Nuclear Solid State Physics Using Ion Beams

Eduardo Alves

The Nuclear Solid State Group is formed with staff from ITN and the Nuclear Physics Centre of the University of Lisbon and is responsible for the operation of the ITN **Ion Beam Laboratory (IBL)**. The laboratory is equipped with a 2.5 MV Van de Graaff accelerator (able to run up to 3.1 MV) and a 210 kV High fluence Ion Implantor. Two other facilities are installed at ISOLDE/CERN comprising two **emission channelling lines** and one **hyperfine interaction laboratory.**

Major activities of the Group are focussed on the processing and characterisation of advanced materials using ion beam based techniques. The work carried out during the last two decades allowed the group to achieve a large expertise on the field of ion beams. The recognition of the work done is well patented by the large number of National and International collaborations. Most of these collaborations started through bilateral contracts, namely with the universities of Seville, Madrid, Bonn, Knoxville (USA), Budapest, amongst others.

It is also worth to note the number of projects, both European and National, with the participation or leaded by members of the group.

The combination of ion beam and Hyperfine Interaction (HI) techniques provided us with a powerful tool to explore, modify and model the behaviour of new materials. Lately the research activities of the group were centered mainly in two kinds of materials: **Semiconductors and Insulators.** Studies in semiconductors include the doping of ***GaN*** and ***ZnO*** with optically and electrically active ions. These two wide bandgap semiconductors are under intense research all over the world due to the possibility of developing optoelectronic devices working in the visible wavelength range of the electromagnetic spectrum. Our activity aims at the optimization of the implantation conditions of the dopants. Other relevant research work is being carried out in quantum well structures. An intense study of the structural properties of GaN/InGaN structures is under way in collaboration with the University of Aveiro and Strathclyd.

The work in insulators is a continuation of ongoing projects or bilateral collaborations to modify the optical and electrical properties of α**-**$\mathbf{Al_2O_3}$ and **laser materials** (KTP and RTP)**.** Recently a bilateral project was started with the University Carlos III in Spain to study the doping of MgO with transition metals. Besides this, research has also been continued on the **synthesis of new compounds, namely of metastable intermetallic compounds in Al, Ti** and **Mg** based **alloys** using ion implantation. It must be also pointed out that due to the potential of ion beam techniques to study thin films and multilayers important work has been continued in the characterisation of **magnetic thin films for magnetic spin valves and tunnel junctions.** The activity in the technology programme of the **European Fusion Development Agreement** in association with the Centro de Fusão Nuclear of the Instituto Superior Técnico was pursued as a strategic effort to include ITN in the European Fusion activities. Deliverables in several tasks were obtained, namely the study of compatibility of SiC/SiCf with other structural materials, surface studies of Be pebbles and structural studies of the new Eurofer(ODS) steel. Finally, the group has implemented and maintains for 7 years an experimental infraestructure at the **ISOLDE/**CERN radioactive ion beam facility where the Emission Channeling (EC) technique is, applied to studies of lattice site location of impurities in semiconductors and insulators. In addition Perturbed Angular Correlations are used to characterize the charge carrier blocks and charge ordering in High-Tc superconductors and on colossal magnetoresistive oxides. Actually our projects further afford the development of new fast 2D- Si electron PAD detectors to extend the use of EC to probe elements only available by using short-lived isotopes (like ^{27}Mg).

Besides these research activities the group as also strongly engaged in training graduate and undergraduate students, through the supervision and work of M.Sc. and Ph.D. students, Final year degree thesis and Socrates Students as well. In 2003, 44 papers were published in international Journals and 41 accepted for publication. The main achievements of the research developed in 2003 are summarised in the following pages.

Nuclear Solid State Physics Using Ion Beams

Research Team

Researchers(*)

- E. ALVES, Auxiliary researcher
- R.C. da SILVA, Auxiliary researcher
- L.C. ALVES, Research assistant (75%)
- J.G. CORREIA, ITN Contract
- N. BARRADAS, Auxiliary researcher, Reactor (15%)
- A.R. RAMOS Auxiliary researcher, Reactor (15%)
- A. KLING, Auxiliary researcher, Reactor (10%)
- M.R. DA SILVA Auxiliary Prof., IST
- A.A. MELO, Associate Professor [1]
- U. WAHL, FCT Post-Doctoral,
- K. LORENTZ, European Post-Doctoral fellowship

Students

- L. PRUDÊNCIO, Ph.D. student, FCT grant
- JOANA VAZ PINTO, Ph.D. student, FCT grant
- E. RITA, Ph.D. student, Univ. Lisboa, FCT grant
- C.P. MARQUES, Ph.D. student, ITN grant
- A.C. MARQUES, Ph.D. student, FCT grant

Technical Personnel

- J. ROCHA
- F. BAPTISTA
-L. SEQUEIRA

(*) Also members of CFNUL.
[1] Retired from University of Lisbon.

Funding (€)

Research Projects:	76.287,12
Total:	**76.287,12**

Publications

Journals:		44 and 41 in press
Proceedings:		4 and 6 in press
Conf. Communications:		22
Other publications:		2
Theses:	Ph.D.	1

Multiple Quantum Wells

E.Alves, N. Barradas[1], R. Correia[2], S. Pereira[2], N. Franco[3], A. D. Sequeira[3], E. Wengler[4], W. Wesch[4]

Objectives

Among the collaborations with other institutions the ion beam laboratory of ITN runs several projects in the field of multiple quantum wells and quantum dots. The objective of the research at ITN is to provide information like composition, thickness, interface roughness, epitaxy and crystalline quality of the multilayer structures.

Results

The work carried out in this field was concentrated mainly in two systems: Semiconducting InGaN/GaN multilayers and $Ge_{1-x}Si_x$ multilayers and quantum dots. The research work in the GaN/InGaN system is driven by the performance of InGaN-based devices which is responsible for the intensive research developped during the last decade. Our purpose was to study and correlate the composition gradient and strain with the optical properties of the InGaN films. The capabilities of ion beam techniques to provide this information are well establish.

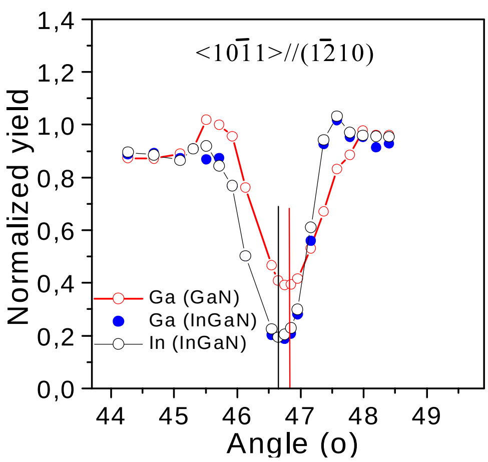

Fig. 1- Angular scan along the tilt <10-11> axis revealing the misalignment between the InGaN film and the GaN substrate.

Our results show a good crystalline quality for the films despite the residual strain figure 1. Moreover a pulling effect was also found associated with the increase of the In concentration.

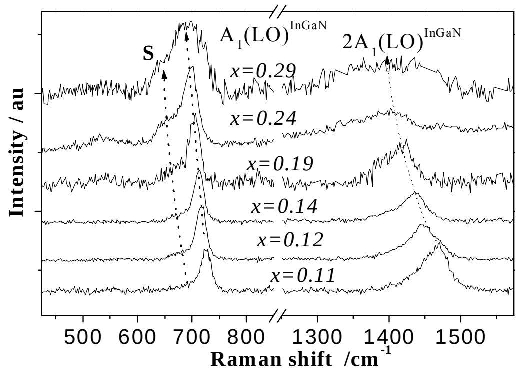

Fig. 2- Raman spectra for InGaN films with different compositions where the Raman shift is visible.

Figure 2 shows the Raman spectra of InGaN/GaN multilayers with different composition as obtained by RBS. A broadening as well as a monotonic shift to lower frequency of the peak assigned to $In_xGa_{1-x}N$ A_1(LO) phonon is clearly observed when x increases. The frequency for the A_1(LO) mode *versus* x for the relaxed samples shows a linear frequency variation as theoretically expected for an one-mode behaviour alloy.

Published, accepted or in press work

1 E. Alves, U. Wahl, R. Correia, S. Pereira, B. De Vries, A. Vantomme, Annealing behavior and lattice site location of Er implanted InGaN, *Nucl. Inst. and Meth.* B206 (2003) 1042-1046

2. M.R. Correia, S. Pereira, E. Pereira, J. Frandon, M. A. Renucci, E. Alves, Raman studies of A_1 (LO) phonon mode on relaxed and pseudomorphic InGaN epilayers, *Appl. Phys. Lett.* 83 (2003) 23.

3. E. Wendler, W. Wesch, E. Alves, A. Kamarou, Comparative study of radiation damage in GaN and InGaN by 400 kev Au implantation, *Nucl. Inst. and Meth. B*

[1] Nuclear reactor group, ITN.
[2] Condensed Matter Physics group, ITN.
[3] Dep. de Física, Universidade de Aveiro 3810-193 Aveiro, Portugal.
[4] Friedrich-Schiller-Universtität Jena, Institut für Festkörperphysik, Jena, Germany

Annealing Studies of Rare-Earth implanted GaN

K. Lorenz, U. Wahl, E. Alves, S. Dalmasso[1], R.W. Martin[1], K.P. O`Donnell[1], T. Wojtowicz[2], P. Ruterana[2], S. Ruffenach[3], O. Briot[3]

Objectives

Optical doping of GaN with rare earth (RE) ions results in light emission spanning the entire visible spectral range and opens the possibility to develop integrated, all-nitride light-emitting devices for several applications in display technology. In the frame of the European Research Training Network project RENiBEl (Rare Earth doped Nitrides for high Brightness Electroluminescent devices) we study RE doping by ion implantation into GaN films. The combination of Rutherford backscattering/channelling (RBS/C) and optical measurements performed at our partner institutions provides a powerful tool allowing determination of the lattice site of the RE ions as well as the level of incorporated damage and the correlation of these structural features with the optical properties of the system. As annealing of GaN is known to be difficult it is important to choose carefully the implantation parameters in order to diminish the damage produced during the implantation.

Results

In this years work we focussed on the implantation of Tm into GaN. Detailed studies of the influence of various implantation parameters such as implantation fluence and temperature on structural and optical properties were performed. Channeling scans along two major axes showed that a large fraction of Tm ions occupies Ga-sites after implantation.

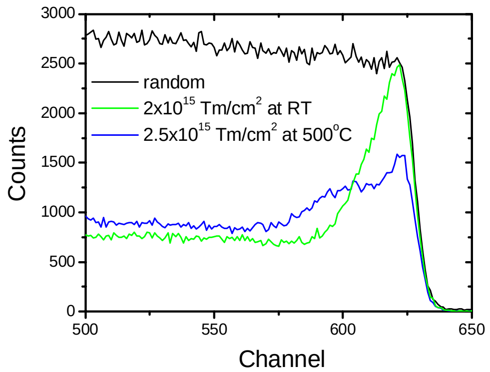

Fig. 1 - RBS/C random and <0001>-aligned spectra of GaN implanted with Tm at RT and at 500 °C.

The damage build-up and the substitutional fraction depend strongly on the implantation conditions. For implantation at room temperature already relatively low implantation fluences lead to an amorphisation of a thin surface layer as could be proved by TEM. RBS/C measurements show that this amorphisation can be inhibited by implanting at elevated temperature as it can be concluded from the RBS/C spectra in figure 1. While for RT implantation the damage peak in the aligned spectrum reaches the random level indicating an amorphisation of the implanted layer, for implantation at 500°C the backscattering yield is significantly reduced. At the same time a broadening of the damage and the Tm profile is observed.

After annealing the samples implanted at higher temperature also show higher substitutional fractions. Directly after implantation the samples do not show any Tm-related luminescence. Annealing results in sharp emission lines in the blue and near infra-red spectral region due to intra-4f transitions of the Tm^{3+} ion as measured with cathodoluminescence at RT (figure2).

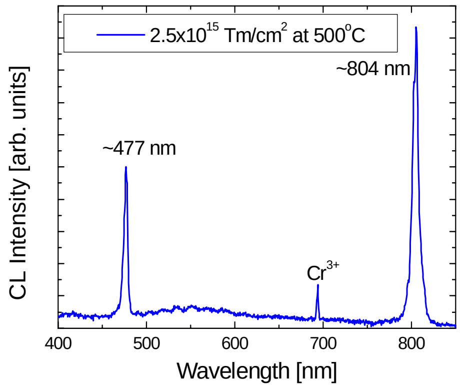

Fig. 2 - CL spectrum of GaN implanted with Tm at 400 °C and annealed for 120 s at 1000 °C.

Published, accepted or in press work

1. K. Lorenz, E. Alves, U. Wahl, T. Monteiro, S. Dalmasso, R.W. Martin, K.P. O`Donnell, R. Vianden, Implantation and amealing studies of tm-implanted GaN, Materials Science & Engineering B 105, Dec 2003.
2. K. Lorenz, U. Wahl, E. Alves, T. Wojtowicz, P. Ruterana, S. Dalmasso, R.W. Martin, K.P. O`Donnell, two papers to be published in the proceedings of the MRS Fall Meeting, Boston, 1-5 December.
3. U. Wahl, E. Alves, K. Lorenz, T. Monteiro, A. Vantomme, B. De Vries, "Lattice location and optical activation of reare-earth implanted GaN", Materials Science & Engineering B 105, Dec 2003.

[1] Department of Physics, University of Strathclyde, Glasgow, G4 0NG, U.K.
[2] LERMAT, FRE 2149, CNRS-ENSICAEN, 1450 Caen, France.
[3] GES, Université de Montpellier II, 34095 Montpellier, France.

Transition Metal Colloids formed by Ion Implantation on Sapphire

C. Marques, E. Alves, R.C. da Silva, N. Franco, A. Sequeira, C. McHargue[1], M. M. Cruz[2], A. L. Stepanov[3]

Objectives

The optical response of clusters reflects their electronic structure, which strongly depends on the particle size and shape, i.e. depends on its morphology. Thus control of the clusters morphology allows tailoring and tuning the optical absorption of the system created. The formation of noble metal nanoclusters in transparent materials is of high technological interest and has long been used to colorize glasses due to the optical resonances in the visible spectrum, related to plasmon resonance of the metal/alloy considered. These linear and also other nonlinear effects can be used in all sorts of optical devices, from switches to real time holography. Our purpose is to create encapsulated dispersions of nanoprecipitates and to characterize both the structure and the optical properties of the matrix-precipitate ystem.

Results

The implantation of high doses (up to 2×10^{17} ions/cm^2) of metals (Cu, Ni, Fe, Co, Ti, Au, Ag, Mn, Co) in sapphire produced buried layers of metallic clusters. RBS-C measurements show that the implanted layer is left in a highly disordered state, whose magnitude depends also on the crystalline orientation of the sample. The encapsulated system obtained is modified after annealing at high temperatures (up to 1573 K), leading to crystalline recovery of the matrix and the formation of oxide phases when annealed in air or a concentrated colloid distribution of metallic clusters when in vacuum. The dimensions and morphology of these nanostructures can be estimated through optical absorption experiments (figure), since those are related to the position and width of the metal surface plasmon resonance bands. It was observed that the matrix crystalline orientation plays also an important role on the shape of the precipitates. PL measurements are under way in order to correlate the emission with the morphology of the precipitates. XRD measurements were also performed to identify the stoichiometry of the metallic or oxide phases.

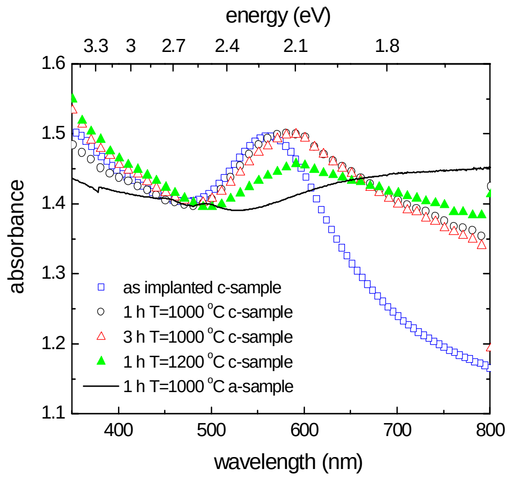

Figure – Optical absorption spectra in the visible range of α-Al_2O_3 samples implanted with 6×10^{16} Au·cm^{-2} and annealed in reducing atmosphere.

Published, accepted or in press work

1. C. J. McHargue, L. C. Ononye, E. Alves, C. Marques, L. F. Allard, "The effect of temperature on the structure of iron-implanted sapphire" accepted Nucl. Inst. and Meth. B.
2. E. Alves, C. Marques, R.C. da Silva, T. Monteiro, J. Soares, C. McHargue, L.C. Ononeye, L.F. Allard, "Structural and optical studies of Co and Ti implanted sapphire", Nucl. Instr. and Meth. B 207 (2003) 55.
3. C. Marques, E. Alves, R. C. da Silva, M. R. Silva, A. L. Stepanov, "Optical changes induced by high fluence implantation of Au ions on sapphire", accepted Nucl. Instr. and Meth. B.
4. E. Alves, C. Marques, L. C. Ononeye and C. McHargue "Defect production on nitrogen implanted sapphire", accepted Nucl. Instr. and Meth. B.

[1]University of Tennessee, Knoxville, USA.
[2]Dep. Física da Faculdade de Ciências da Universidade de Lisboa.
[3]Kazan Physical – Technical Institute, Russian Academy of Sciences, Kazan, Russian Federation.

Buried Nanoprecipitates in Magnesium Oxide

J.V. Pinto, R.C. da Silva, E. Alves, M.M Cruz[1], M. M. Godinho[1], B. Savoini[2], R. Gonzalez[2], B. Fernandes[3], A.D. Sequeira[4]

Objectives

Ion beams are used to modify the sub-surface region of technologically interesting oxide materials, without altering its bulk properties. In particular novel phases, metallic or intermetallic, can be formed buried inside insulating oxides. These layers can be useful for switching, signaling, data transport.

Our work concentrates on the ion beam synthesis of metallic and metal like nanoprecipitates buried in the crystaline refractory oxide MgO, and on the study and characterization of its structural and electromagnetic properties and stability.

Results

***Ion beam synthesis of metallic nanoprecipitates in crystalline* MgO**

High dose implantations of Li, Fe, Co, Ni and Cu ions were carried out in MgO single crystals to promote the formation of metallic precipitates. MgO doped with Ni and Co during crystal growth, that have undergone TCR treatments were also studied for comparison. RBS-C, XRD and optical measurements were used for its characterization. Magnetization measurements M(H) and M(T) were performed in Fe-, Co- and Ni-doped MgO. A superparamagnetic behaviour was found for Co- and Ni-implanted MgO crystals, with a blocking temperature of 12 K in the case of MgO(Co), Fig.1. These findings indicate that Co and Ni clusters form in MgO with dimensions estimated in the range 3-10 nm. Annealings induce further precipitation of the metal ions and growth of the clusters, leading to development of hysterisis in the magnetization curves. Similarly, in MgO:Ni crystals that have undergone TCR at 1600 K, Ni clusters were found with dimensions that reach 200 nm and have well defined orientations with respect to the crystalline host MgO. These distributions of larger nanocrystals display distinctive ferromagnetic behaviour.

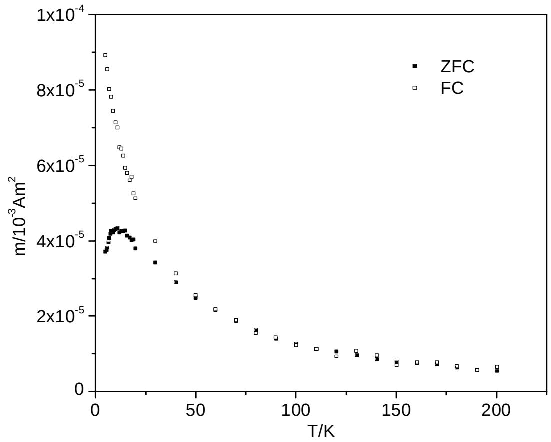

Fig.1: zero field cooled (ZFC) and field cooled (FC) magnetic moment versus temperature for one annealed 250 keV cobalt implanted MgO single crystal.

Published, accepted or in press work

1. E. Alves, R.C. da Silva, B. Savoini, D. Cáceres, I. Vergara, R. González,Y. Chen, Radiation-damage recovery in undoped and oxidized Li doped MgO crystals implanted with lithium ions, *Nucl. Instr. and Meth. B*, 206 (2003) 148-52.

2. B. Savoini, D. Cáceres, I. Vergara, R. González, R.C. da Silva, E. Alves, Y. Chen, Optical and mechanical properties of MgO crystals implanted with lithium ions, *Journal of Applied Physics*, accepted.

3. 3. M.M. Cruz, R.C. da Silva, R. Gonzáles, E. Alves and M. Godinho, Magnetic behaviour of Co and Ni implanted MgO. *Journal of Magnetism and Magn. Materials*, accepted.

4. J.V. Pinto, R.C. da Silva, E. Alves, Y. Chen, M. Tardío, R. Ramírez, R. González, Electrical conductivity of as-grown and oxidized MgO:Li crystals implanted with Li ions, *Nucl. Instr. and Meth. B*, accepted.

5. B. Savoini, D. Cáceres, R. González, J.V. Pinto, R.C. da Silva, E. Alves, Y. Chen, Copper nanocolloids in MgO crystals implanted with Cu ions, *Nucl. Instr. and Meth. B*, accepted.

6. J.V. Pinto, R.C. da Silva, E. Alves, M.J. Soares, T. Monteiro, R. González Stability and optical activity of Er implanted MgO, *Nucl. Instr. and Meth. B*, accepted.

[1] Dep. Física, Faculdade de Ciências da Universidade de Lisboa.
[2] Escuela Politécnica Superior, Universidad Carlos III, Madrid (Spain).
[3] Dep. Materiais, Faculdade de Ciências e Tecnologia da Universidade Nova de Lisboa.
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Study of Single Crystalline Manganite Thin Films and Magnetic Multilayers

E. Alves, C. Marques, N. Barradas, V. S. Amaral[1], J. M. Vieira[2], J. P. Araújo[3], H.-U. Habermeier[4], D. Babonneau[5]

Objectives

The magnetic properties of manganites are related with the manganese valence ratio (+3/+4) which can be modified by doping with divalent ions. For thin films these properties present also a strong dependence on strain and microstructure. One of the main problems in the research field is related with growth of high quality doped and undoped stoichiometric $LaMnO_3$ films. The residual strain and the structural quality of the films as well as the composition are some of the parameters playing an important role (not completely understood) on the electric and magnetic properties. Our goal is to study the influence of these parameters on the magnetic behavior of manganite films.

Also the ordered metallic phases such as FePt and CoPt, with very large magnetocrystalline anisotropy and regarded as attractive candidates for high density of recording media, were studied under this activity.

Results

The films were prepared by excimer laser ablation using ceramic targets and were deposited at substrate temperature 770ºC on single crystalline STO, with thicknesses in the range of 20 to 500 nm. We observed that the crystalline quality is intrinsically related with the stoichiometry and lattice mismatch between the films and the substrate. The growth of defects at the interface is one way to decrease the strain energy due to this lattice mismatch. The strain energy is released through the formation of extended defects (dislocations) which lead to an increase on the minimum yield of the aligned spectrum close to the film/substrate interface. The results obtained also suggest that the extended defects are oriented along the growth direction ([100]). The high level of defects observed in the thicker film (C) is an indication for lower residual strain. The residual strain/structural quality plays an important role on the magnetic transition temperature (figure 1).

We have also studied $C/(FePt/C)_{\times 20}$ multilayers deposited at different temperatures by ion beam sputtering. Using RBS at grazing angles of incidence, we determined the stoichiometry of the nanoparticles and the multilayer periodicity (figure 2).

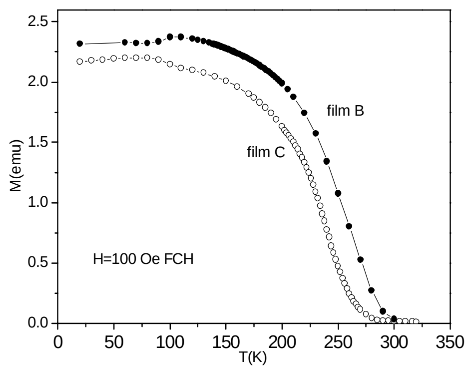

Fig. 1- Temperature dependence of the magnetization of the $LaSrMnO_3$ films; the thinner film (B) presents a higher Curie temperature.

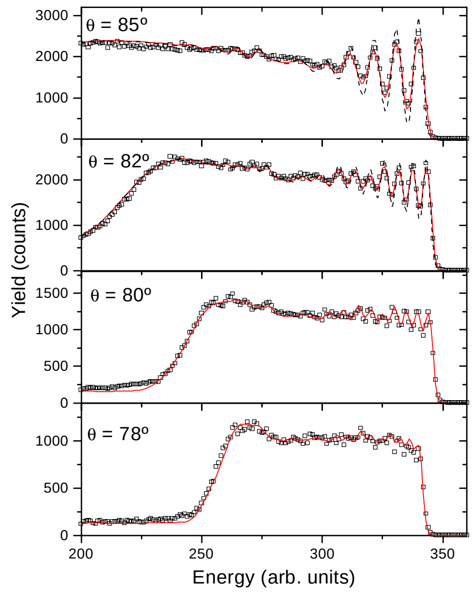

Fig. 2- RBS spectra of a C/(FePt/C)×20 multilayer obtained with different tilt angles.

Published, accepted or in press work

1. E. Alves, C. Marques, V. S. Amaral, J. P. Araújo, G. Cristiani, H.-U. Habermeier, J. M. Vieira, "Ion beam studies of single crystalline manganite thin films", *Nucl. Inst. and Meth. B.*, accepted.
2. N. P. Barradas, E. Alves, D. Babonneau, "Characterization of FePt nanoparticles in FePt/C multilayers", *Nucl. Instr. Meth. B.* accepted.

[1] Dep. de Física and CICECO, Universidade de Aveiro, P-3810-193 Aveiro, Portugal.
[2] Dep. de Eng. Cerâmica e Vidro and CICECO, Universidade de Aveiro, P-3810-193 Aveiro, Portugal.
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[4] Max-Planck Institute fur Festkorperforschung, D-70569 Stutgart, Germany.
[5] Laboratoire de Metallurgie Physique, Université de Poitiers, F-86960 Futuroscope, France.

Contribution of Nuclear Microprobe Analysis for the Characterisation of Nuclear Fusion Reactor Materials and Mineralogical Samples

L. C. Alves, E. Alves, A. Paúl[1], M. R. da Silva, A. La Barbera[2], G. Piazza[3], J. D. Lulewicz[4], J. P. Leal[5], M. C. Freitas[5], A. Mateus[6], J. Figueiras[6], M. Barroso[6], J. Ferreira[7], M. O. Figueiredo[10], J. Mirão[11]

Objectives

Determination of impurity contents on advanced materials for nuclear fusion reactors as well as the study of their behaviour in fusion relevant conditions.

Elemental composition and elemental spatial distribution in minerals for the study of the ore formation history.

Results

Ion beam techniques combined with INAA and ICR allowed to determine impurities in amounts above the radiological desired level. That was the case for Si (>500 μg/g), Co (>33 μg/g) and Nb (7 μg/g) as well as some indication of the presence of undesired elements as U and Ag. Uniform spatial distribution of major constituents was also ascertained.

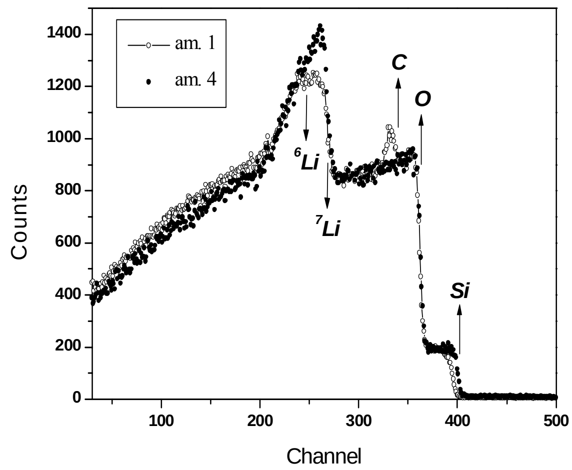

Fig. 1 – RBS spectra from Am 1 (6Li enriched) and Am 4 (natural 6Li enrichment) Li pebbles samples. The existence of the C surface layer referred in the text can also be identified in this figure.

Four different procedures were used to produce orthosilicate pebbles at FZK. The Am 4 sample type revealed that the C compound layer that was found at the surface of all the other analysed samples was successfully removed. The nominal 20% enrichment on ^{6}Li in sample Am 1 was measured to be achieved. These results can be identified in the graph of fig. 1.

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[2] Association EURATOM-ENEA, CR-Frascati, Rome, Italy.
[3] FZK, Forschungszentrum Karlsruhe,Germany.
[4] CEA, Saclay, France.
[5] ITN, Chemistry, Sacavém, Portugal.
[6] CREMINER, FCUL, Portugal.
[7] IGM, Porto, Portugal.

Complementary analysis of tantalates, volcanic glass and sulfosalts were carried out with nuclear microprobe techniques. Results of the elemental spatial distribution on sulfosalts can be seen in fig. 2, and in particular the Ag zonation on the rim of a Ag bearing tetrahedrite.

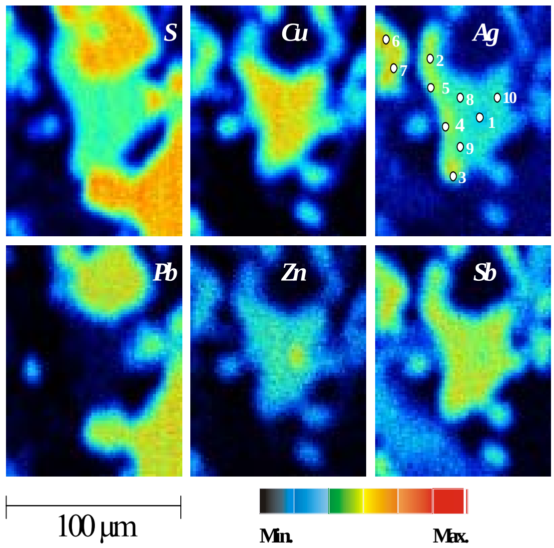


Fig. 2 – Elemental distribution maps in a sample showing tetrahedrite and galena minerals, obtained during a 100×100 μm^2 scan with a 2 MeV proton beam.

Published, accepted or in press work

1. M. Barroso, J. Figueiras, L. C. Alves, A. Mateus, "Chemical Nature of Ag-Bearing Tetrahedrites from the Enfermarias Prospect (Moura, Portugal) Revealed by EPMA and Micro-PIXE Analysis", Proceedings of IV Congresso Ibérico de Geoquímica, Coimbra, July 2003.
2. L. C. Alves, E. Alves, M.R. da Silva, A. Paúl, A. La Barbera, "Li Ceramic Pebbles Chemical Compatibility with Eurofer Samples in Fusion Relevant Conditions", submitted to ICRFM11, Japan, December 2003.

Ion Implantation doping of ZnO

E. Rita, E. Alves, U. Wahl, J.G. Correia, A.M.L. Lopes[1], T, Monteiro[1], J.C. Soares[2],
The ISOLDE collaboration [3]

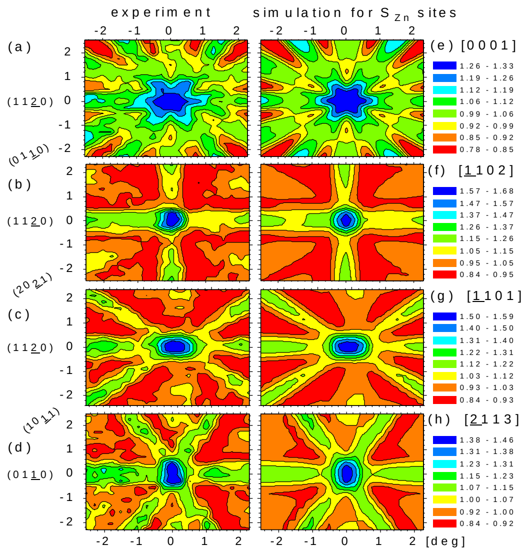
Objectives

This work explores the implantation of possible electrical and optical dopants into ZnO, a wide bandgap II-VI semiconductor of interest in optoelectronics. The implantations and RBS/C studies were carried out at ITN, EC and PAC measurements at CERN/ISOLDE, and the optical studies were done in collaboration with the University of Aveiro.

Results

1. Lattice sites and stability of implanted Cu and Ag

The lattice location of Cu and Ag in single-crystalline zinc oxide was studied by means of the emission channeling (EC) technique. We found that in the as-implanted state Cu and Ag atoms occupy mostly ideal substitutional Zn sites, however, following annealing at 600°C and above Cu and Ag were mainly located on sites that are characterized by large rms displacements (0.3-0.5 Å) from Zn sites.



Figures - (a)-(d): β^- emission channeling patterns from ^{67}Cu-implanted ZnO. (e)-(h): best fits of simulated channeling patterns to the experimental yields, corresponding to ^{67}Cu on ideal S_{Zn} sites.

2.Rare-Earth - Tm implantations

Tm^+ ions were implanted into ZnO and the defect recovery, the Tm lattice location and its optical activation investigated by means of the Rutherford Backscattering/Channeling Spectrometry (RBS/C) and Photoluminescence (PL) techniques. We found that Tm mainly occupies Zn sites and that annealing at 800°C in air maximizes its luminescence. While annealing at higher temperatures improved the ZnO defect recovery, it also lead to Tm segregation to the surface. We have evidence that the optical activation of Tm ions is influenced by the defect density in their environment.

3. Investigation of dopant local environment

Studies of the local environment and point defect annealing after the low dose implantation of ^{111}Ag were carried out with perturbed angular correlation (PAC). Besides the characteristic electrical field gradient (EFG) from Ag on Zn sites, two other EFGs were found, suggesting the presence of specific defects. Further elements investigated in ZnO by this technique are the donor ^{111}In and the acceptor ^{73}As.

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Perturbed Angular Correlations and Emission Channeling at ISOLDE/CERN

J. G. Correia, E. Alves, E. Rita, U. Wahl, A. C. Marques, J. P. Araújo[1], S. Rodrigues[2], S. Dias[2], A. M. L. Lopes[2], J. C. Soares[3], M. R. Silva[3] and the ISOLDE collaboration[4]

Objectives

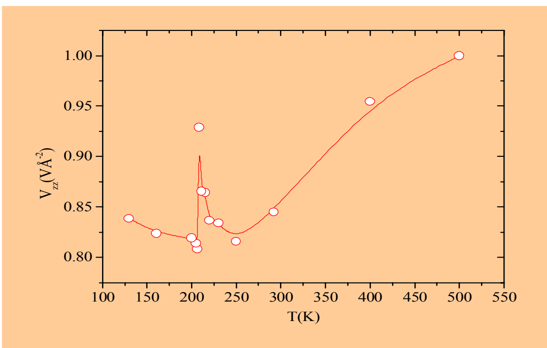
The e^--γ Perturbed Angular Correlations (e^--γ PAC) Emission Channeling (EC) and electron - gamma PAC facility/infrastructure at ISOLDE -CERN has been implemented and is maintained by the nuclear solid State Physics group of ITN and CFNUL. The scientific work is actually centered in three research subjects approved by the ISOLDE Scientific Committee (INTC) *1) IS368 "Lattice Site Location of Transition Metals in Semic. 2) IS390 "Studies of colossal Magnetoresistance oxides with radioactive isotopes, 3) IS360 "Studies of High-Tc Superc. doped with rad. Isotopes"*. A technical development project is further supported by our group on new, fast, self-triggered PAD electron detectors. The aim is to enlarge the number of doping elements to be probed with the EC technique when determining lattice site location of low dose of impurity dopants in materials.

Results

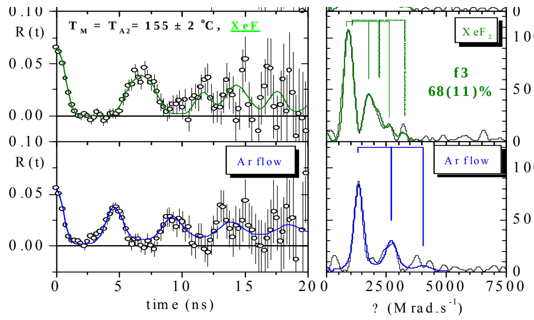
1. IS368 – A new addendum to this proposal has been approved by the INTC in Nov. 2003. Examples of its work in ZnO can be read of this report.

2. IS390 **–** The structural and magnetic environment in manganites is studied with the PAC technique to probe local changes of the charge distribution and magnetic fields. The picture shows the first observation / measurement of a sharp transition on local lattice deformations in the beginning of charge order as a function of temperature in Pr0.65Ca0.35MnO3, in the antiferromagnetic phase.

3. IS360 **–** Perturbed Angular Correlation (PAC) experiments have been performed with ^{199m}Hg, on Hg1201 (n=1) to study comparative doping efficiency of O_δ^{2-} and F_δ^-. The electric field gradients (EFG) at ^{199m}Hg atoms were measured in the presence of



Oxygen introduced under pressure or Fluorine introduced via *soft-chemistry* of XeF_2 mixing. The *principal* component of the EFG, Vzz, and the asymmetry parameter, η, are the source of information for determining the O_δ and F_δ lattice sites and grouping into the Hg-planes. The results for fluorine doping are quite impressive, completely changing the fields as a function of δ_F. At the higher concentrations (δ_F estimated to ~ 40%) an EFG with lower ω_0 = 890 Mrad/s and asymmetry parameters η ~ zero was found. By performing FLAPW first principle calculations of EFGs for several defect configurations, now using internal parameters relaxation methods counterchecked with high quality neutron diffraction data, we have shown this year that the favorite configuration for F to be in, when in high concentrations, is to align along interstitial rows <100> or <010> as shown in the figure.



FLAPW simulated relaxed lattice with shifts amplified x10. EFGs calculated after relaxation.

Published, accepted or in press work

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Magnetic Tunnel Junctions and Spin Valves

N. P. Barradas, E. Alves, S. Cardoso[1], P. P. Freitas[1]

Objectives

ITN has a long-standing collaboration with the INESC magnetic systems group led by Prof. Paulo de Freitas. The role of ITN is to provide structural characterisation of the highly complex advanced magnetic systems produced at INESC. This is a highly interactive collaboration that has proved to be very successful.

Results

This year the work was concentrated in the compositional characterisation of spin tunnel junctions and spin valves [1-2]. These are made by two thin magnetic layers separated by a thin insulating one. In tunnel junctions, the tunnelling current across this barrier layer depends on the relative alignment of the magnetisations of the two magnetic layers. Hence such a device can be used either as a reading head (in which the external field - e.g. from a hard disk - leads to a change in the resistance of the system), or as a non-volatile memory (where the external field forces the direction of the magnetisation).

Determination of the composition of the thin to ultra-thin AlN_xO_y layers used in tunnel junctions is not trivial, and is a challenge to analytical techniques in general. On the one hand, we organised a round robin experiments for that purpose, in which 13 laboratories took part [3]. On the other hand, we developed an artificial neural network algorithm to analyse the RBS data obtained [4].

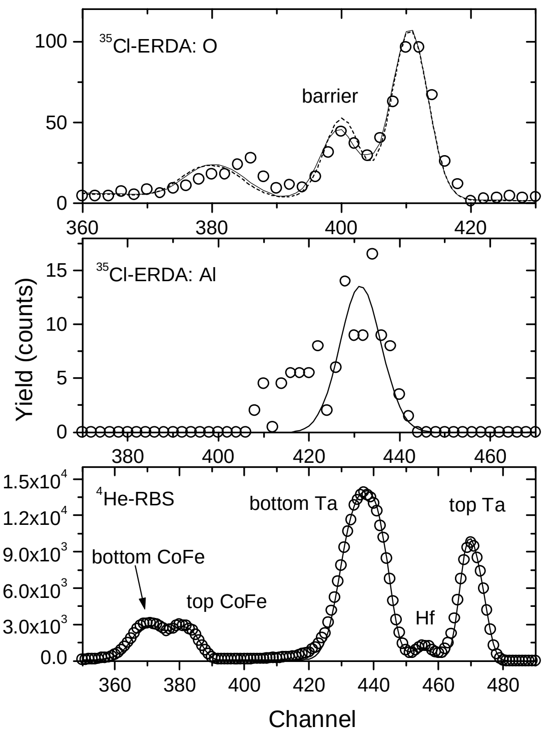

Fig. 1 - Fits (solid lines) and data (circles) obtained for a Si/Ta 70Å/CoFe 35Å/ HfAlOx /CoFe 35Å/Ta 30Å tunnel junction.

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RBS/PIXE analysis of GaInAsSb and GaAlSb films for TPV cells

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Objectives

ThermoPhotoVoltaic (TPV) cells convert the energy from thermal sources into electricity.

For developing higher efficiency cells, ternary and quaternary gallium antimonide based alloys are required, because the suitability of their bad gap.

In this way, films of GaAlSb and GaInAsSb were grown by MOVPE technique on GaSb substrates under different growth conditions in order to obtain the optimization of the growth parameters.

The stoichiometry and thickness of the deposited layers was determined for both systems.

Results

The GaAlSb layers were grown on (100) oriented substrates with off-orientation towards (111)A, (111)B or (110). In figure 1 is shown the RBS spectra for a GaAlSb film. From the fit, the Al concentration was extracted. It was found that the Al incorporation is strongly influenced by the substrate misorientation.

For the GaInAsSb layers a combination of RBS technique and grazing angle of incidence PIXE experiments was needed. Due to the similar atomic weight of the elements (Ga/As and In/Sb) the RBS technique is not able to distinguish the As and In signal when the concentration is very low, as in our case. On the other hand, PIXE is an ideal technique for these conditions, because it is able to detect elements, with high accuracy, even in small concentrations; although it cannot distinguish the depth profile. In this way, the grazing detection condition is used to avoid the detection limits due to the presence of the Sb in the substrate.

Finally, and taking into account the chemistry of the problem, the combination of the areal density data from RBS technique and the As/In concentration from PIXE, will give us the composition and the thickness of the layer $Ga_xIn_{1-x}As_ySb_{1-y}$.

In figure 2 is shown the raw RBS spectra for one of the GaInAsSb layers studied. From the fitting, and tacking into account that the indium replaces the gallium and the arsenic replaces the antimony, the areal density (the number of atomic layers crossed by the He^+ beam) is extracted. From PIXE analysis the As and In concentration is determined.

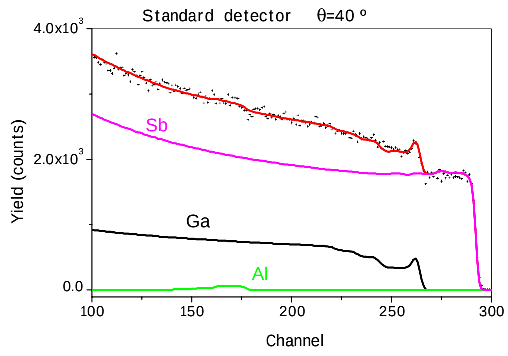

Fig. 1 – RBS spectra taken at 40º scattering angle, fit and individual element contributions with a beam of He at 2 MeV produced in Van de Graff accelerator, for GaAlSb film.

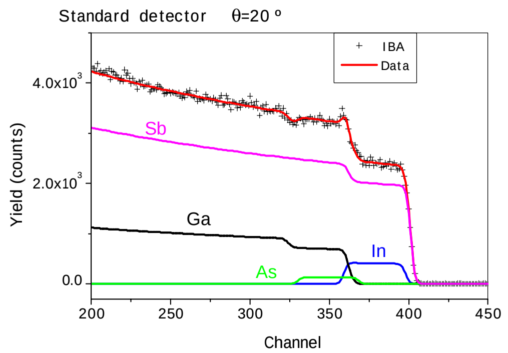

Fig. 2 – RBS spectra taken at 20º scattering angle, fit and individual element contributions with a beam of He at 2 MeV produced in Van de Graff accelerator, for GaInAsSb film.

Dependence of the As and In incorporation in the film with the III/V flow ratio used during the growth was determined.

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[1] FISE, Freiburg, Germany

Dependence of TFT performance on the Dielectric Characteristics

A. R. Ramos, E. Alves, G. Lavareda[1,2], C. Nunes de Carvalho[1,2], E. Fortunato[1], A. Amaral[2,3]

Objectives

The research on amorphous silicon Thin Film Transistors (a-Si:H TFTs) is focused in four key aspects: The reduction of a) leakage current (I_{OFF}) and b) threshold voltage (V_{Th}), c) the increase of the field effect mobility (μ_{FE}) and d) the good stability of devices. To achieve these goals, besides the improvement of the semiconductor layer, it is necessary to obtain a good quality dielectric material. This quality is quantified by the bulk properties such as the resistivity and dielectric constant, but also by the interface dielectric/a-Si:H properties. This work exploits the ternary system (Si, N, C) aiming at the production of a material with good insulating electrical properties and improved interface with amorphous silicon.

Results

The ternary system (Si, C, N) was exploited in order to determine the production conditions which lead to a material suitable for gate dielectric application in TFTs. RBS and ERD measurements were performed to determine the elemental composition of the films.

In the case of silicon nitride, films having N/Si = 1.52 present less incorporated hydrogen and show the highest resistivity and density. The stoichiometric N/Si ratio of 1.33 is achieved for [SiH_4] estimated in the range 21.7–26.8%, having slightly more N–H than Si–H bonds. TFTs with different types of silicon nitride present different characteristics. The highest field effect mobility - 0.36 $cm^2V^{-1}s^{-1}$ - is obtained in TFTs using nitrides with N/Si = 1.52, which corresponds to the lowest hydrogen concentration. TFTs with N-poor nitrides present a strong degradation of their electrical characteristics with voltage application, lowering μ_{FE} from 0.31 to 0.11 $cm^2V^{-1}s^{-1}$ and increasing V_{th} from 2.90 to 9.94V. Their initial values are recovered after a 150ºC–1h annealing. C(V) measurements in MIS (Metal-Insulator-Semiconductor) structures confirm that voltage "stress" induces charge trapping in the nitride/semiconductor interface, for N-poor films. Therefore, the best nitrides for TFT gate dielectric applications are slightly N-rich, containing about 21.5% of hydrogen, bonded preferentially to nitrogen. SiC_XN_Y materials produced with low SiH_4 concentration (20%) presenting resistivities grater than 10^{14} Ω.cm were found to be promising gate dielectrics and were applied to a-Si:H TFT devices.

The performance of these TFTs was compared to those made with standard SiN_X dielectric and also with SiC_X dielectric. Results show that threshold voltage and leakage current decrease with increasing carbon and hydrogen concentrations, while the mobility presents a maximum of 0.43 $cm^2V^{-1}s^{-1}$ for the sample with the composition $SiC_{0.3}N_{1.3}$:$H_{0.93}$, corresponding to gas percentage of SiH_4=20%, CH_4=20% and NH_3=60%. Small amounts of carbon in the SiN_X network can improve the mobility in more than 13%. Carbon-rich SiC_XN_Y presents good off-state characteristics, like low threshold voltage and leakage current, but lower on-state performance due to its lower dielectric constant and bulk resistivity.

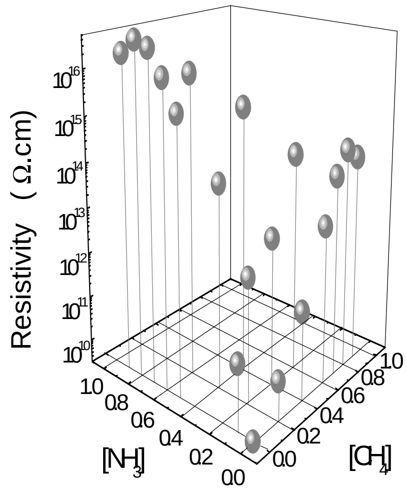

Figure. Resistivity (ρ) of the SiC_XN_Y films as a function of the gas mixture ([NH_3] and [CH_4]).

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[2]Centro de Física Molecular, Complexo I, IST, Av. Rovisco Pais, 1049-001 Lisboa, Portugal.
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Nanocrystalline silicon and silica-titania films doped with Er

E. Alves, , A.R. Ramos, A.C. Marques[1], R.M. Almeida[1], M.F. Cerqueira[2]

Objectives

The photoluminescence (PL) efficiency of Er-doped silica-titania planar waveguides, prepared by sol-gel processing, may be enhanced through the incorporation of neutral Ag^o metal particles. On the other hand, the presence of residual Si-OH groups and the reproducibility of the doping process limit the optical activity of the Er^{3+} ions. In this work, we have used RBS, combined with ERD, in order to study the incorporation behaviour of Ag and H species during the heat treatments used to densify the films.

Other studies in this field were focused on the doping of nanocrystalline Si with Er. The optical activity of Er+ is related with the matrix and concentration.

Results

The results indicate a homogeneous distribution of Er in the as-deposited films. On the contrary, Ag displays a bimodal in-depth profile centred at the film surface and at the film/substrate interface. Annealing at up to 700 °C eventually leads to in-diffusion and concentration of all the Ag species at the latter interface. The hydrogen concentration maintains nearly the same values after annealing at 500 °C and 900 °C, with apparently larger values for temperatures in between. During the heat treatments, the Er profile remains stable. The effect of the heat treatments on the chemical species present in the film deposited on silicon is shown in figure 1.

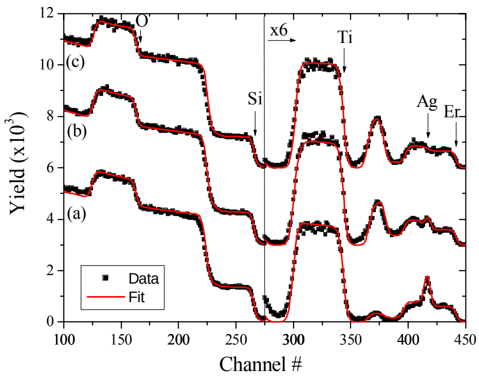

Figure 1. RBS spectra of (a) as deposited sample; (b) 500ºC, 6 min annealed in air; (c) 900ºC, 6 min annealed in air. Spectra obtained with a 2.0 MeV $^4He^+$ beam, tilt angle 45º and scattering angle 180º.

The high mobility of silver and the presence of a significant residual concentration of H in the films could explain the differences in the PL behaviour of waveguides with and without Ag, or with different OH contents. Figure 2 shows the effect of the annealing treatments in the photoluminescence behaviour of Er: the Er^{3+} PL is quenched by the presence of OH groups and the corresponding lifetimes increase with the heat treatment temperature, due to the progressive removal of these species.

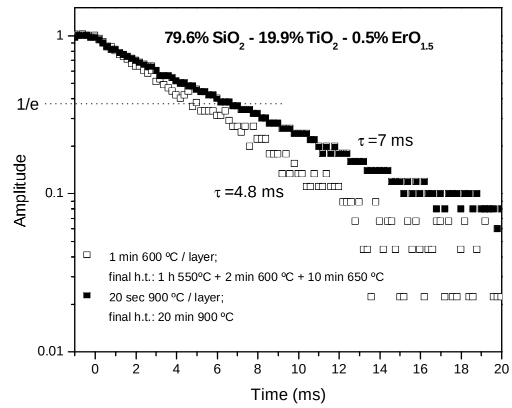

Figure 2. Fluorescence decay of Er^{3+} ions at 1.5 μm, as a function of time, for a 79.6% SiO_2 - 19.9% TiO_2 - 0.5% $ErO_{1.5}$ (mol%) waveguide,

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Effect of an Interfacial Oxide Layer in the Annealing Behaviour of Au/a-Si:H MIS Photodiodes

A. R. Ramos, E. Alves, H. Águas[1], L. Pereira[1], I. Ferreira[1], E. Fortunato[1], R. Martins[1]

Objectives

This work studies the effect of an interfacial silicon oxide layer placed between Au and a-Si:H on the performance of MIS (Metal-Insulator-Semiconductor) photodiodes. We used RBS and SIMS (secondary ion mass spectroscopy), to study the composition changes of the a-Si:H/Au interface, before and after annealing the contact, while SEM (scanning electron microscopy) and AFM (atomic force microscopy) where used to observe the modifications of the surface morphology after de diffusion process.

Results

The results show that in Au/a-Si:H contacts without the presence of an interfacial oxide the Au diffuses into the silicon, even at room temperature, compromising the stability of these contacts (see figure 1). If exposed to O from the air, the a-Si:H oxidises in the regions where the Au diffused and a strong change occurs in the surface morphology with an increase of roughness, as the SEM picture in figure 2 demonstrate. On the other hand, the presence of an interfacial silicon oxide layer of at least 5 Å placed between the Au and the a-Si:H stabilizes the contact by avoiding the Au diffusion, even if the contact is annealed at 150ºC, in which case where we observed an improvement of the rectification nature of the contact.

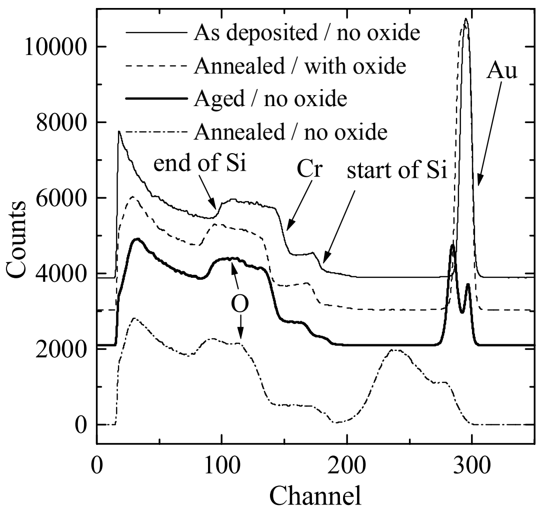

Figure 1. RBS measurements on; as deposited Schottky (without surface oxide) diode; annealed MIS diode; aged Schottky diode; and annealed Schottky diode.

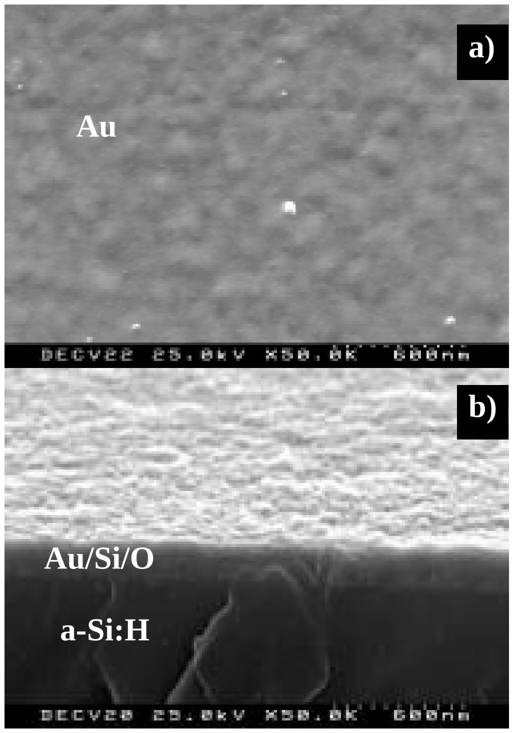

Figure 2. SEM images of: a) Au surface of a MIS diode after annealing; b) surface and cross section of an aged Schottky diode.

Published, accepted or in press work

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Study of Oxynitride Coating Thin Films deposited by Reactive Magnetron Sputtering

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

Objectives

Stoichiometric titanium nitride (TiN) is one of the most important technological coating materials, not only because of its excellent tribological properties but also due to a good chemical stability. It is used in a wide range of applications, which vary from a protective material for machine parts and cutting tools to diffusion barriers in semiconductor technology In this work we exploit the properties of the Ti-N-O ternary system. Both Titanium oxynitride compounds and TiN_x compounds were studied. The properties of TiN_xO_y are related to the oxide/nitride ratio and can be tailored playing with this ratio. We have studied the electrochemical properties of substoichiometric TiN_x coatings aiming at an accurate understanding of its evolution as a function of the N content.

Results

Highly homogeneous TiN_xO_y films were grown by r.f. reactive magnetron sputtering with a reactive gas mixture of N_2 + O_2. The phase composition can be tailored by the substrate bias controlling the oxygen incorporation. It is mainly the oxygen reactivity and kinetic energy of the impinging ions that determine the film properties. Crystalline quality of the films improves significantly when the oxygen concentration is reduced. The successfully and reliable deposition of TiN_xO_y films, using a conventional and not expensive technique, is a step forward to expand their technological applications.

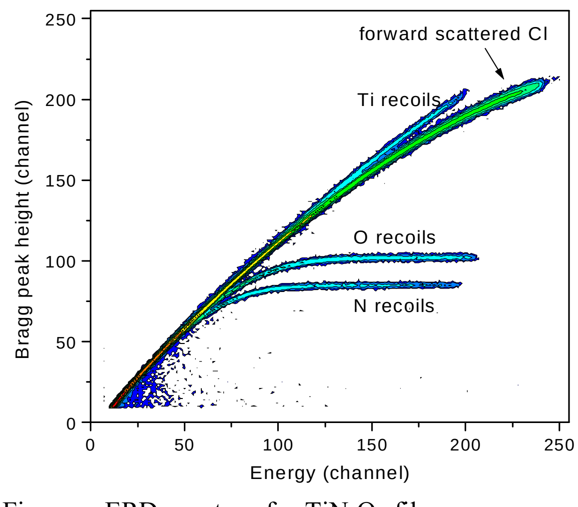

Figure – ERD spectra of a TiN_xO_y films were grown by r.f. reactive magnetron sputtering.

In the TiN_x system best corrosion behaviour was obtained in the TiN films containing low percentages of N, i.e. in which the α-Ti phase is present, characterised by a dense structure. Films near to the stoichiometric condition revealed the lowest corrosion resistance. It was observed that in these samples corrosion is influenced by the characteristics of the columnar structure of the film, which is likely to make the diffusion of the solution species down to the substrate more difficult, when the structure becomes denser as a consequence of the increase in the nitrogen content. Also, the lower thickness of these films, when compared to the low nitrogen containing films, may explain the relatively less protective character of these coatings. A relatively high corrosion resistance was found in the 30 at.% N sample, which was attributed to the fine-grained and compact structure characteristic of this sample. In addition, the 30 at.% N film presents a very high hardness, which makes it a promising solution for situations in which high corrosion and wear resistances are required.

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Advanced Data Analysis for IBA

N. P. Barradas, A. Vieira[1]

Objectives

Ion Beam Analysis (IBA) is a cluster of techniques dedicated to the analysis of materials. Data analysis is normally done in an interactive way, requiring a dedicated expert. Our goal is to automate IBA data analysis.

Results

We have previously developed a code, the IBA DataFurnace, that uses the simulated annealing algorithm to automatically analyse IBA data. A review of its features was now published [1]. We also published a review of the simulated annealing, Bayesian inference and artificial neural networks (ANN) algorithms [2]

We also developed a new and more efficient ANN algorithm to classify complex data [3]. We developed ANNs both to analyse data as such, extracting the relevant desired parameters in an instantaneous way [4,5] and in a code suitable for automated control of an experimental setup, given an interface to the relevant hardware [6].

We recently developed an algorithm to include different models of roughness in RBS data analysis in a fast way. We now investigated the limits of validity and application of the models and methods, and applied them to specific cases [7,8].

We used Bayesian inference to determine the stopping power of ^{4}He in Al_2O_3 [9].

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Ion Beam Modification and Analysis of Oxide Laser Materials

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Objectives

The dopants introduced into laser materials dominate their optical properties. Ion beams provide powerful tools to study the properties of materials by means of Rutherford backscattering (RBS), Particle Induced X-Ray emission (PIXE), Nuclear Reaction Analysis (NRA) and channeling. On the other hand ion implantation can be used to engineer the defect structure of materials or to create new kinds of materials. Combining these techniques with photo-luminescence and absorption spectroscopy provides deep inside into relevant properties of laser materials.

Results

Samples of bismuth tellurite were implanted with 800 keV Au^+ and 450 keV Ag^+ ions to fluences between $5\times10^{15}cm^{-2}$ and $2.5\times10^{16}cm^{-2}$ at room temperature. The samples were annealed at temperatures of 600ºC for Au, 450ºC for Ag and 500ºC for Au/Ag implanted crystals. Strong optical absorption maxima at about 630 nm (Au) and at about 570 nm (Ag) indicate the formation of noble metal clusters. For Au and Ag co-implanted samples the optical absorption shows a complex structure with a peak around 600 nm which is associated with mixed Au/Ag clusters [1].

Single crystalline samples of potassium titanyl phosphate (KTP) and rubidium titanyl phosphate (RTP) were implanted with 150 keV Er^+ ions to a fluence of $5\times10^{15}cm^{-2}$ at room temperature. The samples were subjected to annealings at a temperature of 640ºC. Photoluminescence (PL) studies were performed at 10 K revealing that Er centers in recrystallized KTP are similar to those observed in KTP:Er single crystals. In the case of RTP a Rb loss is observed and the PL seems to be due to the overlapping emissions of Er^{3+} like in bulk RTP:Er crystals and t in a Rb-deficient phase ($ErPO_4$) [2].

$NaBi(WO_4)_2$, $NaBi(MoO_4)_2$ and $LiBi(MoO_4)_2$ single crystals grown by the Czochralski technique have been doped with Pr^{3+}. The exact Pr concentrations in the crystals, which are essential for the interpretation of the optical properties were determined by PIXE. The investigation of the fluorescence dynamics confirmed the assumption that these laser hosts are disordered materials, i.e., the Na and Bi exchange part of their nominal lattice sites [3]. Studies on the related $LiYb(MoO_4)_2$ laser host using NRA for the detection of Li showed that crystals were grown with the correct lithium-rich stoichiometry (Fig. 1).

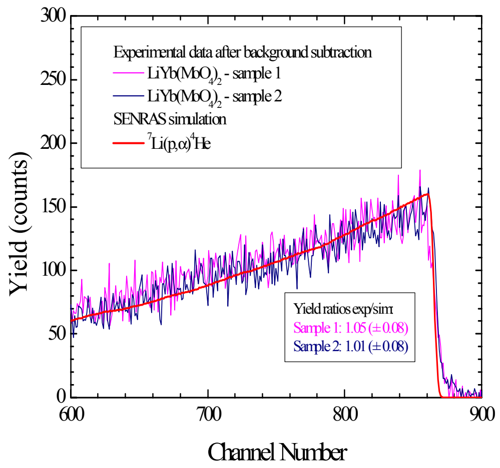

Fig. 1 - Spectrum of the α-particles released in the $^7Li(p,\alpha)^4He$ reaction for1.8 MeV protons impinging on $LiYb(MoO_4)_2$ and the corresponding SENRAS simulation.

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Laboratory Operation and Development

M. Ribeiro da Silva

New RBS two axis goniometer

Some instabilities were detected in the data acquisition and control of the old goniometer installed in the Van de Graaff accelerator RBS line. A complete new two axis goniometer has designed and built (Fig. 1).

This new device intended for general RBS channelling analysis is also optimised for on-line sample implantation in conjunction with the ITN-Physics high fluency Ion Implanter. It can accept different sample mountings including temperature controlled stages from –30° to 500°C in continuous operation. The operation of the goniometer can be full automatized with the angular displacements assured by computer driven step motors and the absolute and relative positioning controlled with the help of optical encoders.

Fig. 1 – RBS two axis goniometer

Unit main parameters and characteristics: Effective sample area up to 15x15 mm^2; Angular = 0,04°; □= 0,02° and. □resolution: Maximum sample viewing angle: front side 70°, back side 50°.
The maximum obtainable vacuum is better than 1.10^{-6} mbar due to an extensive clean construction with a maximum use of stainless steel and ceramic parts.

High pressure and temperature tube furnace

A new versatile general purpose tubular furnace was developed and assembled (Fig. 2).

This unit presents the following parameters: maximum operation temperature 1100°C, controlled by Pt thermocouple coupled to a high level PID process controller; maximum sample dimensions: 30 mm long, 18 mm diameter cylinder of solid or powder sample; the furnace can work with an atmosphere ranging from HVAC to a pressure of 5 bar of inert or slight aggressive gas.

In the Fig. 2 we can find a general view of the setup.

LN_2 distribution

The LN_2 distribution in the Van de Graaff accelerator and Vacuum Test Laboratory area was improved with the acquisition of large capacity mobile dewars.

Also a new general purpose liquid gas dipstick based on LED operation, was developed and assembled.

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