Advanced Materials Research

Eduardo Alves

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

The recent research work of the group has been focused on the study of advanced materials with high technological impact, e.g. wide band gap semi-conductors and nanostructures. Wide bandgap semiconducting (III-nitrides) and oxide (ZnO) materials are the base of an emerging class of optoelectronic devices operating in the visible wavelength range of the electromagnetic spectrum being under intense research worldwide. The potential of these materials for spintronics applications is also being investigated. Our work aims at the optimization of the implantation conditions of magnetic and optically active dopants in these materials.

Other exciting study is being carried out in quantum dot (QD) multilayered structures. An intense research of the structural properties and Rare Earth doping of GaN/AlN QD layers continued in collaboration with Universities of Aveiro and Grenoble.

The work in insulators comprise the modification of the optical and structural properties of α-Al_2O_3 as well as the study of nanoprecipitates formed in rutile by high fluence implantation doping with transition metals.

Taking advantage of the potential 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 and University of Minho.

The activities under the technology programme of the European Fusion Development Agreement (EFDA), in association with the Centro de Fusão Nuclear of the Instituto Superior Técnico was focused on the study of beryllium intermetallics, the characterisation of the new Eurofer (ODS) steel and the study of surface erosion and ^{2}H retention in W and Mo irradiated under ITER working conditions.

In addition to the research activities the group reinforced is commitment with the training of graduate students, through the supervision of new M.Sc. and Ph.D. thesis. Also worth to be mentioned were the two new researchers joining the group under the Ciência 2007 programme.

All the referred activities are funded by projects, both European and National (FCT), either in collaboration with other Institutions or lead by members of the group. Of particular importance are the new 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 planned to start next 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.

The scientific output of the group in 2008 was:

Publications (peer reviewed journals): 49
Conference and workshop contributions: 3 invited, 24 oral and 14 posters.
Running projects: 21

Researchers(*)

E. ALVES, Principal (Group Leader)
R. C. SILVA, Princpal
U. WAHL, Principal
K. LORENZ, Aux.
V. DARAKCHIEVA, Aux. (from July 2008)
L. C. ALVES, Aux. (75%)
N. P. BARRADAS, Aux. (10%)
A. R. RAMOS, Aux. (10%)
A. KLING, Aux. (10%)

Students

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Damage build-up and annealing of Eu-implanted GaN

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Objectives

Rare earth doped GaN is widely studied with respect to applications in electroluminescent devices. Eu-doped GaN with its characteristic red emission arising from the intra-4f shell transitions of trivalent Eu^{3+} is of special interest because the efficiency of conventional GaN-based LEDs and lasers is high in the blue spectral region but is strongly quenched in the green and red.

Ion implantation is a powerful tool to introduce controllable impurity concentrations at precise depths below the surface with the added capability to dope selective areas. However, it creates lattice damage which may affect the optical properties due to the introduction of non-radiative decay paths. High temperature annealing is usually necessary to remove the implantation damage and thereby activate rare earth ions optically.

Results

The implantation damage build-up in wurtzite phase GaN (W-GaN) was studied in detail using a combination of Rutherford Backscattering Spectrometry/channelling (RBS/C) and transmission electron microscopy (TEM). Four regimes could be identified (Fig. 1): In the low fluence regime strong dynamic annealing keeps the damage accumulation rate low. The retained damage consists mainly of point defect clusters and low concentrations of basal and prismatic stacking faults. For medium fluences (regime II), implantation damage increases steeply; an increasing number of stacking faults is observed. In regime III the damage in the bulk of the samples, in particular the production of stacking faults, saturates while the damage at the surface increases further. Above a threshold fluence of 2-3×10^{15} Eu/cm^2 a highly damaged surface layer is formed consisting of randomly oriented nanocrystallites and voids.

Previously it was shown that a thin AlN layer grown on top of GaN successfully protects the GaN surface from dissociation during high temperature annealing

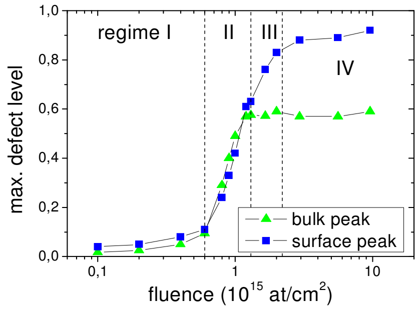

Fig. 1: Damage build-up curve for 300 keV Eu-implantation into W-GaN: The relative defect level at the maximum of the surface and bulk damage peak in RBS/C spectra as a function of the fluence.

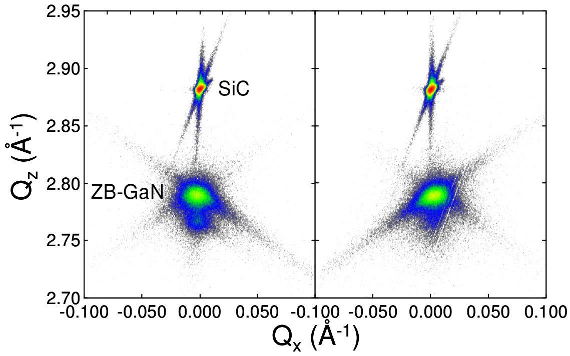

Fig. 2: X-ray diffraction reciprocal space maps around the (002) lattice point of ZB-GaN after implantation of 1×10^{14} at/cm^2 (left) and 1×10^{15} at/cm^2 (right) Eu ions at 300 keV.

and a strong increase of Eu-related luminescence with annealing temperature up to 1300 ºC was observed for samples implanted with a fluence of 1×10^{15} Eu/cm^2. Interestingly, for lower fluences the highest luminescence intensity is found at lower annealing temperature due to the sensitive balance of implantation defect removal and defect formation during annealing despite the AlN-capping. This defect formation could be suppressed by performing the annealing at extreme nitrogen overpressure of 1 GPa which led to an exponential increase of luminescence intensity up to temperatures as high as 1450ºC for a sample implanted with 1×10^{13} Eu/cm^2. This sample only showed one major Eu luminescence centre while a second, possibly defect related centre, is usually observed for higher implantation fluences.

Furthermore, Eu-implantation of cubic (zincblende) GaN (ZB-GaN) was investigated.

The implantation damage causes an expansion of the lattice parameter (Fig. 2), similar to what is observed in W-GaN. Unlike the case of W-GaN no preferential surface damage was observed. However, ZB-GaN is partially converted to W-GaN during high temperature annealing. Eu was found on a high symmetry interstitial site in contrast to W-GaN where Eu occupies near-substitutional Ga-sites. Optical activation was achieved after annealing and different Eu emission lines could be assigned to Eu-centres residing within W-GaN inclusions and Eu-centres in cubic ZB-GaN.

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Rare Earth implantation of AlN for light emission from IR to UV

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Rare Earth (RE) doped group-III nitride semiconductors (GaN, AlN, InN and their alloys) attract much research interest due to their unique optical properties with narrow and temperature stable emissions ranging from infrared to ultraviolet. As host for REs, AlN with its large band gap allows energetically high lying RE levels to be exploited and to decrease thermal quenching of the luminescence.

In this work Eu ions were implanted into AlN with a wide fluence range from 10^{14} to 10^{17} at/cm^2. The damage build-up was investigated by Rutherford Backscattering/channelling (RBS/C) revealing sigmoidal shaped damage build-up curves that indicate efficient dynamic annealing. Strong ion channelling effects were observed when the implantation was performed along the <0001> direction of the wurtzite lattice. Compared to random implantation the range of the implanted ions doubles and the defect density is reduced efficiently. For the highest fluence and random implantation a buried amorphous layer is formed which was confirmed by transmission electron microscopy analysis. After annealing all samples show bright Eu-related photoluminescence (PL) in the red spectral region. The PL intensity is strongly dependent on the lattice damage, being higher in samples implanted with low fluences and in channelled geometry.

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Defect studies and optical activation of Yb doped GaN

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Wide band-gap semiconductors, particularly III-nitrides, became one of the most studied materials during the last decades. These compounds are the base of a new generation of optoelectronic devices operating in the UV-Blue region of the electromagnetic spectrum. Incorporation of rare-earth (RE) ions into nitrides creates new routes to build all-nitride electroluminescent devices, using the sharp intra-4f^n transitions of these elements. The introduction of the RE ions in the nitride lattice during the growth or by ion implantation creates defects which influence the optical behaviour of the doped region.

In this work we report the results on Yb implanted GaN. A combination of techniques (Rutherford backscattering/Channeling and Photoluminescence) was used to assess the mechanisms responsible for the optical and structural behaviour of the doped materials. Lattice site location experiments showed that Yb is incorporated into positions slightly displaced from the Ga-site. Clearly the optical activity of the RE could be enhanced by orders of magnitude reducing the number of non-radiative recombination paths related with defects.

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Free electron behavior in InN

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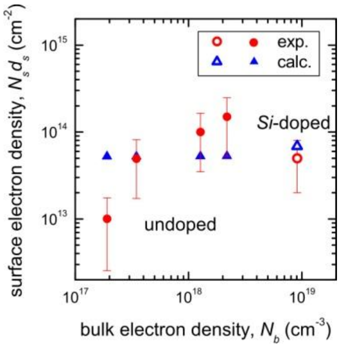
We have used precise measurements of the Optical Hall effect in InN using magneto-optical generalized ellipsometry at IR and THz wavelengths, which allows decoupling of the surface accumulation and bulk electron densities in InN films by non-contact optical means and further to precisely measure the effective mass and mobilities for polarizations parallel and perpendicular to the optical axis. We have performed studies of InN films with different thicknesses, free-electron densities, surface orientations and polarities, which enable an intricate picture of InN free carrier properties to emerge. Electron accumulation is found to occur at polar, semi-polar and non-polar surfaces of wurtzite and zinc-blende InN and the surface charge shows distinct dependence on the bulk free-electron density (see figure). Striking findings on the scaling factors of the bulk and surface electron densities with film thickness points to an additional doping mechanism or different evolution of dislocation density from currently accepted picture and suggest that the complexity of the surface chemistry might have been underestimated in the existing models.



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Structural and compositional analysis of near-lattice matched $Al_{1-x}In_xN$/GaN (0.08<x<0.21) epitaxial layers

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

The ternary alloy AlInN is attracting much research interest due to the possibility of growing it lattice matched to GaN for an InN molar fraction of ~17-18%. However, the growth of AlInN is rendered difficult by problems of phase separation during epitaxial growth, driven by a large disparity in cation sizes as well as by differences in thermal properties of the binary constituents, leading to a wide immiscibility region. A large number of AlInN/GaN bilayers with alloy layer thicknesses from 20 nm to 200 nm were grown using metal organic vapour phase epitaxy. The InN content, ranging from 8 to 21%, was controlled by means of the growth temperature. The structural properties were studied using Rutherford Backscattering/channelling spectrometry (RBS/C), X-ray diffraction (XRD) and atomic force microscopy (AFM). For nearly all samples the InN content measured *directly* by RBS is lower than that determined from XRD measurements assuming Vegard's rule, indicating a deviation of the AlInN/GaN system from this empirical law. The surface for closely lattice matched samples was smooth with RMS roughness values as low as 0.3 nm. Lower InN contents cause an increase of V-shaped pits on the surface relaxing tensile strain while higher InN contents lead to the formation of three dimensional islands. Detailed analysis of XRD rocking curves showed that the quality of the GaN buffer layer is a critical parameter for the AlInN film quality in agreement with TEM results revealing that most threading dislocations are formed inside the GaN layer and continue in the AlInN film.

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Unintentional impurities and dislocations in InN films and their role for the unintentional n-type conductivity in InN

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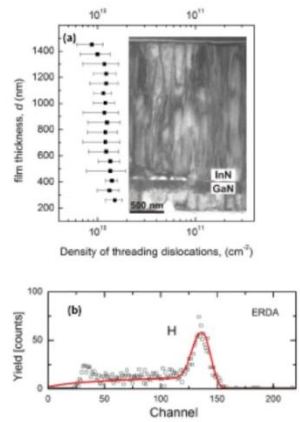
We have studied the unintentional impurities profiles and the evolution in the dislocation density in InN films with different free electron concentrations and using different nucleation schemes. The goal is to unravel the cause for the unintentional doping mechanisms in InN, which is a key issue to enable further progress in the InN based technology. We found that the dislocation densities are similar in InN films with free electron concentrations that differ by more than an order of magnitude, indicating that dislocations associated with native defects could not play a decisive role for the unintentional conductivity in InN (top figure). We also found significant amount of H in the films being much larger at the surface than in the bulk (bottom figure). Surprisingly, the amount of H is estimated to be larger in the films with lower free electron concentrations, both in the bulk and at the surface. On the other hand, no O (>0.1 at%) could be detected in the InN films but some traces of C are detected in the films, specifically for those with low free electron concentrations. Our results further suggest that minimizing the amount of O in combination with H passivation might be a successful route for enabling p-type doping of InN.



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Lattice parameters and E_2 phonons in $In_xAl_{1-x}N$

V. Darakchieva, M-Y. Xie[1] F. Tasnadi[1], I. A. Abrikosov[1], J. Kamimura[2] and K. Kishino[2]

The lattice parameters of InxAl1-xN in the whole compositional range using first-principle calculations were studied. Deviations from Vegard's rule are obtained via the bowing parameters, $\delta a = 0.0412 \pm 0.0039$ Å and $\delta c = -0.060 \pm 0.010$ Å, which largely differ from previously reported values. Implications of the observed deviations from Vegard's rule on the In content extracted from x-ray diffraction are also inferred. We also combine these results with x-ray diffraction and Raman scattering studies on InxAl1-xN nanocolumns with $0.627 \leq x \leq 1$ and determine the E2 phonon frequencies versus In composition in the scarcely studied In-rich compositional range.

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Structural and optical properties on thulium-doped LHPG-grown Ta_2O_5 fibres

N. Franco, E. Alves, M. Macatrão[1], M. Peres1[1], C.P.L. Rubinger[1], M.J. Soares[1], L.C. Costa[1], F.M. Costa[1], T. Monteiro[1], B.Z. Saggioro[2], M.R.B. Andreeta[2] and A.C. Hernandes[2]

Structural, spectroscopic and dielectric properties of thulium-doped laser-heated pedestal Ta_2O_5 as-grown fibres were studied. Undoped samples grow preferentially with a single crystalline monoclinic structure. The fibre with the lowest thulium content (0.1 at%) also shows predominantly a monoclinic phase and no intra-$4f^{12}$ Tm^{3+} recombination was observed. For sample with the highest thulium amount (1.0 at%), the appearance of a dominant triclinic phase as well as intraionic optical activation was observed. The dependence of photoluminescence on excitation energy allows identification of different site locations of Tm^{3+} ions in the lattice. The absence of recombination between the first and the ground-state multiplets as well as the temperature dependence of the observed transitions was justified by an efficient energy transfer between the Tm^{3+} ions. Microwave dielectric properties were investigated using the small perturbation theory. At a frequency of 5 GHz, the undoped material exhibits a dielectric permittivity of 21 and for thulium-doped Ta_2O_5 samples it decreases to 18 for the highest doping concentration. Nevertheless, the dielectric losses maintain a very low value.

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Gallium oxide waveguiding nanowires doped with luminescent elements

K. Lorenz, E. Alves, E. Nogales[1], B. Méndez[1], J. Piqueras[1], J.Á. García[2]

Monoclinic gallium oxide (β-Ga_2O_3) presents the widest band gap among the transparent semiconductor oxides oxides, around 5 eV, while still behaves as a semiconductor due to the presence of a native donor band. Besides, it has a fairly high refractive index in the visible range, about 1.85-1.91, which makes it a promising material for waveguiding purposes.

In this work we study the doping of β-Ga_2O_3 nanowires either by diffusion or ion implantation. The spatial and spectral properties of the luminescence from individual wires was studied both by cathodoluminescence (CL) in a SEM and micro-photoluminescence (micro-PL) in an optical microscope. Luminescence sharp peaks and broad bands from the dopants in the visible and infrared ranges of the spectrum are observed in single nanowires. Homogeneous luminescence yield along the nanowires is shown in the CL images. Waveguiding of the light emitted by the luminescent ions in the nanowires is shown in the micro-PL images. The shape of the characteristic broad red Cr^{3+} luminescence spectrum at room temperature depends on the dimensions of the wires when their width is of the order of the emission wavelength. Luminescence from several rare earth ions has also been obtained and several emitting centers have been identified. Waveguiding of external blue, green and red light which does not excite the luminescent centers was also observed in the nanowires.

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Tuning of oxidation states in the $LaNiO_{3-\gamma}$ perovskite around the insulator-metal transition

N. Franco, R.. M. C. da Silva, B. Berini[1], N. Keller[1], B. Pigeau[1], Y. Dumont[1], E. Popova[1]

The $LaNiO_{3-\gamma}$ perovskite has been studied during its reoxygenation process at the reversible insulator-metal transition by spectroscopic ellipsometry for different pressures and temperatures conditions. First, it was demonstrated that the reoxygenation dynamics increases with both increasing pressure and temperature. Considering the temperature dependent experiments, two regimes of kinetics have been identified: a slow reoxygenation dynamics at low temperature (below 523 K) and fast dynamics above 623 K. Second, contrary to our expectations, the reoxygenation process of a preliminary reduced sample is completed after a sufficient time delay even for the smallest investigated temperature of 473 K or oxygen pressure of 0.03 μbar, respectively. Modeling the change in extinction coefficient as a function of time during the reoxygenation, it was found that the oxygen diffusion coefficient varies from 4.2×10^{-14} to 1.1×10^{-13} cm^2/s depending on the temperature (473 K to 523 K). This oxygen diffusion constant is similar in magnitude order to those observed in the YBCO superconductor. At the light of these investigations, a pulsed oxygen injection was used, allowing a control of the injected oxygen quantity and by consequence, it was possible to precisely tune the oxidation state for a $LaNiO_{3-\gamma}$ film between the reversible reduced sample and the stoichiometric compound. *In situ* ellipsometry measurements are simultaneously performed to follow changes in optical constants.

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Ion-implanted magnetic nanolayers of wide band gap semiconductors for spintronics applications

U. Wahl, E. Alves, R.C. da Silva, J.G. Correia, A.C. Marques, K. Lorenz, L. Pereira[1], J.P. Araújo[1], M.M. Cruz[2], R.P. Borges[2], M. Godinho[2], M. Peres[3], A.M.L. Macatrão[3], T. Monteiro[3]

This project investigates the possibility to fabricate diluted magnetic semiconductors by means of ion-implantation of transition metals or rare earths into single-crystalline starting materials such as the wide band gap semiconductors GaN and ZnO, and some selected semiconducting oxides such as $SrTiO_3$, $BaTiO_3$, $KTaO_3$, and TiO_2. The role of defects created during implantation as a possible source of magnetism or as a prerequisite in modifying the magnetic properties of the implanted impurities is addressed as well.

During this year single crystals of ZnO and $SrTiO_3$ were implanted with the stable isotopes ^{55}Mn, ^{56}Fe, ^{59}Co and ^{58}Ni at various fluences ranging from 1×10^{15} cm^{-2} to 1×10^{17} cm^{-2} and implantation energies of 60 keV or 200 keV. In addition some ZnO samples were implanted with high fluences (>1×10^{17} cm^{-2}) of N or Ar. Following thermal annealing treatments at various temperatures up to 1250°C and the RBS/C results showed that the crystalline structure of 1×10^{16} cm^{-2} or lower fluence implanted ZnO and $SrTiO_3$ (figure) almost fully recovers from implantation damage through annealing in air above 1000°C. The study of the macroscopic magnetic moments of the samples as a function of measurement temperature and applied magnetic field by means of SQUID is currently under way at the Universities of Lisbon and Porto. Preliminary results indicate magnetic moments related to the presence of ferromagnetism in some of the samples, with its intensity depending on the chosen implantation conditions and annealing procedures. Superparamagnetic behavior was observed in high-fluence Ni implanted ZnO indicating the formation of magnetic nano-aggregates, whereas Co and Mn implanted ZnO seems to be mostly paramagnetic. The optical properties of some of the ZnO:Fe samples were characterized using photoluminescence at the University of Aveiro and intraionic Fe^{3+}-related emission found in all cases.

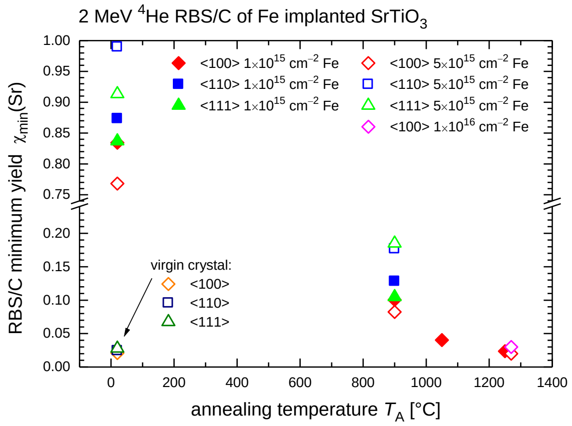

Figure: RBS/C minimum yields from Fe-implanted $SrTiO_3$ single crystals as a function of implanted fluence and annealing temperature (30 min in air).

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Structural and magnetic properties of oxides implanted with transition metals – ZnO implanted with Co, Ni and Mn: influence of the dopant in the magnetic behaviour

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

We extended previous work to the investigation of the behaviour of the transition ions Co, Ni and Mn introduced in ZnO by ion implantation. Single crystals of ZnO were doped with magnetic ions Co, Ni or Mn, using ion implantation with fluences in the range of 1, 2, 5×10^{16} cm^{-2} to 1×10^{17} cm^{-2} and energy of 150 keV. The structural and magnetic properties of such samples were studied after implantation. As-implanted samples present different magnetic behaviours that are related with the atomic concentration of the implanted species: samples with lower concentrations display paramagnetic behaviour while only for the highest implantation fluence superparamagnetic behaviour was observed, indicating formation of nm-sized magnetic aggregates. Study of the behaviour of the implanted ZnO crystals upon thermal treatments is under way in order to understand the role of the dopant and its concentration, in particular the occurrence or absence of magnetic anisotropy in correlation with the orientation of metallic aggregates in the ZnO structure.

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Defect induced magnetism: magnetism in Ar- and N-implanted ZnO and rutile

R.P. Borges[1], M.M. Cruz[1], M. Godinho[1], N. Franco, R.C. da Silva

The finding of ferromagnetic order associated with lattice defects triggered research focused on the role of lattice defects as the source of magnetic moments and their relation to magnetic ordering. Within this context rutile and ZnO single crystals were implanted with Ar or N ions with energy of 100 keV and fluences up to 1×10^{17} cm^{-2} and 2×10^{17} cm^{-2} respectively. In both materials ferromagnetic behaviour was observed at room temperature after implantation.

Although trace amounts of transition metal impurities were identified in the virgin ZnO crystals, it was shown that they are magnetically inert and cannot account for the observed magnetic behaviour. In rutile, no impurities other than the implanted species were detected. Consequently the ferromagnetic behaviour is attributed to defects created during implantation. The ferromagnetic behaviour is suppressed in ZnO after consecutive annealings in air at 400 ºC and 500 ºC. Annealing the Ar-implanted rutile single crystals at 800 ºC induced partial recovery of the lattice structure and a decrease of the measured ferromagnetic moment, while it did not change significantly for rutile implanted with nitrogen. The observed evolution in temperature can be explained by the annealing out of implantation defects, confirming their importance towards the magnetic behaviour of ZnO and rutile. Study of similar systems formed by implantations with 200 keV ions is also under way in order to assess the influence of denser, deeper implantation damage.

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Structural and optical properties of nitrogen doped ZnO films

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Zinc oxide is getting an enormous attention due to its potential applications in a variety of fields such as optoelectronics, spintronics and sensors. The renewed interest in this wide band gap oxide semiconductor relies on its direct high energy gap (E_g~3.437 eV at low temperatures) and large exciton binding energy ZnO. However to reach the stage of device production the difficulty to produce in a reproducible way p-type doping must be overcome.

In this study we discuss the structural and optical properties of ZnO films doped with nitrogen, a potential p-type dopant. The films were deposited by magnetron sputtering using different conditions and substrates. The composition and structural properties of the films was studied combining X-ray diffraction (XRD), Atomic force microscopy, Rutherford backscattering (RBS), and heavy ion elastic recoil detection analysis (HI-ERDA). The results show an improvement of the quality of the films deposited on sapphire with increasing RF power with a preferentially growth along the c-axis. The ERDA analysis reveals the presence of H in the films and a homogeneous composition over the entire thickness. The photoluminescence of annealed samples evidences an improvement on the optical quality as identified by the well structured near band edge recombination.

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Temperature behavior of damage in Sapphire implanted with light ions

E. Alves, C. Marques, G. Sáfrán[1], Carl J. McHargue[2]

In this study we compare and discuss the defect behavior of sapphire single crystals implanted with different fluences (1×10^{16} to 1×10^{17} cm^{-2}) of carbon and nitrogen with 150 keV. The implantation temperatures were RT, 500 ˚C and 1000 ˚C to study the influence of temperature on the defect structures. For all the ions the Rutherford backscattering-channeling (RBS-C) results indicate a surface region with low residual disorder in the Al-sublattice. Near the end of range the channeled spectrum almost reaches the random indicating a high damage level for fluences of 1×10^{17} cm^{-2}. The transmission electron microscopy (TEM) photographs show a layered contrast feature for the C implanted sample where a buried amorphous region is present. For the N implanted sample the Electron Energy Loss Spectroscopy (EELS) elemental mapping give evidence for the presence of a buried damage layer decorated with bubbles. Samples implanted at high temperatures (500 ^{o}C and 1000 ^{o}C) show a strong contrast fluctuation indicating a defective crystalline structure of sapphire.

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Ge-Nanocrystal Formation and Oxide Matrix Evolution during Annealing of LPCVD-SiGeO Films

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Group-IV semiconductor nanocrystals in oxide matrices are important for applications of optoelectronic devices compatible with standard CMOS technology. It has been previously shown that Low Pressure Chemical Vapor Deposition (LPCVD) at low temperatures (400ºC -500ºC) and subsequent annealing of SiO_2:Ge single and multi-layers enables the formation of Ge-nanocrystals that show blue-violet luminescence. The present study focuses on the behavior of oxygen deficient SiGeO layers that may allow the formation of Si and/or SiGe nanoparticles. Layers deposited with various gas flow ratios were annealed at temperatures between 600ºC and 1100ºC to segregate possible excess of Si and Ge in the form of nanoparticles. The composition depth-dependence of the as-deposited oxide layers and its evolution during annealing was investigated by grazing incidence RBS. Further Transmission Electron Microscopy was used in order to study the formation of nanoparticles and complement the RBS results. For low GeH_4:Si_2H_6 flow ratios and deposition temperatures of 450 ºC or lower, the deposited film consists of a SiO_2 matrix incorporating Ge. After annealing of the samples with SiO_2 matrices at temperatures of 600 ºC or higher, quasi-spherical isolated Ge nanocrystals with diameters ranging from 4.5 to 9 nm are formed. In the samples deposited with low flow ratios, the original SiO_2 matrix holds its composition.

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Memory effect on CdSe nanocrystals embedded in SiO2 matrix

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Atom-like energy levels within quantum dots with enhanced interaction of zero-dimensional electron states promises novel quantum devices for future logic and memory applications. CdSe nanocrystals (NCs) have recently attracted enormous attention because of both their light-emitting and charge storage abilities. Memory effects in the electronic transport in CdSe NCs prepared by organic techniques have been observed and characterised. Recently an optical memory effect has been obtained in SiO_2 structures containing CdSe NCs prepared by ion beam synthesis. In this work, we studied CdSe NCs embedded in SiO_2 grown by RF-sputtering technique and post annealed.

A memory effect (flat-band voltage shift) has been first obtained in SiO_2/CdSe NC structures. The composition and the thickness of the CdSe/SiO_2 films were estimated by spectroscopic ellipsometry, Rutherford Backscattering (RBS), and Scanning Electron Microscopy (SEM). The electrical and memory behaviour were studied by capacitance-voltage (C-V) and memory window measurements. Three different samples have been studied: two SiO_2 layers with different CdSe content and a reference sample without CdSe NCs. The structures were grown on p-type Si and electrical contacts were deposited on both sides of the wafers by evaporation of Al. The typical C-V characteristics obtained on samples A, B and C are presented in the figure. Samples with embedded CdSe NCs exhibited memory effect, while no shift of the C-V curve for the SiO_2 reference sample (A) was obtained, a significant shift of the curves on the left during the forward scan was observed for samples B and C. This negative voltage shift due to negative bias (related to the metal electrode) can be attributed to hole injection from the substrate, or to an electron emission from CdSe NCs.

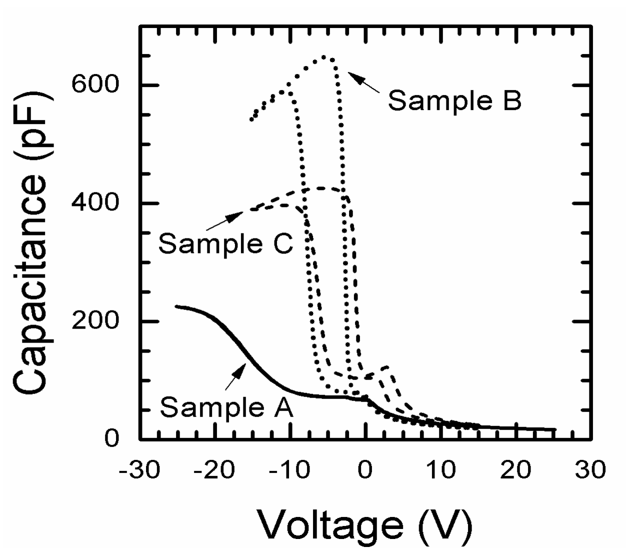

Figure: C-V behaviour of the reference SiO_2 film (A) and with CdSe NCs (B and C).

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Microstructure characterisation of ODS-RAFM steels

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New Reduced Activation Ferritic Martensitic (RAFM) ODS stuls are being developed for fusion reactors study of different RAFM ODS Eurofer 97 batches were preformed with a nuclear microprobe and scanning and transmission electron microscopy. X-ray elemental distribution maps obtained with proton beam scans showed a homogeneous composition of all the batches within the proton beam spatial resolution and, in particular, point to a homogeneous distribution of ODS (yttria) nanoparticles along the Eurofer 97 matrix. This assumption was partially confirmed by transmission electron microscopy, with the observation of the nanoparticles over the entire matrix in one of the samples. Scanning electron microscopy coupled with energy dispersive spectroscopy made evident the occurrence of enriched chromium carbide precipitation. Precipitates occurred preferentially along GBs in three of the batches and have a globular shape on the last one, which points to the existence of different thermo-mechanical histories. Additional electron backscattered diffraction experiments revealed crystalline textures and the ferritic structure of the ODS steel samples.

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Production and Characterisation of Titanium Beryllides with Fine-Grained Structure

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Within the frame of the European Helium Cooled Pebble Bed (HCPB) blanket development, a considerable effort is developed to the qualification of ceramic breeder and beryllium neutron multiplier pebble beds. As the tritium inventory of beryllium pebbles has a main impact on the attractiveness and safety of the entire HCPB blanket, a major goal of the materials development is to maximize the tritium gas release under operating conditions. Preliminary investigations revealed that beryllides like $Be_{12}Ti$ may be much more suitable as neutron multiplier in future fusion power plants compared to pure beryllium. Titanium beryllides promise faster tritium release, much smaller swelling and better compatibility with stainless steel. However, considerable work is still required to develop efficient production methods for beryllide pebbles. During the last 6 years the firm GVT (Goraieb Versuchstechnik), located in Forschungszentrum Karlsruhe (FZK), in collaboration with FZK scientists has carried out a number of manufacturing tests aimed to produce titanium beryllides with fine grains. Microstructural analyses revealed that fabricated specimens consist, mainly, of titanium beryllides and have fine-grained structure.

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Nuclear fusion materials: Deuterium retention in W*

L.C. Alves, N. P. Barradas, G.M. Wright[1], E. Alves

Profiting from its fine thermal properties, W is expected to be used as a plasma-facing component on a Nuclear Fusion Reactor. Besides, during the deuterium(D)-tritium(T) reactor operation, it is desirable that the materials facing the plasma may present low hydrogenic retention. W has been reported as having low hydrogenic solubility but all the experiments have been done in conditions were the ion flux densities are very low when compared to the ones expected in a nuclear fusion reactor. In this work, poly-crystalline samples were exposed in the linear plasma generator Pilot PSI, to high flux densities ($\sim 10^{24}$ $D/m^2.s$) and the final sample D distribution profile measured with nuclear reaction analysis while the total D content compared with thermal desorption spectroscopy. It should be noticed that the nuclear reaction analysis allowed obtaining information on small sample areas (~ 1 mm^2) and then establishing correlation with attained sample surface temperature, measured by pyrometry. The most important result is that W targets, in fact, retain very little D, and that there is no clear dependence with the incident ion fluence. However there are indications that low surface sample temperature (1000K – 1600K was the measured experimental temperature range) may significantly increase the D retention and further work is then needed.

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Structural and mechanical properties of AZOY thin films deposited at room temperature

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Transparent oxide semiconductors have been investigated intensively for basic optoelectronics devices like Thin Film Transistor (TFTs). Integration of oxides with different funcionalities promise new possibilities for technological developments. Transparent conducting Al-doped ZnO films were deposited on glass and electroactive polymer substrates (poly vinylidene fluoride-PVDF), (by dc and pulsed dc magnetron sputtering). The films were deposited at room temperature varying the argon sputtering pressure, after optimizing other processing conditions. All ZnO:Al films are polycrystalline and preferentially oriented along [002] axis. Electrical resistivity around 3.3×10^{-3} □.cm and optical transmittance at 550 nm of ~85% have been obtained in AZOY films deposited on glass while a resistivity of 1.7×10^{-2} □.cm and transmittance at 550 nm of ~70% has been attained in similar coatings on PVDF. Resistivity seems to be strongly influenced by the roughness of the PVDF substrate. As shown in Fig. 6b, the film thickness has influence on both resistivity and transmittance. Increasing film thickness yields a reduction in the film resistivity. This phenomenon is associated with the crystallinity improvement as thickness increases, the concomitant increase of carrier mobility [Aga04] and with the increase of film thickness itself. As reported by other authors, the resistivity of thin films is affected by surface roughness of the substrate. Indeed the high roughness of the substrate leads to a non uniform morphology in the film [For03]. Likewise, high surface roughness increases the multiple reflections, leading to a reduction of the overall transmittance.

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Chemical and electronic structure influence on the electrical behaviour of decorative zirconium oxynitride thin films

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The goal of this study was the investigation of decorative zirconium oxynitride, ZrO_xN_y, thin films prepared by dc reactive magnetron sputtering, using a 17:3 nitrogen to oxygen ratio gas mixture. The colour of the films changed from metallic-like to very bright yellow-pale and golden yellow for low gas mixture flows (from 0 to about 9 sccm) to red-brownish for intermediate gas flows (values up to 12 sccm). With further increase of the reactive gas flow, the samples showed a significant decrease of brightness values and the colour changed from red-brownish to dark blue (samples prepared with 13 and 14 sccm).

The films deposited with gas flows above 14 sccm showed only apparent colorations due to interference effects. This change in optical behaviour from opaque to transparent (characteristic of metallic to insulating-type materials), promoted by the change in gas flows injections, revealed that significant changes were occurring in the films characteristics (namely in the type of bonds and structural arrangements) and thus opening new potential applications for the films, beyond those of purely decorative ones. Taking this into account, the electrical behaviour of the films was investigated as a function of the reactive gas flow and correlated with the observed chemical, electronic and structural features, which could reveal their potential to be used in electrically-based applications. The dependence of the composition with the flow rate was measured by RBS and indicated in the figure.

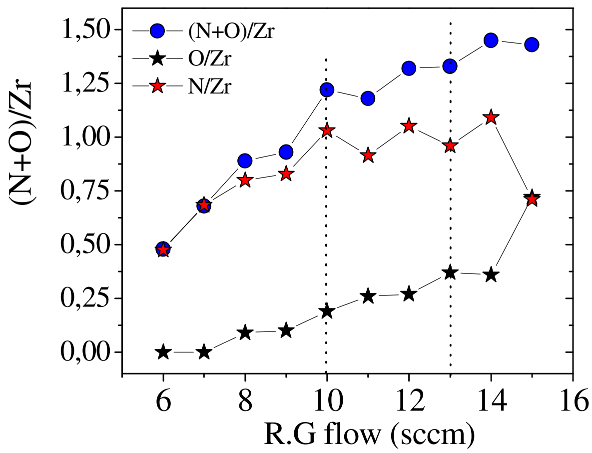

Figure: Composition dependence of the zirconium oxynitride films with the reactive gas flow.

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Structural and optical characterization of Er implanted sapphire

C. Marques, R.C. da Silva, E. Alves

Rare-earth doping of insulating and semiconductor materials is a common method for producing systems with tuned optical properties. The optimum optical performance is highly dependent on the concentration of the doping species as well as on the local environment surrounding these ions. In this work, sapphire crystals were implanted at RT with a low fluence of Er ions, to a maximum concentration of 0.1 at. %. After implantation RBS-C measurements reveal a considerable amount of radiation defects with some Er ions already in regular lattice sites of the host matrix. The optical emission measured by IBIL shows a weak IR line, characteristic of Er transitions. Annealing in air at 1573 K removed the radiation damage almost completely, the fraction of Er ions in regular sites increasing to 50 %. The characteristic emissions are now very intense, with all transitions visible. XRD analysis revealed the presence of Er_2O_3 with <111> parallel to c-axis of sapphire. On the other hand, similar annealings in vacuum produce less extensive crystalline recovery and comparatively lower intensities of the characteristic emissions. The optical absorption spectra at this stage showed what may be a surface plasmon resonance band of Al nano aggregates in the UV region. This may be explained by the lower enthalpy of formation of Er_2O_3 as compared to Al_2O_3, leading to Al dislodging and aggregation.

N-doped photocatalytic titania thin films on active polymer substrates

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Active polymer substrates have found their way in the semiconductor industry as a base layer for flexible electronics, as well as in sensor and actuator applications. The optimum performance of these systems may be affected by dirt adsorbed on its surface, which can also originate mechanisms for the degradation of the polymer. In this collaboration we studied titanium dioxide (titania) semiconductor photocatalytic thin films deposited by unbalanced reactive magnetron sputtering on one of the most applied and investigated electroactive polymer: poly(vinilidene fluoride), PVDF. In order to increase the photocatalytic efficiency of the titania coatings, a reduction of the semiconductor band-gap has been attempted by using a nitrogen doping. Rutherford Backscattering Spectroscopy was used in order to assess the composition of the titania thin films, whereas Heavy Ion Elastic Recoil Detection Analysis provided the evaluation of the doping level of nitrogen. X-ray Photoelectron Spectroscopy provided valuable information about the cation-anion binding within the semiconductor lattice. The photocatalytic performance of the titania films have been characterized by decomposing an organic dye illuminated with combined UV/visible light.

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Characterization of Roman Glasses of historical interest with external PIXE and PIGE

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

Archaeological campaigns in the site of the Roman *villa* of Quinta da Bolacha at Amadora, Portugal, which was discovered in 1979 during prospection of a Roman aqueduct, provided a recollection of many different types of materials and objects. The archaeological works that followed allowed identifying sealed contexts attributed to the 3rd and 4th centuries AD, together with revolved contexts of uncertain dating. In general, the unearthed objects and the available historical information consistently point to the 3rd and 4th centuries AD as the main occupation periods of the *villa*.

Seeking to materially define those moments, glass fragments recovered from the different contexts were analysed non-destructively by ion beam techniques. Because of their poor state of conservation, namely the delamination of the surfaces, the museological objects could not be analysed in vacuum, making the external beam analysis a better and only option for their study. Combination of the PIXE and PIGE techniques in the newly available external microbeam analytical end-station allowed establishing a clear correlation between the composition of the fragments and their contexts of origin. Grouping by similarity of composition further allowed associating some fragments from one of the revolved contexts to well defined ones.

Fig.: Experimental mount and glass fragment sample analysed in the external beam. Coloured dots indicate analysed spots.

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Studies in mineralogy and cultural heritage using focused ion beams*

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In addition to the work performed in the biomedical and material science fields, focused ion beams have been applied in the study of geological/mineralogical and cultural heritage materials, in collaborations with the Univ. of Lisboa, Univ. Nova of Lisboa, Univ. of Évora, Univ. of Minho and INETI.
In the mineralogy field the work involved the characterisation of old mine slag for the evaluation of the amount of valuable metals and also of pyrite ore characterisation in terms of Indium contents and possible type of mineral bearing it.

In the cultural heritage field work, other than the one performed with the external beam, was done in the study of 17th century ceramic wall tiles and of 17th century Arraiolos tapestries. The purpose of this last study was to analyse the tapestry fibres in order to identify the mordants used by the Arraiolos dyers as well as the composition of the different dyes. In what concerns to the ceramic wall tiles, analysis were performed in order to identify the pigments originating the different colours (yellow, blue, white).

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* Projects POCI/CTE/61700/2004 and PTDC/CTE-GIN/67027/2006.
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[2] INETI.;
[3] Univ. of Évora;
[4] Univ. of Minho;
[4] Univ. Nova of Lisboa.

External microbeam line setup*

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

The new external microbeam analytical end-station became fully operational and available on a routine basis. The installation of a laser-assisted target positioning system with micrometer precision stages, of a He flow system for reducing beam scattering and lower energy X-rays attenuation, along with use of a HPGE detector for simultaneous PIGE analysis, extended its capabilities, allowing a more reliable determination of low Z elements, in particular the quantitative determination of Na as a complement to PIXE analysis. This is of particular importance for the analysis of glass matrices as in the case of a number of archaeological Roman glass fragments of historical importance which characterisation could be better established by the experimental determination of the Na contents.
In collaboration with the Restoration and Conservation Department of Universidade Nova de Lisboa, the new external microbeam line setup system was also applied in the study of stained glasses from the Mosteiro da Batalha, both for the characterisation of the bulk material and the identification of the colour pigments and decorations used, allowing to obtain clues concerning the manufacturing processes and techniques. Extending this work to museological and non-museological glasses existing in Portugal and dating from the XV century to the present is under way.

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* Project POCTI/CTM/60685/2004
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Artificial neural networks for real time in-situ RBS analysis

N.P. Barradas, D. Smeets[1], J. Demeulemeester[1], A. Vantomme [1], A. Vieira[2], C. M. Comrie[3], C. Theron[4]

Real time RBS characterization, i.e. performing the analysis during thin film growth has been applied to sputter processes, implantation studies, diffusion experiments and solid phase reactions. In the case of a solid phase reaction for example it has been demonstrated that kinetic parameters such as the apparent activation energy and the diffusion coefficient can be obtained from a single real-time RBS measurement. However, the large number (thousands) of RBS spectra obtained during a typical real-time RBS experiment and the time consuming analysis associated with that has until now obstructed the real breakthrough of this technique. We revived the Artificial Neural Networks work that we had developed years ago, to solve this issue. Entire systems are solved after a few days of work, as opposed to months previously. One paper was published in 2008 in Applied Physics Lettes, and several are under preparation.

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AMS Line Software and Hardware upgrade

N. Franco, H. Luís, E. Alves

Since the recent acquisition, by ITN physics department, of a Tandetron accelerator from the CSIRO lab in Australia, there has been an effort to upgrade and modernize some of the dated features of the system, namely the computer control system as and the hardware controller (DACs) that was by today standards obsolete. So there was a complete replacement of the old Apple based system to a newer PC Windows based system. The control software developed using the LabVIEW programming language was fully rebuild and reviewed from Apple OS, new facilities were introduced like the control of new lines, e.g. ^{14}C line, as well the data analysis. The upgrade of the DACs to a 16 bit resolution and Ethernet based communication allow the improvement of the resolution of the controllers, the addiction of new controllers and the system reliability.

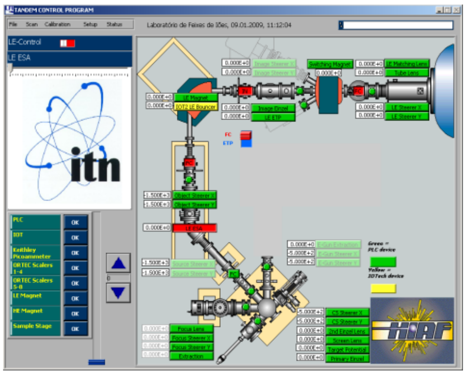

Fig. 1 – Computer interface showing the Control Program.

A new AMS facility installed at the ITN

H. Luís[1,2], N. Franco, L. Gasques[2], M. Fonseca[1,2], A. P. Jesus[1,2], E. Alves[2]

AMS (accelerator Mass spectrometry) is an extremely valuable tool for the detection of certain nuclides and measurement of their concentrations down to levels that can be within the 10^{-12} to 10^{-15} range. This sensitivity makes it popular in many fields of science, such as nuclear physics and astrophysics, geology, archaeology, medicine and several others. In the last year the first results in the micro-AMS system were obtained. The micro-AMS system at the physics unit is based on a 3 MV Tandetron accelerator and has the ability to perform AMS using a micro-beam that allows the study of isotopic ratios within samples with a very high spatial resolution. Its main features are a specially adapted HICONEX source and a fast bouncer system. In the past year the computer control system was updated, and also a lot of work was done in terms of the alignment of the beam transport system, and the resolution of several problems related to the complexity of the system. Several beams have been tested, including carbon, sulphur and gold, however since the ^{32}S beam is the most easy to obtain we have mainly used it in the test of the several components of the system. Some of the test results obtained with sulphur beam is shown in the figure. The system will be fully operational once the instabilities affecting the last section of the beam transport system after the HE Magnet were fixed.

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Advanced data analysis for IBA

N.P. Barradas, M.A. Reis, C. Jeynes[1]

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 other hand to automate the data analysis. In 2008 we continued 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 routinely calculate the influence of the pulse height defect effect in energy spectra. We developed a method to determine non-Rutherford cross sections from simple RBS spectra using Bayesian inference data analysis. Six papers were published in 2008 in international journals.

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