Advanced Materials Research

Eduardo Alves

The Advanced Materials Research Group (GIMA) operates most of the experimental facilities at the Ion Beam Laboratory (IBL) and XRD Laboratory. 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 XRD laboratory is fitted with two X-ray diffractometers, the High-temperature Hotbird diffractometer, and one Bruker AXS - D8 Discover diffractometer.

The group explores and develops ion beam techniques, X-ray diffraction and reflectometry techniques to study advanced materials e.g. wide band gap semi-conductors nanostructures, oxides and other functional materials in collaboration with other groups. Among the wide band gap materials our major interests is focused on group nitrides and ZnO. These are the base of an emerging class of optoelectronic devices operating in the visible range of the electromagnetic spectrum. The potential of these materials for spintronics applications is being investigated with University of Aveiro and Faculty of Sciences of University of Lisboa. Our work aims at the optimization of the implantation conditions of magnetically and optically active dopants. In addition an intense research on the structural properties and Rare Earth doping of GaN/AlN quantum dots (QD) layers continued in collaboration with Universities of Aveiro, Grenoble and Strathclyde.

The work in insulators comprises the modification of the optical and structural properties of α-Al_2O_3 by ion implantation.

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, was focused on the study of beryllium intermetallic and the study of surface erosion and redeposition processes as well as ^2H retention during JET operation.

Training and Education continued as a major commitment of the group firmly linked to research activities through the supervision of M.Sc. and Ph.D. thesis. Also worth to be mentioned is the new researcher joining the group under the Ciência 2008 programme.

All the referred activities have been 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"*, EURATON 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).

These 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): 54
Conference and workshop contributions: 3 invited, 13 oral and 12 posters.
Running projects: 21

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Erosion and re-deposition processes in JET tiles studied with ion beams

L.C. Alves, E. Alves, N.P. Barradas, P.A. Carvalho[1], R. Mateus[1], J.P.Coad[2], J. Likonen[3]

Introduction

The rising demand for energy associated with the need to reduce greenhouse gas emissions produced by fossil fuels makes nuclear fusion an important resource in the energy scenario for the future. Materials in fusion reactor are subject to high mechanical, thermal and magnetic loads. These are more prominent in plasma facing components which are exposed to extreme working conditions during the lifetime of fusion devices where erosion and re-deposition processes due to plasma interactions are dominant. This work addresses the behaviour of JET Outer Poloidal Limiters and Divertor tiles (Fig.1) under plasma irradiation.

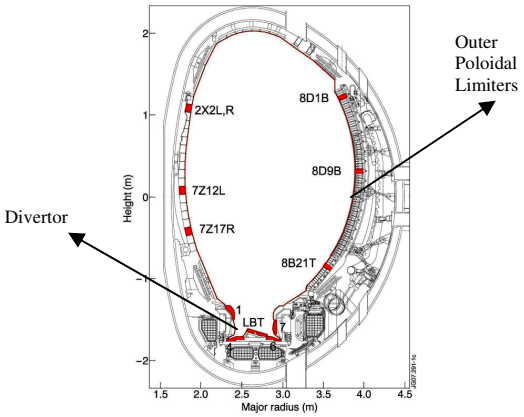

Fig.1 JET Chamber.

Results

Standard Carbon Fiber Composites and W coated tiles overlaid with a 10 micron layer of C on top were studied with RBS/PIXE to understand the erosion/redeposition processes occurring in this region of the reactor chamber. The surface morphology was studied with electron microscopy. The retention of hydrogen isotopes in the tiles was measured combining NRA and ERDA techniques (this is mostly ^{2}H from the fuelling gas, but ^{3}H is also present as a result of D-D fusion reactions and ^{1}H coming from the atmospheric exposure). Figure 2 shows a typical example of the results where the RBS, NRA and ERDA spectra obtained in the central region of tile 8D9B. Besides the presence of W, some other metal impurities and oxygen are present in the sample. Furthermore the profiles are continuous within the entire region probed by the ion beam. The ERDA and NRA results (inset of fig.2) confirm the presence of large amount of D distributed in the first microns of the samples. Since the energy of the ^{3}He particles was 2.3 MeV the deuterium inside the samples is probed down to 6-7 µm. The thickness of the layer containing the impurities was measured by cross section analysis with a proton microbeam. The cross section maps for Ni and W show a complete overlap of the depth profile of the impurities in the first 10-15 µm of the samples. It was also observed an increase of the Ni concentration at the ends of the tile (Fig. 3). These results are related with the plasma dynamics during operation: in the first moment the interaction happen with the tiles at the center, like tile 8D9B, causing net

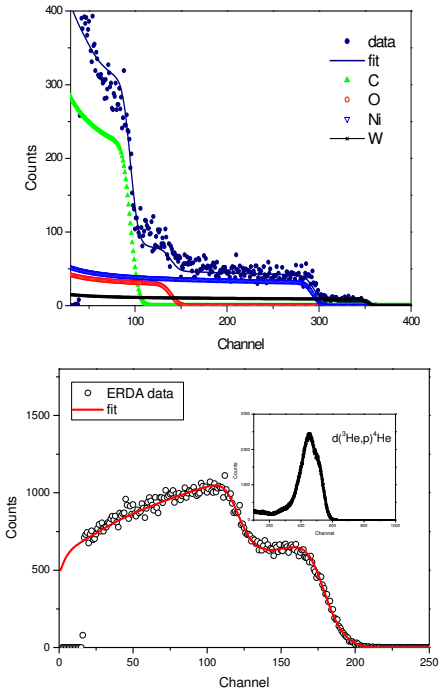

Fig.2 RBS, ERDA, NRA depth profiling for ^{1}H and ^{2}H.

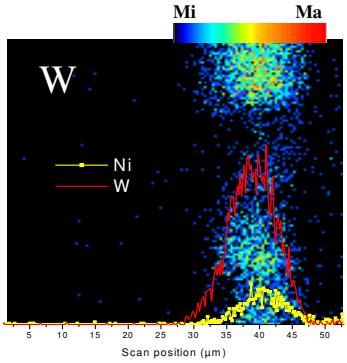

Fig.3 µPIXE cross-section analysis of a poloidal tile.

erosion, which is responsible for the removal of the 10 µm thick C coating. In this case even the W layer was removed since the composition of the first microns contains a mixture of elements from the plasma (Fe, Be, O and mostly Ni), arising from the inconel components of the chamber walls. The results also suggest that fuel retention occurs during the redeposition of the plasma impurities.

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Influence of temperature and plasma composition on deuterium retention in refractory metals*

E. Alves, L.C. Alves, N.P. Barradas, R. Mateus[1], P.A. Carvalho[1], G.M. Wright[2]

Work has been performed for the study of tungsten and molybdenum metals exposed to high flux densities (~ 10^{24} D/m^2.s) and low temperature (T_e ~ 3 eV) deuterium plasmas in Pilot-PSI irradiation facility. The hydrogenic retention in polycrystalline W and Mo targets was studied with ^{3}He nuclear reaction analyses (NRA) and elastic recoil detection analysis (ERDA). The NRA results clearly show a two dimensional radial distribution of the deuterium with a minimum at the center and a maximum close to the edge of the targets. These distribution correlates well with the thermal profile of the sample surface, where a maximum of ~ 1600 K was measured at the center decreasing to ~ 1000 K in the edges. A maximum deuterium fluence retention of 5 x 10^{15} D/cm^2 was measured. The values of the retained fractions ranging from 10^{-5}-10^{-6} were measured with thermal desorption spectroscopy (TDS) and compares well with IBA results. Moreover the presence of C in the plasma and its codeposition increases the retention of D in the region where a C film is formed. Both NRA and TDS results show no clear dependence of retention on incident fluence suggesting the absence of plasma related traps in W under these conditions.

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Enhancement of the photocatalytic nature of nitrogen-doped PVD-grown titanium dioxide thin films

E. Alves, N.P. Barradas, C.J. Tavares[1], S.M. Marques[1], T. Viseu[1], V. Teixeira[1], J.O. Carneiro[1], F. Munnik[2]

In order to increase the photocatalytic efficiency of titania coatings it is important to enhance the catalysts absorption of light from the solar spectra. Bearing this in mind, a reduction of the titania semiconductor band-gap has been successfully attempted by using nitrogen doping from a co-reactive gas mixture of N_2:O_2 during the titanium sputtering process. The as deposited thin films were mostly amorphous, however after a thermal annealing at 500 ºC in vacuum the crystalline polymorph anatase and rutile phases have been developed, yielding an enhancement of the crystallinity. Spectroscopic ellipsometry experiments enabled determining the refractive index of the films as a function of wavelength, whilst alloyed optical transmittance estimating indirect band-gap of these coatings, which decreases as the N-doping increasing. Combined RBS and HI-ERDA experiments, and subsequent spectra refining, revealed the atomic composition, thickness and level of atomic nitrogen doping of the titania layers. It was found that for a maximum of 1.19 at% of nitrogen in the titania lattice there was a clear improvement on the UV-photocatalytic efficiency towards degrading a chosen dye, acting as a pollutant.

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The role of composition, morphology and crystalline structure in the electrochemical behaviour of TiN_x thin films for dry electrode sensor materials

E. Alves, N. P. Barradas, L.T. Cunha[1,2], P. Pedrosa[1,2], C.J. Tavares[2] F. Vaz[2] and C. Fonseca[1]

A morphological, structural and electrochemical study of titanium nitride (TiN_x) thin films, obtained by DC reactive sputtering on titanium substrates, was carried out for a wide range of compositions ($0 < x < 1.34$) aiming at selections the best coatings for dry biomedical electrodes. The films displayed a columnar-type structure, with morphologies strongly dependent of the composition: a compact and smooth surface was found for the Ti-rich films, ($x < 1$), whereas the N-rich films, ($x \geq 1$) displayed a rough and porous structure. The electrochemical study of the TiN_x films was performed in synthetic sweat, aiming at simulating contact with skin. The voltammetric analysis showed anodic currents higher for TiN_x films than for titanium under low and medium polarization potentials, whereas for potentials beyond 2 V the blocking behaviour of the TiN_x films allowed them to display lower current values. The passive dissolution currents in the sub-μA/cm^2 range and the charge transfer resistances of the order of the MΩ proved the excellent stability of all films in sweat conditions. Finally, the electrochemical noise analysis showed that the near-stoichiometric and N-rich films display the lowest noise, being therefore the most suitable for electrode applications, where signals in the microvolt range, such as the electroencephalographic (EEG) signals, are to be monitored.

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Strain dependence electrical resistance and cohesive strength of ITO thin films deposited on PVDF

L. Rubio-Peña[1], C. Oliveira[2], L. Rebouta[2], S. Lanceros-Mendez[2], C. Tavares[2],E. Alves

Transparent conducting Indium Tin Oxide (ITO) films have been deposited, by dc and pulsed dc magnetron sputtering, on glass and electroactive polymer - poly vinylidene fluoride – (PVDF) substrates. PVDF substrates with thicknesses of 28 μm and 110 μm were used and samples were prepared at room temperature varying the partial pressured oxygen. The deposition rate was about 20 nm/min and the thicknesses of the films were about 100 nm. Electrical resistivity around $8.4x10^{-4}$ Ω.cm have been obtained for films deposited on glass, while a resistivity of $1.7x10^{-3}$ Ω.cm have been attained in similar coatings on PVDF. Tensile tests were performed in order to investigate the cohesive strength of the coating and the influence of mechanical strain on the electrical properties. During the elongation test, the crack onset strain, the crack density at fragmentation saturation, as well as, the evolution of the sheet resistance has been measured. The crack onset strain is similar for the different ITO coatings and is unaffected by the substrate thickness (28 and 110 μm). The crack onset strain occurs for nominal strains around 2 %. The sudden increase in the resistance of the sample correlates with onset of cracks**.** The cohesive strength of the coating was evaluated from the crack onset strain and found to be between 2.8 GPa and 3.0 GPa.

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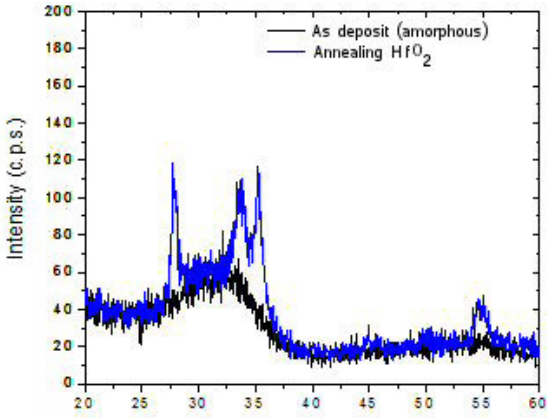
Study of multicomponent HfO_2.Al_2O_3 Gate Dielectrics

Z.L. Pei[1], L. Pereira[1], G. Gonçalves[1], P.Barquinha[1], A.M.B. Rego[2], R. Martins[1], E. Fortunato[1],N. Franco, E. Alves

Hafnium oxide–aluminum oxide (HfAlO) dielectric films were cosputtered using HfO_2 and Al_2O_3 targets, and their properties are studied in comparison with pure HfO_2 films. The X-ray diffraction studies confirmed that the HfO_2 films are nanocrystalline with monoclinic phase. The as-deposited HfAlO films with a chemical composition $(HfO_2)_{0.86}(Al_2O_3)_{0.14}$ are amorphous even after annealing at 500 °C. Further, the cosputtered films show a slight reduction in leakage current. The leakage current density may be significantly reduced below 3×10^{-10} Acm^{-2} at an electric field of 0.25 $MVcm^{-1}$ when applying the proper radio-frequency bias to the substrate.



XRD spectrum of HfO_2 films.

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Study of diffusion and oxidation processes in Zr/Hf/Zr trilayers during annealing

A.Kling, J.C. Soares[1]

The Zr/Hf system is highly interesting due its various technical applications, e. g. the production of gate oxide layers with high dielectric coefficients by oxidation of Zr/Hf multilayers. For annealing under air or oxygen atmosphere the desired oxidation process may compete with unwanted metal diffusion triggered by the high chemical similarity of the two metals. Therefore the influence of these effects at low (500 ºC) and high (1200 ºC) annealing temperatures were studied by RBS in a Zr-Hf-Zr trilayer system (each metal layer 50 nm thick). For the measurements He^+ ion beams with energies of 2.0, 2.3 and 2.525 MeV were used. The 2.525 MeV He^+ beam allowed using the small resonance in the scattering MeV ^{16}O occurring at 2.484 MeV and which facilitates the detection of oxygen in the surface region of the samples. In the case of low temperature annealing it is found that the oxidation progresses slowly starts from the surface while the interfaces between the metal layers remain stable. On the other hand, high temperature annealing leads to an asymmetric Hf-diffusion into the surface and the interior Zr-layer which is provoked by anomalous diffusion due to a phase transition in Zr. This results in two mixed Zr-Hf oxide layers sandwiching a pure HfO_2 layer.

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Photosensitivity of nanocrystalline ZnO films grown by PLD

C.P. Marques, E. Alves, R. Ayouchi[1], L. Bentes[1], C. Casteleiro[1], O. Conde[2], A.M.C. Moutinho[3], H.P. Marques[3], O. Teodoro[3], R. Schwarz[1]

Due to their direct band gap of 3.3 eV and their high electrical conductivity, thin films of zinc oxide (ZnO) are widely used in practical applications such as transparent conducting oxides for flat panel displays, solar cells, ultraviolet (UV) lasers and thin films transistors. The properties of ZnO thin films grown by laser ablation of ZnO targets on (0 0 0 1) sapphire (Al_2O_3), under substrate temperatures around 400 °C were analysed. The films were characterized by different methods including X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD) and atomic force microscopy (AFM). XPS analysis revealed that the films are oxygen deficient, and XRD θ–2θ scans and rocking curves indicate that the ZnO thin films are highly *c*-axis oriented. All the films are ultraviolet (UV) sensitive. The films deposited at higher temperatures show crystallite sizes of typically 500 nm, a high dark current and minimum photoresponse. In all films we observe persistent photoconductivity decay. More densely packed crystallites and a faster decay in photocurrent is observed for films deposited at lower temperature. Sensitivity is maximum for the films deposited at lower temperature.

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Structural and optical properties of $Zn_{0.9}Mn_{0.1}O$/ZnO core-shell nanowires designed by pulsed laser deposition

L.C. Alves, N. Franco, and E. Alves, V.E. Kaydashev,[1] E.M. Kaidashev,[1] M. Peres,[2] T. Monteiro,[2] M.R. Correia,[2] N.A. Sobolev[2]

Core-shell nanowires (NWs) have recently attracted considerable attention as possible candidates for building blocks of nanoscale devices in photonics, spintronics, and magneto-optics. Core-shell ZnO/ZnMnO nanowires on *a*-Al_2O_3 and GaN (buffer layer)/Si (111) substrates were fabricated by pulsed laser deposition using a Au catalyst. Two ZnO targets with a Mn content of 10% were sintered at 1150 and 550 °C in order to achieve the domination of paramagnetic MnO_2 and ferromagnetic Mn_2O_3 phases, respectively. RBS and XRD reveal that the decrease in the PLD target synthesis temperature from 1150 ºC to 550 °C leads to a change in the target compound and to a change in the nanowire shell morphology from smooth to snowflake like. Cluster mechanism of laser ablation is a source of possible incorporation of secondary phases to the wire shell. Raman spectroscopy under excitation by an Ar^+ laser revealed a broad peak related to the Mn-induced disorder and a red shift in the A_1-LO phonon. Besides the UV emission, a vibronic green emission band assisted by a ~71 meV LO phonon is also observed in the photoluminescence spectra. Core-shell structures with smooth shells show a high exciton to green band intensity ratio (~10) even at room temperature.

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Characterization of mesoporous ZnO:SiO_2 films obtained by sol-gel method

R.M.S. Martins, N. Franco, V. Musat [1], A. Mücklich [2], E. Fortunato[3]

ZnO:SiO_2 films are intensively investigated for optical and electronic applications. Additionally, porous ZnO:SiO_2 films are very interesting as catalyst and gas sensing materials. The present study reveals the effect of the withdrawal speed on the structure, microstructure and optical properties of mesoporous ZnO:SiO_2 films obtained by sol-gel method. The morphology of the films was investigated by atomic force microscopy and the overall structure was studied by X-ray diffraction. The structure and size of the zinc oxide nanoparticles embedded in the silica matrix were investigated in more detail by transmission electron microscopy. These techniques have shown ZnO:SiO_2 films with cracks-free mesoporous morphology and high efficient embedding of ZnO nanoparticles exhibiting predominant (100) orientation. Furthermore, the studies on the optical properties revealed a variation of the optical transmittance (in visible and near infrared) and the optical band gap value with withdrawal speed. It was shown that ZnO:SiO_2 nanocomposites films possessing ZnO particles (100) oriented, with possible special applications in non-linear optics, could be prepared by low-temperature crystallization sol-gel method.

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[3] CENIMAT/I3N, Faculty of Sciences and Technology, New Univ. of Lisbon, Campus da Caparica, 2829-516 Caparica, Portugal.

Electrical, structural and optical characterization of copper oxide thin films as a function of post annealing temperature

V. Figueiredo[1], E. Elangovan[1], G. Gonçalves[1], N. Franco, E. Alves, S.H.K. Park[2], R. Martins[1], E. Fortunato[1]

Copper oxide (CO) is a proven p-type transparent conducting oxide (TCO) semiconductor for fabricating variety of devices. The CO films in the present study were obtained through the conventional thermal oxidation of electron beam evaporated metallic copper, and the physical properties of the films were studied in order to find the possibility to use them in p–n junction-based applications. Copper oxide thin films were obtained by annealing at temperatures ranging between 100 ºC and 450 ºC. XRD studies confirmed that the cubic Cu phase of the as deposited films changes into single cubic Cu_2O phase and single monoclinic CuO phase, depending on the annealing conditions. The crystallite size is varied between 12 nm and 31 nm and the lattice parameters of cubic Cu and Cu_2O phases are estimated to 0.360 nm and 0.426 nm, respectively. The films with Cu_2O phase showed p-type characteristics and the conductivity decrease linearly with decreasing temperature, which has confirmed the semiconductor nature of the deposited films. The calculated activation energy varies between 0.10 eV and 0.16 eV and the direct band gap was estimated between 1.73 eV and 2.89 eV.

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Magnetism in wide band gap semiconductors implanted with non-magnetic ions

R.P. Borges[1], M.M. Cruz[1], M. Godinho[1], P. Venezuela[2], M.D. Moreira[2], A.T. Costa[2], N. Franco, R.C. da Silva

Single crystals of ZnO, TiO_2 and $LaAlO_3$ have been implanted with Ar ions of 100 keV energy and fluences of 1×10^{17} and 2×10^{17} cm^{-2} at room temperature. The Ar implanted crystals displayed a week ferromagnetic-like behaviour between 10 K and 400 K. The results show that this behaviour cannot be assigned to impurities or the formation of secondary phases or aggregates and is ascribed to the lattice defects induced by the implantation. Band structure calculations (spin polarised density functional calculations) were performed in the case of ZnO considering Zn interstitials and O vacancies. No net magnetic polarisation was found for O vacancies, but in the case of Zn vacancies a magnetic moment of 1 μB was obtained. Nitrogen implanted rutile single crystals also exhibited ferromagnetic behaviour up to 380 K. By annealing at 1073 K recovery of the lattice structure lead to the decrease of the ferromagnetic moment in the case of Ar-implanted samples, while it did not change significantly in the samples implanted with nitrogen.

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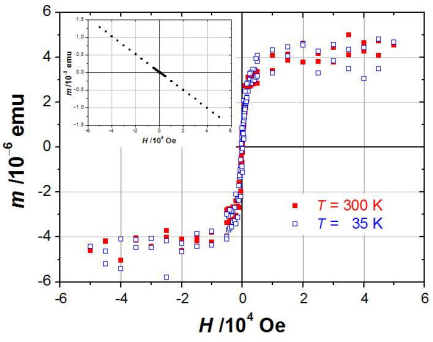
Structural properties and magnetic behaviour of ZnO implanted with Co, Ni and Mn

R.P. Borges[1], M.M. Cruz[1], M. Godinho[1], U. Wahl, E. Alves, R.C. da Silva

Investigation of the behaviour of Co, Ni and Mn ions implanted in ZnO continued, yielding new results. ZnO single crystals were doped with Co, Ni or Mn by ion implantation with fluences of 1, 2, 5×10^{16} cm^{-2} and 1×10^{17} cm^{-2} and energy 150 keV. As-implanted samples display different magnetic behaviours that are related with the atomic concentration and implanted species: single domain Ni magnetic particles form while no evidence of aggregation is found for Co and Mn ions that remain diluted in the matrix. Thermal treatment induces formation and growth of metallic aggregates: it promotes aggregation of Co ions that were diluted in the matrix immediately after implantation, while for higher fluence Ni implantations it induces growth of the aggregates formed by implantation. Remarkable agreement was found between standard X-ray and magnetisation measurements: samples where no metallic phases can be observed are always paramagnetic while the observation of diffraction peaks associated with the metallic aggregates is always associated with superparamagnetism or ferromagnetism.



[1] CFMC-Univ. de Lisboa, and Dept. de Física, Fac. de Ciências, Campo Grande, Ed. C8, 1749-016 Lisboa, Portugal.

Rare earth doping of ZnO epitaxial layers by ion implantation

S.M.C. Miranda, K. Lorenz, E. Alves, H.D. Sun[1], M. Peres[2], T. Monteiro[2], T. Geruschke[3], R. Vianden[3]

Zinc Oxide (ZnO), being a transparent wide band gap semiconductor ($E_g = 3.437$ eV at 2 K) with wurtzite structure, presents itself as a versatile oxide for transparent electronics and interesting host for optically active rare earth (RE) ions. In this work, ZnO epilayers grown by metal organic vapour phase epitaxy on (0001) sapphire substrates were doped with Pr and Eu by ion implantation, with fluences of 1×10^{13} cm^{-2} and 1×10^{15} cm^{-2}. The as-implanted samples were either annealed in air for 20 minutes in a tube furnace or rapid thermal annealed (RTA), for 2 minutes, in a nitrogen atmosphere. The samples were characterized by RBS-C. The presented results indicate that in the as-implanted samples most of the RE are incorporated into substitutional Zn-sites. Furnace annealing at 1000 ºC recovers the implantation damage but leads to an out-diffusion of the RE. RTA was then tried and results in less out-diffusion but lattice damage is not fully recovered at 1000 ºC and the RE ions are now found mainly in interstitial sites. No RE emission was found after annealing upon excitation above the band gap using a HeCd laser.

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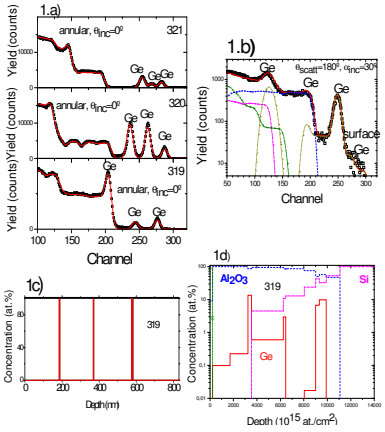
Multilayers of Ge nanocrystals embedded in Al_2O_3 matrix: vibrational and structural studies

E. Alves, N.P. Barradas, S.R.C. Pinto[1], Pedro Sá[1], A. Khodorov[1], A.G. Rolo[1], M.J.M. Gomes[1], A. Chahboun[1,2], Maja Buljan[3], R.J. Kashtiban[4], U. Bangert[4]

Nonvolatile memory (NVM) devices with floating gate structure are being used widely at present for which a long retention times are very important. Nanocrystals (NCs) floating gate has been demonstrated to improve the retention time as compared with conventional continuous floating gate. Ge NCs are good candidates for this function due to their smaller bandgap which promotes better retention and faster writing/erasing times. A set of [Al_2O_3/Ge/Al_2O_3] multilayers were grown by pulsed laser ablation with different layer thicknesses and repetition. The grown samples were annealed at 900 ºC to encourage the formation of Ge nanocrystals. RBS (figure) and TEM confirmed the multilayers system. GISAXS demonstrates the presence of Ge nanoclusters. Room temperature I-V measurements showed a weak carriers trapping in the system. This was explained by the leakage provoked by Ge diffusion through the multilayers.



a) RBS spectrum for (Ge/Al_2O_3)x3 multilayer as grown. Fits assuming the Ge is organised in QDs (solid lines) and in mixed Al_2O_3 layers (dashed lines) are shown b) RBS spectrum after annealing and corresponding fitted depth profile for the same sample as grown c) and after annealing d).

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Probing alloy disorder in AlGaN ternary alloys

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The alloy and lattice disorder as well as the annealing behaviour of $Al_xGa_{1-x}N$ films on sapphire substrate after implantation was studied using two different approaches. Perturbed angular correlation (PAC) measurements using the ^{181}Hf(^{181}Ta) probe show that the strength of the electric field gradient, which is caused by the wurtzite structure of the host lattice at the probe site, varies linearly with the concentration x of AlN in the ternary compound. The uniformity of this hyperfine interaction has its minimum at x~0.6. The photoluminescence linewidth of implanted optically active Eu ions was seen to have a maximum for x~0.6 decreasing for lower and higher AlN contents.Both techniques were proved valid to study alloy and lattice disorder in the AlGaN ternary and showed a maximum disorder in the same compositional region as seen for excitons in this alloy.

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Structural and photoluminescence studies of erbium-implanted nanocrystalline silicon thin films

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A large improvement efficiency in light emission from Si-based materials is required if fully integrated silicon optoelectronics is to become reality in the near future. Rare-earth doping of silicon has consistently attracted scientific interest as a strategy to achieve this goal, together with the exploitation of quantum confinement in silicon nanocrystals. Hydrogenated amorphous and nanocrystalline silicon thin films deposited by hot wire (HW) and radio-frequency plasma-enhanced chemical vapour deposition (RF-PECVD) were erbium-implanted. Their pre-implantation structural properties and post-implantation optical properties were studied and correlated. After 1 h annealing at 150 °C in nitrogen atmosphere only amorphous films showed photoluminescence (PL) activity at 1.54 µm, measured at 5 K. After further annealing at 300 °C for 1 h, all the samples exhibited a sharp PL peak positioned at 1.54 µm, with a FWHM of ~5 nm. Amorphous films deposited by HW originated a stronger PL peak than corresponding films deposited by RF, while in nanocrystalline films PL emission was much stronger in samples deposited by RF than by HW. There was no noticeable difference in Er^{3+} PL activity between films implanted with 1×10^{14} cm^{-2} and 5×10^{15} cm^{-2} Er fluences. Severe PL quenching was observed in all samples when measured at RT. PL temperature dependence study indicates that the hydrogen content plays an important role on the Er^{3+} PL quenching.

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Europium implantation and high temperature annealing of AlN

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AlN was implanted with 300 keV Eu ions within a wide fluence range from 4×10^{14} cm^{-2} to 1.4×10^{17} cm^{-2}. The damage build-up was investigated by RBS-C. Sigmoidal shaped damage build-up curves indicate efficient dynamic annealing. A regime with low damage increase for fluences below 10^{15} cm^2 is followed by a strong increase for intermediate fluences. For the highest fluences the damage curve rises slowly until a buried amorphous layer is formed. High temperature annealing was performed in nitrogen atmospheres at low pressure (1300 ºC, 10^5 Pa) or at ultra-high pressure (1450 ºC, 10^9 Pa). Implantation damage was found to be extremely stable and annealing only resulted in slight structural recovery. For high fluences out-diffusion of Eu is observed during annealing. Nevertheless, photoluminescence (PL) measurements show intense Eu-related red light emission for all samples with higher PL intensity for the high temperature high pressure annealing.

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Annealing and implantation studies of self-assembled GaN quantum dots

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The influence of post-growth thermal annealing and ion implantation on the structural and optical properties of self-assembled GaN quantum dots (QD) in GaN QD/AlN superlattices was studied. X-ray techniques suggest smooth and high quality interfaces of the stacked multilayer structures for the as-grown, annealed and implanted samples. High-angle annular dark field images by scanning transmission electron microscopy show an intermixing between the GaN QD and AlN spacers after annealing and the QD emission shifts to lower energies (red shift) for big dots and to higher energies (blue shift) for small dots, reflecting two competitive processes taking place during the thermal annealing. Implantation leads to an increase of the *c* lattice parameter within the implanted region of the superlattice which is partly reversed during post-implant thermal annealing. Implantation induced intermixing of the layers is observed.

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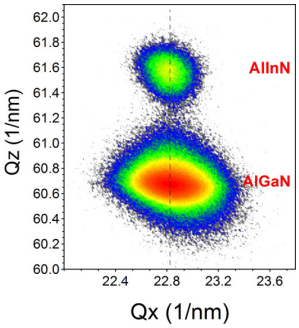
Comparison of the structural properties of $Al_{1-x}In_xN$ films grown on $Al_{1-y}Ga_yN$ templates with different AlN contents

S. Magalhães, K. Lorenz, N. Franco, E. Alves, S. Pereira[1], R.W. Martin[2], K.P.O'Donnell[2], I.M. Watson[3]

The group III nitride semiconductors with their wide and direct band gap are interesting for the fabrication of optoelectronic devices. The purpose of this work is to compare the structural properties of AlInN films grown on GaN and AlGaN buffer layers with different in-plane lattice constants. 100 nm thick AlInN films were grown by metal organic vapour phase epitaxy on pre-grown $Al_{1-y}Ga_yN$ templates with AlN contents varying from 0 % to 33 %. Structural, compositional and morphological analyses were performed using RBS/C, XRD and AFM. The AlInN alloy films grown on GaN have a smoother surface than those grown on AlGaN and have a lower RBS/C minimum yield in the growth direction indicating a higher crystalline quality. There are no lateral AlInN composition variations but some defect phenomena change the alloy crystal quality for different sections of the wafer. XRD reciprocal space maps reveal the absence of macroscopic tilts and show that the films are completely strained (pseudomorphic to the different buffer layers).



RSM of a pseudomorphic film.

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Electron accumulation at nonpolar and semipolar surfaces of InN

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A peculiar intrinsic electron accumulation was found to occur at polar (0001) oriented InN films. The surface electron accumulation complicates the electrical measurements masking the true bulk conductivity, which hinders the assessment of doping in the material, in particular p-type doping. Understanding the surface electron accumulation properties is a key to make further progress in this direction. In particular, surfaces other than the polar c-plane should be explored in order to probe the possibility to minimize or eliminate the surface electron accumulation density. We have studied the free electron properties of nonpolar (11-20)- and semipolar (10-11)-oriented wurtzite InN films by generalized infrared ellipsometry (GIRSE). We have demonstrated the sensitivity of GIRSE to the surface charge accumulation layer and find a distinct surface electron accumulation to occur at all surfaces. The obtained surface electron sheet densities are found to vary from $0.9x10^{13}$ cm^{-2} to $2.3x10^{14}$ cm^{-2} depending on the surface orientation and bulk electron concentration. The upper limits of the surface electron mobility parameters of 417 cm^2/Vs-644 cm^2/Vs are determined and explained in the light of electron confinement at the surface.

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Determination of the stiffness constants of $In_xAl_{1-x}N$ ($0 \le x \le 1$) alloys

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$In_xAl_{1-x}N$ alloys have potential applications in light emitting devices from the red to the deep UV range of the spectrum, and in high-power/high speed electronics. Despite the intense research in the field most of the fundamental alloy properties, such as the band gap, are still controversial and others, such as the alloy stiffness constants, remain unknown. We have obtained for the first time the stiffness constants of $In_xAl_{1-x}N$ alloys in the whole compositional range by *ab-initio* calculations and determined their deviations from Vegard's rule to be $\delta_{C_{11}} = 98.6$ GPa , $\delta_{C_{12}} = 22.7$ GPa, $\delta_{C_{13}} = -4.4$ GPa and $\delta_{C_{33}} = 6.3$ GPa. We have shown that the error in the alloy composition extracted from the lattice parameters of polar *c*-plane oriented films could be reduced by taking into account the deviations from Vegard's rule in the lattice parameters and stiffness constants. We also have determined the effect of the deviations from Vegard's rule on the accuracy of In composition for $In_xAl_{1-x}N$ films with nonpolar orientations (*m*- and *a*-plane) and on the piezoelectric polarization for polar $In_xAl_{1-x}N$ films.

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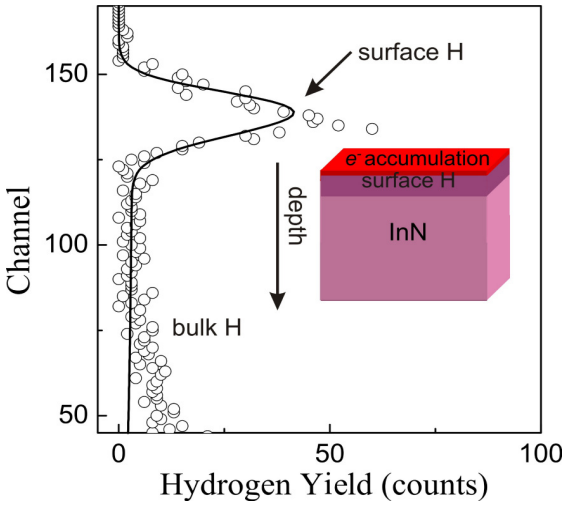
H impurity depth profiles and unintentional doping in InN films

V. Darakchieva, K. Lorenz, N.P. Barradas, S.M.C. Miranda, E. Alves, W.J. Schaff,[1] C.L. Hsiao,[2] L.C. Chen,[2] L.W. Tu,[3] T. Yamaguchi,[4] Y. Nanishi[4]

InN and its alloys with GaN hold a great potential for applications in advanced solar cells, high-speed electronics, THz emitters and sensors. One of the most critical issues is understanding the origin of the unintentional n-type doping in InN materials. Unintentionally introduced H is among the most plausible sources of doping. We have determined the H depth profiles in a large number of state-of-the-art InN films with different surface orientations and bulk free carrier concentrations by ERD analysis. Enhanced concentrations of H were found in the near surface regions of the films indicating post-growth surface contamination that could not be removed upon thermal annealing. Furthermore, we have found that the bulk free electron concentrations in the c-plane oriented InN films scale with the bulk H concentrations, indicating a major role of H in unintentional doping. The near surface H may have significant implications for the surface electron properties of InN and act as reservoir for co-doping the bulk.



Elastic recoil detection profiles of hydrogen in a representative InN film with c-plane orientation and free electron concentrations in the mid 10^{18} cm^{-3} range. An enhanced concentration of H is revealed in the near surface region of the film and a bulk concentration of H of 0.5 at% is estimated from the fit (solid line) to the experimental data (circles).

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Structural properties of nonpolar InN films

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Nitride materials with nonpolar surface orientations (i.e. with the c-axis parallel to the growth plane) offer attractive opportunities to avoid the built-in electric fields in nonpolar nitride heterostructures and therefore overcome some of the current issues in the performance of light emitting diodes. However, the nonpolar growth results in typically poorer structural properties and the presence of anisotropic strain in the films, which in turn will affect the device-relevant properties of the material. We have studied the structural characteristics of nonpolar a- and m-plane InN films with respect to the growth conditions needed for minimizing the rotational disorder and enhance the lateral coherence lengths in the films. We have complemented these with results on the minimum yield determined from RBS-C experiments. Furthermore, we have measured the lattice distortions in the main crystallographic directions.

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Strain measurements in nitride heterostructures by ion channelling: experiment and Monte Carlo simulations

A Redondo-Cubero[1,2], K Lorenz, E. Alves, R. Gago[2,3], S. Fernández-Garrido[1], P.J.M. Smulders[4], E. Muñoz[1], E. Calleja[1], I.M. Watson[5]

Ion steering effects in the interface of heterostructures (HS) can strongly influence the shape and position of angular channelling scans leading to considerable error in the determination of strain by ion channelling. With the help of Monte Carlo simulations, three composition/strain regions for AlInGaN/GaN HS were established for a typical beam of 2 MeV alpha particles corresponding to different intensities of the steering potential and in which strain measurements by ion channelling are (a) correct, (b) possible but require corrections and (c) not possible due to steering effects. Furthermore, the influence of the beam energy on the determination of the strain state with ion channelling was addressed: experimental results show that steering effects at the HS interface are more intense at lower ion energies. The experimental angular scans have been well reproduced by Monte Carlo simulations, correlating the steering effects with the close encounter probability at the interface. Consequently, limitations in the determination of the strain state by ion channelling can be overcome by selecting the adequate beam energy.

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Tin and germanium rich chalcopyrite from the Barrigão mine

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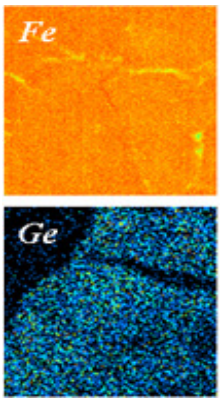
Ore samples from the Barrigão mine, located in the Iberiam Pyrite Belt, close to Neves Corvo, were investigated in order to determine the contents of technological important elements. Whole-rock analysis showed tin and germanium contents sometimes exceeding 200 µg/g, thus revealing their potential economical interest. Further analysis was performed through Nuclear Microprobe techniques in polished or thin section samples for obtaining elemental distribution maps and so be able not only to identify and map the several minerals present but also look for the minerals responsible for bearing the germanium and/or tin. Containing essentially tenantite and chalcopyrite, the obtained maps clearly showed that the Ge was only present in some chalcopyrite grains with contents ranging from 0.3%-0.6%. These grains also contained some amounts of tin (up to 1%) as well as arsenic (~0.15%). Electron Microprobe Analysis further explored the most interesting samples zones mapped by Nuclear Microprobe analysis. Due to its improved beam spatial resolution it may ascertain if Ge and Sn are contained in submicroscopic phases.



Composition maps of a Ore sample.

[1] INETI/LNEG, Alfragide; [2] CREMINER/FCUL; [3] INETI/LNEG, Beja; [4] INETI/LNEG, S. Mamede de Infesta.

Ni-Ti surface modification for enhanced biocompatibility and corrosion in biomedical applications

R.M.S. Martins, N. Barradas, E. Alves, D. Henke[1], J.C.S. Fernandes[2]

Ni-Ti Shape Memory Alloy is characterized by a specific stress–strain diagram that is different from the deformation behaviour of conventional materials but similar to that of living tissues. In spite of its attractive mechanical properties, the use of Ni-Ti raises concerns due to its high Ni content. Ni release from Ni-Ti depends on its corrosion resistance, which relies on the presence of a passive oxide layer (TiO_2). In this study a method (using plasma-immersion ion implantation) is being implemented to modify and improve the Ni-Ti alloy surface for biomedical applications, without deteriorating the mechanical properties, aiming at forming a Ni-depleted surface, to serve as a barrier against out-diffusion of Ni. Ion implantation of oxygen into Ni-Ti has been carried out, in order to promote selective oxidation of Ti, leading to a Ni-depleted surface.

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The effect of Mn, Fe and Cu ions on potash-glass corrosion

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

The corrosion behaviour of model potash-glasses doped with Mn, Fe or Cu ions, was studied by analysing the glass surfaces after exposure to different simulated weathering conditions. Glass samples with 56 mol.% SiO_2, 24 mol.% CaO and 20 mol.% K_2O were prepared, with 1 mol% of the different metal oxides, a composition analogous to that of the medieval stained glasses of the XV century from the Monastery of Batalha. The experimental conditions used reproduced well the natural corrosion processes found in ancient glasses of similar composition and weathered through five centuries: silica-rich layers containing $CaCO_3$ were identified on the surface, with more than one such layers developing during longer weathering periods, indicating progression through a sequence of dissolution steps. The elemental profiles obtained from the attacked surfaces, showed that upon weathering glasses doped with Cu, Mn or Fe, develop a layer richer in the doping ions on the surface. Addition of ions Cu, Mn or Fe, was found to influence corrosion: the initial rates are always higher in Cu, Mn or Fe doped glasses than in undoped glasses. Copper containing glasses display the faster initial corrosion. The measurements also allowed establishing a correlation between the presence of the doping ions and the increase in the number of Si–O groups in the surface of the glasses. This is viewed as favouring the adsorption of hydronium ions and the later exchange with e.g. K and Ca.

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Study of colouring elements in Portuguese medieval stained glasses: techniques and mechanisms

M. Vilarigues[1], P. Fernandes[1], L.C. Alves, R.C. da Silva

Non-destructive characterisation of glasses from the 15th, 16th and 20th centuries, and belonging to Mosteiro de Santa Maria da Vitória, Batalha (Portugal), continued through use of the nuclear microprobe. Fe, Cu and Pb were the main elements identified in the grisailles of all studied periods, and Ag and Cu found in the glasses decorated with yellow silver painting. Their distributions allowed establishing a definite relation between the compositions found and the periods of production, as well as correctly reassigning the manufacturing period of some samples: the glasses analysed could be divided into two major groups: potash glasses (from the 15th and 16th centuries) and soda-lime glasses (from conservation-restoration works from the beginning of the 20th century). It was established that the grisailles were produced with a mixture of Fe, Cu and Pb, the main differences in composition laying in the contents of Pb: about 5 mol% in the original grisailles, more than 10 mol% for those produced in the 20th century. Their structure was found not to vary significantly with the production period: Fe and Cu concentrated in the form of a 15 μm thick dispersion of solid grains in the centre most region of a 30 μm thick lead silicate layer, indicating that the temperatures used were sufficient to bind the fondant to the glass substrate, but not enough to melt Fe or Cu. In what concerns yellow silver staining, the Cu-Ag association found from the elemental maps, indicates that not only a Cu-Ag mixture must have been used as the materials source, but annealing processes as well, whereby silver penetrated into the glass and precipitated, leaving Cu behind at the surface.

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***In-situ* X-ray diffraction studies during co-sputtering deposition of Ni-Ti shape memory alloy films**

R.M.S. Martins, N. Schell[1], J. von Borany[2], K.K. Mahesh[3], R.J.C. Silva[3], F.M., Braz Fernandes[3]

Ni-Ti films have attracted much interest as functional and smart materials and are considered attractive candidates for micro-electro-mechanical systems (MEMS) applications. However, the deposition of Ni-Ti films with definite stoichiometry and high purity remains a challenge. In this study near equiatomic (≈50.0 at.% Ti–Ni) and Ti-rich (≈50.8 at.% Ti–Ni) Ni-Ti polycrystalline films (thickness values up to 800 nm) have been deposited by magnetron co-sputtering using a chamber installed into the six-circle diffractometer of the Rossendorf Beamline (ROBL) at the European Synchrotron Radiation Facility. The *in-situ* X-ray diffraction studies enabled the identification of different steps of the structural evolution during films processing. Films exhibiting a (100) preferential orientation for the B2 phase have been successfully produced. A continuous increase of the B2(200) diffraction peak intensity has been observed for depositions on a 140 nm amorphous SiO_2 buffer layer heated at 520°C (without substrate bias voltage, V_b). A (100) texture has been observed for films as thick as 800 nm. Near equiatomic and Ti-rich Ni-Ti films deposited without and with V_b on a TiN coating with a topmost layer formed by <111> oriented grains have shown a preferential growth of <110> oriented grains of the B2 phase from the beginning of the deposition. Ni-Ti films with a thickness as low as 100 nm exhibit a (110) preferential orientation for the B2 phase.

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AMS analysis of low dose Pt implantation in Si

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In order to study the role of isotopic effect on optical spectra, pure ^{28}Si single crystals were implanted, at an energy of 60 keV, with ^{194}Pt and ^{198}Pt, with nominal fluences around 1×10^{14} cm^{-2}. Since a natural Pt target was used for the implantation some isotopic contamination is expected, despite the mass selection. AMS, particularly Microbeam AMS, was employed for the verification of the implanted isotopic ratio and the determination of the amount of ^{195}Pt and ^{196}Pt contamination. For tuning up the beam transport system, a platinum powder target was used, producing current intensities at the high energy side of around 50 pA for the +4 charge state of the different isotopes. For the implanted samples, currents up to 1×10^{3} particles per second were obtained. The isotopic ratios were found to be 0.76±0.06 for $^{198}Pt/^{194}Pt$, 0.14±0.03 for $^{195}Pt/^{194}Pt$ and 0.24±0.03 for $^{196}Pt/^{194}Pt$.

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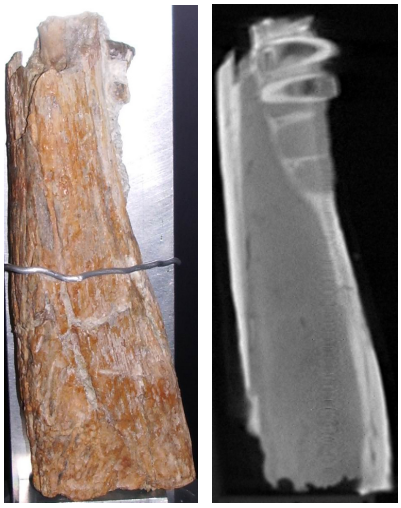
Dinosaur and crocodile fossils from the Mesozoic of Portugal: neutron tomography

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

Portugal is ranked seventh with regard to dinosaur taxa and the area of Lourinhã is known by the Late Jurassic findings of dinosaurs and other fossils. The paleontological research in this area has been carried out by the "Museu da Lourinhã" in collaboration with the "Universidade Nova de Lisboa" and described in several scientific articles (available at http://publicationslist.org/omateus). In many cases, studies of the external morphological characteristics of the fossils are not sufficient to extract all the information for a paleontological study and, thus, observations of internal morphology are required. Access to the Geesthacht Neutron Facility (GeNF) allowed a unique and non-destructive characterization of the fossils by neutron tomography. A total of 8 samples have been studied: Archosaur nest – Late Jurassic, Crocodylian egg, Temnospondyl jaw – Late Triasic, two Theropod Lourinhanosaurus Antunesi eggs, Baryonyx walkeri jaw, Tomistomidae (?) skull, Tomistomidae (?) jaw (Figure). During the experiments the samples were exposed for 16 s at equally stepped (0.25°) tilts between –180° and 180°. From the single projections, slices perpendicular to the rotation axis were reconstructed by a tomographic reconstruction algorithm using "filtered backprojection." The slices were then collected in an image stack allowing visualization, edition and conversion into different file formats using a 3-D rendering software. The present study gives precious information about the internal morphology of the fossils, providing a direct window into the evolutionary history of development.



Tomistomidae jaw photograph and yz-slice

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A new ion beam analysis data format

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Computational Ion Beam Analysis (IBA) codes such as RUMP, SIMNRA, NDF, and others implement various formats to store the spectral data and to describe the experimental conditions and simulation or fit parameters. Additionally, many laboratories have developed their own internal data formats. These various data formats are isolated applications and generally incompatible. The need for a universal IBA data format (IDF) has been recognised for many years to allow easy transfer of data and simulation parameters between codes, as well as between experimentalists and data analysts. To be effective, the IDF must be transparent (easily read by an IBA practitioner), universal (catering to varying needs), and must include the most common features desired by both experimentalists who collect and archive data and by users who analyse the data. The IDF must also be readily extensible in order to include features specific to individual codes and laboratories, as well as being able to incorporate new features and options in the future. We have developed such a data format. It is currently being implemented in the most popular general purpose IBA data analysis codes.

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Artificial neural networks for real time in-situ RBS analysis

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Two years ago, we revived the Artificial Neural Networks work that we had previously developed, to apply it to real time RBS characterization, i.e. performing the analysis during thin film growth. Entire systems are solved after a few days of work, as opposed to months previously. Two papers were submitted in 2009. This was done in collaboration with the Leuven University, that are the drive behind the real time RBS experiments, basically by transferring the knowledge on ANNs to Leuven, who from now on will continue to develop ANNS. We consider the role of ITN in this research to be now concluded.

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Ion Implantation Automation System

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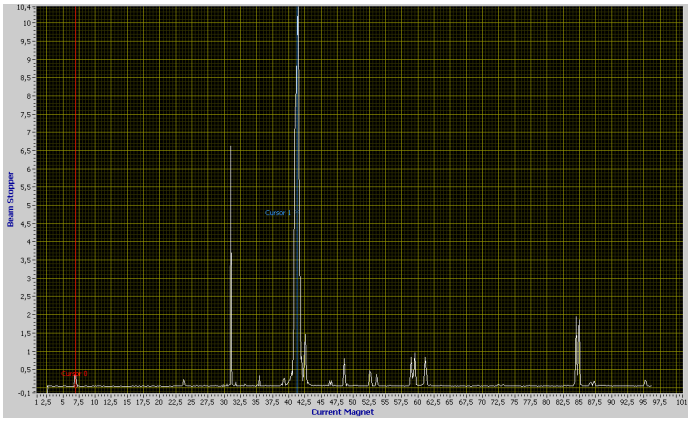
In order to obtain an accurate implantation of an isotope or to avoid the existence of more than one element on the target, mass spectrometry is fundamental. Until recently in ITN ion implanter the mass spectrum has been obtained in a plotter with the magnetic field being controlled manually through the current source. With the system developed, the mass spectrometry is made through a PC application and the mass spectrum is displayed in the PC screen in real time. The system consists of a PC, a data acquisition I/O board composed by multifunction input/output board NI USB-6251 and four electronic modules using optic fiber control. The computer control code uses a LabVIEW synoptic for interaction with the operator.



Mass Spectrum obtain through LabVIEW.

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