], R. Martins[1]

The transition from crystalline silicon aggregates to silicon nanowires (SNWs) films produced by Low Pressure Chemical Vapour Deposition (LPCVD) of pure SiH4 gas on Ni covered corning glass substrates was studied. Scanning electron microscopy (SEM) images and X-ray diffraction patterns confirm that transition when the substrate temperature varies from 823K to 923K. Rutherford Backscattering Spectrometry (RBS) analyses quantify the film composition in depth and the progression of Ni buffer layer from substrate to top film surface. We have demonstrated the production of silicon crystalline films and nanowires by LPCVD technique at relative low temperature (923K) compatible with corning glass substrates workable temperature. We expect that by controlling the Ni buffer layer thickness we may obtain nanowires where Ni is either present at the top surface, or along the entire film to form NiSi silicides. The anisotropic SNWs growth achieved can be a disadvantage for certain applications, those that need straightforward and well aligned nanowires, but can also be an advantage for other applications such as the ones were light must be trapped.

[1] CENIMAT, Dep. de Ciência dos Materiais, Faculdade de Ciências e Tecnologia da Universidade Nova de Lisboa, and CEMOP-UNINOVA, Campus da Caparica, 2829-516, Caparica, Portugal
[2] CICECO, Departamento de Engenharia Cerâmica e do Vidro da Universidade de Aveiro, 3810 193 Aveiro, Portugal

Structural evolution in ZrN_xO_y thin films as a function of temperature

E. Alves, A.R. Ramos, N.P. Barradas, F. Vaz[1], L. Rebouta[1], U. Kreissig[2]

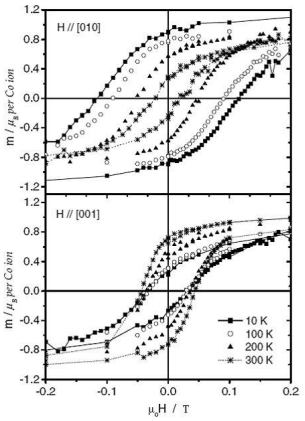
Single-layered zirconium oxynitride (ZrN_xO_y) thin films have been deposited on steel substrates at a constant temperature of 300 °C, by radiofrequency (rf) reactive magnetron sputtering of a pure Zr target in an argon-oxygen-nitrogen atmosphere. The variation of the flow rate of the reactive gases enabled changes in the composition and structure of the films. X-ray diffraction (XRD) and glancing incidence X-ray diffraction (GIXRD) were used to study the as-deposited films and their structural changes during or after heat treatment, from 400 °C to 900 °C, in controlled atmosphere and in vacuum. The as-deposited films revealed the occurrence of a face-centred cubic (fcc) phase (Zr-N type), but a Zr-N oxygen-doped phase (Zr-N-O) may be also present depending on the oxygen content in the films. Heat treatment above 600 °C reveals the appearance of a tetragonal phase of zirconium oxide. The results are discussed as a function of the chemical composition of the films, annealing temperature, and type of the annealing process.

[1] Universidade do Minho, Dept. de Física, Campus de Azurém, 4800-058 Guimarães, Portugal
[2] Forschungszentrum Rossendorf e.V., Postfach 510119, 01314 Dresden, Germany

Stuctural and magnetic properties of oxides implanted with transition metals

J.V. Pinto, M.M. Cruz[1], M. Godinho[1], E. Alves, R.C. da Silva

We extended previous studies on the behaviour of the transition ions Co, Ni and Fe in MgO to the case of TiO_2, a potential candidate for spintronics applications. The goal is to fully characterize the behaviour of the implanted ions. Magnetic, electrical and structural (RBS and XRD) characterization were used to study these systems.



For 10^{17} cm^{-2}, 150 keV Co-implanted single crystalline α-TiO_2 rutile, the as implanted state exhibits superpara-magnetic behaviour attributed to the formation of ~5 nm sized hcp cobalt clusters. Vacuum annealing at 1073 K induces anisotropic ferromagnetic behaviour, the easy axis direction lying in the (001) plane of rutile, due to the $(002)_{Co}$ and $(100)_{Co}$ // $(100)_{rutile}$ orientations of the cobalt clusters (see figure). Enhanced electrical conductivity exhibits also anisotropy at low temperatures, following the same general trend as in reduced rutile. No magnetoresistive effects were detected, indicating that there are no polarization effects of the charge carriers induced by the cobalt ions. Annealing in air promotes the segregation of cobalt to the surface of rutile, pointing to the role of oxygen vacancies in stabilizing the aggregates in reduced rutile. It is suggested that vacancies accommodate at the interface between the aggregates and the TiO_2 host. Results for Ni implanted α-TiO_2 rutile are similar. Studies with Fe have been initiated.

Fig.: Hysteresis loops taken at different temperatures in the hard and easy magnetization in-planar directions for vacuum annealed sample (reproduced from The European Physical Journal-B., 55 (2007) 253-260)

[1] Dep. de Física, Faculdade de Ciências da Universidade de Lisboa.

Friction and wear mechanisms in orthopaedic prostheses: influence of the composition of the periprosthetic fluid[1]

E. Alves, R.C. da Silva, B. Saramago[2]

Osteoarthritis and rheumatoid arthritis often require substitution of natural joints by prostheses, especially of hip and knee. The most common cause of failure and lack of durability of total hip prostheses is related with the generation of wear debris of UHMWPE from the acetabular part, when sliding against the ceramic or the metallic ball which substitutes the femoral head.

Studies of Cl-implanted TiN deposits intended as protective coatings of the metallic components of prostheses continued. The work at ITN concerned the implantation with high fluences of Cl-ions and characterisation of the coatings and of the chlorine distribution profiles in the as-implanted state and after tribological experiments. The Cl-implantations led to significant polymeric wear reduction when HBSS is the lubricant. With BSA added to HBSS, a strong decrease of both friction and polymeric wear is observed both for implanted and non-implanted TiN coatings, but still the Cl-implanted TiN coating led yields the best tribological results. Wear reduction is attributed to the substitution of the hard TiN counterface by a less hard titanium oxide layer which is a less wear-aggressive for the polymer surface, while the behaviour of the friction coefficient is explained by the slow precipitation of calcium phosphate.

[1] Project POCI/SAU-BMA/55493/2004
[2] Departamento de Química, Instituto Superior Técnico

Synthesis of ZnO nanoprecipitates on sapphire through ion implantation
C. Marques, N. Franco, R.C. da Silva, E. Alves

Following previous results obtained on sapphire implanted with Zn, the role of crystalline orientation of the substrate was studied, aiming at the production of a surface layer with ZnO precipitates. Samples of c- and m-cut sapphire were implanted with several fluences of Zn and O and annealed up to 1300 ^{o}C in air or vacuum. RBS, TEM and XRD results show that only m-cut samples implanted with Zn and annealed at 900 ^{o}C in vacuum produced polycrystalline ZnO nanoprecipitates embedded in the host matrix. At higher temperatures most Zn is lost through evaporation while annealing in air favours the production of mixed oxides. Annealing at 900 ^{o}C followed by annealing at higher temperatures is underway in order to increase the crystalline quality of the ZnO precipitates (bulk ZnO is stable up to 1800 ^{o}C).

AFM studies of transition metal implanted sapphire
C. Marques, R. Colaço[1], R.C. da Silva, E. Alves

Changes in surface topography of single crystalline sapphire (α-Al_2O_3) upon heavy ion irradiation were studied with atomic force microscopy (AFM). Prior to implantation the samples were annealed at 1000 ^{o}C in ambient atmosphere to recover from polishing damage. The implantation were carried out with nominal fluences up to 5×10^{17} cm^{-2}. Rutherford backscattering spectrometry was used to measure the implanted fluence and evaluate the extent of the implantation defects. The build-up of surface damage and the changes introduced in the topography of the samples were analyzed with AFM and correlated with the implanted fluence, energy and current density during implantation. The role played by the particular crystallographic orientation of the implanted sample and the nature of the transition metal ion used are also investigated. The development of nanostructures at the surface is observed for the highest implantation fluences, the signature of which is also recognized by the development of plasmon resonance effects seen through optical absorption measurements.

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Miscellaneous
C. Marques, E. Alves

Several studies were carried within collaboration work with other groups, namely:

- IFPAN (Poland): Fe doped GaN samples were analysed through PIXE and RBS under channelling conditions, to measure the iron content as well as the crystalline quality and to locate the Fe atoms on the GaN lattice.
- Wayne State University (USA): *i*) study of the composition of new lead-free solder, extremely important for device industry, was carried by RBS analyses; *ii*) research on vanadium oxides deposited by inorganic and organic sol-gel processes, onto sapphire, aiming at optimizing the deposition conditions to achieve epitaxial growth. Characterization performed by RBS and RBS-C; *iii*) doping of steel by ion implantation and subsequent compositional and structural studies by RBS and RBS-C measurements; *iv*) RBS studies of pure titanium oxide films and of transition metal doped (5% or 7%) titanium oxide films deposited onto crystalline Si or Al_2O_3 samples, aiming at determining its composition.
- FCT-UNL: RBS analyses of Al and Cu thin films deposited on Si or glass substrates, in order to measure the effective gradients for comparison with nominal values from the deposition method; RBS analysis of Al and Ti thin films deposited onto Si substrates in vacuum or in N_2 atmosphere in order to study the influence of the deposition atmosphere and to compare effective and nominal composition gradients.
- IST-UTL: Si-based detectors were irradiated with helium ions or protons and its photoelectric response measured *in situ*. The composition of the targets was also measured. ZnO films deposited with Au onto crystalline Si and Al_2O_3 were analysed with RBS and RBS-C to measure the composition, crystal quality and epitaxy of the films.

Installation of a 3 MV tandem accelerator with IBA and AMS at the LFI-ITN
GIMA *staff and collaborators*

After acquisition and delivery of the 3 MV tandem accelerator in mid 2006, assembly and installation of the equipment and beam lines followed. The accelerator, its HV power supply, vacuum and electrical subsystems were tested and brought to operational status. Operational tests of the H and He (alphatross) ion sources, conventional IBA beam lines and beam transport and steering systems were also tested and brought to operational status. An experimental chamber for PIXE analyses is already operational. The AMS beam line was assembled and the control software migrated to PC-based OS, debugged and upgraded to LabView v7. Work proceeds with the installation of specialised end-station equipment and components.

Characterization of potash-glass corrosion in aqueous solution

M. Vilarigues[1], R.C. da Silva

We continued to study the corrosion processes of potash-glass surfaces in contact with aqueous solutions, using Ion Beam Analysis techniques, Optical Microscopy and Fourier Transform Infra-Red (FTIR) spectroscopy.
Glass samples with base compositions of 56 mol.% SiO_2, 24 mol.% CaO and 20 mol.% K_2O, and added with CuO and MnO as colorants, were prepared.
Testing with and without stirring of the aqueous solutions was used as they lead to different surface morphologies. Silica rich-layers and Ca-carbonates are always found in the surfaces exposed to longer immersion tests. For the tests without stirring the surface reaction is slower and the carbonates structures have time to grow, while stirring accelerates the reaction leading to smaller average size and more uniform distribution of the surface structures. Evidence was found that glass dissolution progresses through a sequence of steps. When Cu, Mn and Fe are introduced in the glass matrix a layer richer in the transition metal ion added is formed in the glass surface. Cu-containing glass displays a faster initial dissolution that may be due to its particular oxidation state (2+). Since the findings indicate that both Cu and Mn are present in the glass matrix in octahedral coordination, while Fe is tetrahedrally coordinated, we suggest that the octahedral configuration eases the exchange between these ions and hydronium ions.

[1] Departamento de Conservação e Restauro, Universidade Nova de Lisboa.

Quality control and oxidation behaviour of Nuclear Fusion Reactor materials*

E. Alves, L.C. Alves, A. Paúl [1], N. Franco, M.R. da Silva [2], J. A. Odriozola [1]

Nuclear microprobe analytical techniques were used for the elemental characterisation of ODS steel samples. The main goal was to ascertain if the inhomogeneity of yttria distribution, that was found in previous analysis of other batches of ODS steels, could again be established. The obtained elemental maps show that, within the beam spatial resolution (~3×4 μm^2), all the elements are homogeneously distributed.
Aiming to a more detailed study of the oxidation behaviour of titanium beryllides, the previously analysed Be-5 at%Ti and Be-7 at%Ti samples supplied by JAERI were analysed before and after two oxidation stages obtained by annealing at 800 ºC in air atmosphere. Both oxidation steps were done during two hours each. The behaviour is similar for both samples, with the RBS spectra showing Be diffusion to the surface and subsequent Be oxidation, and no marked difference for the 2 h or 4 h annealing procedure. The surface formation of BeO was confirmed for the Be-5 at%Ti sample by XRD analysis.

* Euratom/IST association
[1] Instituto de Ciencia de Materiales de Sevilla, Spain; [2] Centro de Física Nuclear da Universidade.de Lisboa, Portugal.

Formation of Nanoclusters by Thermal Oxidation of SiGe and Deposition of Discontinuous SiGe Layers

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Nanoparticles of Ge, Si or SiGe embedded in a SiO_2 matrix are highly interesting for the formation of luminescent devices compatible with established Si-technology. It has been demonstrated previously that their fabrication by direct deposition of discontinuous SiGe layers sandwiched by SiO_2 using conventional low pressure chemical vapour deposition (LPCVD) is a very promising approach. In order to increase the luminescence yield the formation of multilayer structures containing the nanoparticles has been investigated. The dependence of the luminescence emission on structural parameters like the diameter of the nanoparticles and the oxide interlayer thickness, as well as the annealing conditions have been studied using grazing incidence RBS, TEM and cathodoluminescence. Structures with small nanoparticles (3-4.5 nm) separated by thick oxide barriers (≈ 35 nm) annealed at 900 ºC for 60 s yield the maximum intensity as a result of a compromise between the appropriate crystallization of the small nanoparticles and a reduced degradation of their composition by Ge diffusion due to the thick barriers.

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Measurement of proton elastic scattering cross sections for light elements – validation of a "bulk sample" method

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The present research project proposes to measure (p,p) elastic cross sections for nitrogen and lithium in the 500-2500 keV energy range and for scattering angles between 160º and 130º. The measurements will be used, together with existing evaluated cross sections for other light elements, to validate a new automated method of proton elastic cross section measurement. The method will be applied to the determination of cross sections using bulk samples. The research carried out will result in improved data analysis algorithms in existing simulation programs for IBA. The improved algorithm, which accurately calculates proton backscattering spectra in the presence of cross section resonances, constitutes a desirable benchmarking tool for evaluated/measured cross-sections using standard bulk samples.

Appropriate samples for cross section measurements were prepared or acquired and their composition determined by He-RBS. The proton elastic cross sections for nitrogen were determined using a thin aluminium nitride (AlN) film deposited over vitreous graphite. Measurements were performed in the 700-2400 keV energy range and for scattering angles of 160º, 150º and 130º. In addition, the 110º scattering angle was also measured. Cross section values were obtained from the areas of the Au and N peaks, using appropriate formulae. The values thus obtained were compared with the values produced by an automated algorithm. The algorithm application to bulk samples was tested using the same spectra, but concentrating on the C signal. The C cross sections obtained were compared with the evaluated cross-sections found in the literature.

Advanced data analysis for IBA

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Ion Beam Analysis (IBA) is a cluster of techniques dedicated to the analysis of materials. Our goal is, on the one hand, to improve the accuracy of the data analysis by developing advanced physical models and introducing them in computer codes available to the community, and on the othe hand to automate the data analysis. In 2006 the main development was the application of the joint RBS and PIXE analysis capabilities to real cases, revealing the power of such a combined approach. Also, a new model was developed to improve the calculation of RBS and ERDA spectra in the low energy region. Non-Gaussian energy resolution shape was introduced, which is important in the near-surface region in high resolution experiments. An accurate algorithm to calculate pile-up effects was developed. The model to calculate the influence of voids and inclusions was further developed, and the influence of local variations of density is now also included. The work on artificial networks was concluded. Eight papers were published in 2006 in international journals, including a review on IBA software.

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Development of external analytical microbeam at the ITN nuclear microprobe[1]

P.A. Rodrigues, L.C. Alves, R.C. da Silva

Started in the last quarter of 2005 the project aims at developing and installing an external microbeam analytical end-station at the nuclear microprobe facility and fit it with the ion beam techniques, PIXE and RBS, with the intent of applying it mainly to the field of patrimonial studies, particularly artwork and archaeology.

After design, assembly and vacuum testing different solutions a beam transfer end-section became ready to be used in the external beam analytical end-station. Fitted with 100 nm thick Si_3N_4 on Si-frame beam transfer window, beam alignment and focusing were tested. A 70×75 μm^2 fwhm beam was obtained in air 3 mm away from the beam transfer window. Reproducibility tests showed it is currently possible to obtain 80×80 μm^2 beams by using standard alignment and focusing procedures typical of vacuum microprobe, without special care. Tests proceeded with the collection of PIXE and RBS spectra of a Cu-grid and alumina control samples in air and in He atmosphere, 3 mm away from the beam exit window. Helium sprayed at 4-5 l/min improved the beam spread to 60×65 μm^2 fwhm, while allowing resolving a 50 mesh Cu-grid in a 530×530 μm^2 scan. A novel dedicated He injection cap is being designed that fits the RBS detector allowing more efficient spraying of the target irradiation spot. A laser positioning device is also being developed for precise positioning of the target area in relation to the microbeam.

[1] Project POCTI/CTM/60685/2004