Nuclear Solid State Physics Using Ion Beams

Introduction

The activity developed in this area is essentially fundamental research in advanced materials carried out with a team formed with staff from ITN and from the Nuclear Research Centre of Lisbon University.

The main facilities are the Ion Beam Laboratory located in Sacavém and the two hyperfine interactions laboratories located respectively in Lisbon and in ISOLDE/CERN.

Considerable collaboration exists with national and international research groups through bilateral contracts and European programs. In the last years with the installation of the ion implantor facility a big increment of the research in the processing of new materials occurred. In this field falls the formation of silicides by high dose ion implantation currently in progress.

The most relevant results obtained during 1997 are the following:

- The observed magnetoresistance behaviour of granular thin films prepared by high fluence Fe and Co implantation into Ag thin films (~2000 Å). The best value of the magnetoresistance obtained, so far, is 9% at 10 K and 1.5 % at room temperature for a film implanted with Co at a fluence of 8×10^{16} at/cm^2 and annealed at 620 K.
- The study of high T_c superconductores implanted at CERN with Hg^{197m}. An increase in T_c was observed following the characteristic half-life of the transmutation of Hg^{197} to Au^{197}, even with an implanted dose as low as 10^{13} Hg/cm^2.
- The upgrading of the Ion Beam Laboratory with the acquisition and construction of a high energy ion microprobe, to be installed in one of the Van de Graaff beam lines.

The main output are the publications in international journals and the training of MSc and PhD students.

Research Team

Researchers –	11	*	(11 PhD)
Research Students –	7		
Undergraduate Students –	6		
Technicians –	2		

* 3 from university, 4 post-doctoral and 1 pos-doctoral from RPI.

Publications

Journals –	24	+ 30 in press
Proceedings –	7	
Special Publ. –	2	
Conf. Commun.:	4	
Theses:		
Lic. –	2	

10^3 PTE

Expenditure:	**5.768**
Missions:	104
Others Expenses:	2.888
Hardware & Software:	424
Other Equipment:	2.352

10^3 PTE

Funding:		**11.916**
OE/ITN	OF	148[(1)]
External Projects:	1997	9.950
Others		1.818

[(1)] This cost will be covered by external funding

INSULATORS

Nonaxial sites for Er in $LiNbO_3$

L. Rebouta[1], M.F. da Silva[2], J.C. Soares[2,3], D. Serrano[4], E. Diéguez[4], F. Agulló-López[4], J. Tornero[5]

[1] *Univ Minho, Dept Fis, Largo Do Paco, P-4710 Braga, PORTUGAL*

[2] *Instituto Tecnológico e Nuclear, Estrada Nacional 10, 2685 Sacavém, Portugal*

[3] *Centro de Física Nuclear, Universidade de Lisboa, 1699 Lisboa, Portugal*

[4] *Dep. Física de Materiales, Universidad Autónoma de Madrid, Cantoblanco, 28049 Madrid, Spain*

[5] *Dep. de Química Física, Universidad Autónoma de Madrid, Cantoblanco, 28049 Madrid, Spain*

Abstract

Off-axis sites for Er have been identified and investigated in congruent $LiNbO_3$ by ion-beam/channeling techniques. They do not constitute a different crystalline phase and appear to be associated to clusters or amorphous precipitates. The axial Er ions lie at the Li octahedron hut shifted about 0.2 Å from the regular lattice site. Their concentration increases with total Er doping up to saturation level of about 2%.

Applied Physics Letters **70** (1997) 1070-1072.

Diffusion of Nd, Er and Nb in potassium titanyl phosphate

M.J. Martin[1], C. Zaldo[1], M.F. da Silva[2], J.C. Soares[2,3], F. Diaz[4] and M. Aguiló[4]

[1] *Instituto de Ciencia de Materiales de Madrid, Consejo Superior de Investigaciones Científicas, Cantoblanco, 28049 Madrid, Spain*

[2] *Instituto Tecnológico e Nuclear, Estrada Nacional 10, 2685 Sacavém, Portugal*

[3] *Centro de Física Nuclear, Universidade de Lisboa, 1699 Lisboa, Portugal*

[4] *Laboratori de Física Aplicada I Cristalografia, Universitat Rovira I Virgili, 43005 Tarragona, Spain*

Abstract

The concentration profiles of neodymium, erbium and niobium thermally diffused into (001) $KTiOPO_4$ single crystals have been investigated by the Rutherford backscattering spectroscopy. The activation energy E_A, and the pre-exponential factor, D_0, obtained for Nd, Er and Nb are $E_{A,Nd}$ = 5.3 eV, $D_{0,Nd} = 1.3 \times 10^{-11}$ cm^2 s^{-1}; $E_{A,Er}$ = 6.4 eV, $D_{0,Er} = 3.6 \times 10^{-16}$ cm^2 s^{-1} and $E_{A,Nb}$ = 3.8 eV, $D_{0,Nb} = 1.2 \times 10^{-14}$ cm^2 s^{-1}.

Journal of Physics - Condensed Matter **9** (1997) L465-L469.

Chemical Effects on the Amorphization of Sapphire

E. Alves[1], M.F. da Silva[1], J.G. Marques[2], J.C. Soares[1,2] and K. Freitag[3]
[1] *Instituto Tecnológico e Nuclear, Estrada Nacional 10, P-2685 Sacavém, Portugal*
[2] *CFNUL, Univ. Lisboa, Av. Prof. Gama Pinto 2, P-1699 Lisboa, Portugal*
[3] *ISKP, Univ. Bonn, Nussallee Str, D-53115 Bonn, Germany*

Abstract
α-Al_2O_3 single crystals were implanted with similar doses of Pt and Hg ions at room temperature. Although the masses of the implanted species are very close, the observed defect production and annealing behaviour are quite different. In the case of Hg, a fluence of $1x10^{15}$ at/cm^2 fully amorphizes the implanted region and the epitaxial regrowth occurs at low temperature (800^{o}C) with a velocity higher than 52 Å/min. In the case of Pt the same fluence only produces a buried damage layer near the end of the range. The amorphous state is reached after the implantation of $1x10^{16}$ at/cm^2 and the epitaxial regrowth occurs at high temperatures with a velocity of 3 Å/min at 1100^{o}C. After implantation 70% of the Pt ions are in substitutional lattice sites while only 30 % of the Hg occupy regular sites in a displaced octahedral position of the lattice. Hyperfine interaction measurements indicate that Hg can be associated with oxygen forming Hg-O complexes.

Nucl. Inst. and Meth. B (in press).

Lattice Site Location and Annealing Behaviour of W Implanted TiO_2

R.C. Silva[1], E. Alves[1], L.M. Redondo[1], R. Fromknecht[2] and O. Mayer[2]
[1] *ITN, E.N. 10 , 2685 Sacavém, Portugal*
[2] *Institut für Festkörperphysik, P.O.B. 3640, D-76021 Kalsruhe, Germany*

Abstract
The implantation damage and lattice site location of W in TiO_2(rutile) was studied using the Rutherford backscattering technique in the channeling mode. The W ions were implanted at room temperature with fluences in the range of 10^{15} cm^{-2} to 10^{17} cm^{-2} into both <100> and <001> oriented single crystals. The implanted region becomes completely disordered for W doses of 6×10^{15}cm^{-2}. After annealing experiments at temperatures up to 1100 K the results suggest that lattice recovery behave differently, according to the type of TiO_2 single crystal used. While in <001> oriented single crystals partial epitaxial like regrowth of the damaged region is seen, for <100> oriented single crystals a recrystallization process occurs almost to completion. During the damage recovery the samples implanted with higher doses of W loose more than 80% of the W from the implanted region. However, in both cases the W profiles show a double peak structure being one peak at the surface. Detailed angular scans for the main axial directions show that in the case of full recovery more than 80% of the W remaining in the bulk are incorporated in Ti lattice sites. These observations suggest that a strong anisotropy of the lattice recovery and transport properties in W implanted TiO_2. The findings reported here will be correlated with results of the electrical conductivity measurements.

Nucl. Inst. and Meth. B (in press).

Solubility and Damage Annealing of Er Implanted Single Crystalline α-Al_2O_3

E.Alves[1], R.C. Silva[1], M.F. da Silva[1], J.C.Soares[1,2], G.van den Hoven[3], A.Polman[3]
[1] *Instituto Tecnológico e Nuclear (ITN), EN 10, 2685 Sacavém, Portugal;*
[2] *Centro de Física Nuclear da Univ. de Lisboa, Av. Prof.Gama Pinto 2, 1699 Lisboa, Portugal*
[3] *FOM, Inst. for Atomic and Molecular Phys., Kruislaan 407, 1098 SJ Amsterdam, The Netherlands*

Abstract
Er implantation into c-cut sapphire (α-Al_2O_3) was studied using ion beam techniques. Er^+ ions were implanted at room (RT) and liquid nitrogen (LN) temperatures with an energy of 800 keV and fluences in the range of 10^{13} to 10^{16} Er/cm^2. At LN temperatures doses above $3x10^{14}$ Er/cm^2 create a continuous amorphous layer through all the implanted region. The epitaxial regrowth of this layer at 1400ºC leaves a defect free region and is accompanied by the segregation of a great amount of Er to the surface. The retained Er was randomly incorporated in the α-Al_2O_3 lattice, reaching a maximum concentration of $4x10^{19}$cm^{-3}. The implantation of $2x10^{16}$ Er/cm^2 at RT only produces a buried amorphous layer near the end of the range. The near surface region, although heavily damage, remains crystalline. The buried amorphous layer shows an epitaxial recrystallization and some residual defects remain in the region where the Er profile is located. The energy dependence of the dechanneling rate gives an indication that these defects are dislocations. In this case the recrystallization process causes the narrowing of the Er profile with the diffusion of a small amount to the surface. Detailed angular scans along the <0001> and <02 $\bar{2}$ 1> axes show the presence of c-axis aligned Er precipitates.

Nucl. Inst. and Meth. B (in press).

Platinum Implanted Lithium Niobate - Annealing Behavior and Dopant Redistribution

A. Kling[1], J.C. Soares[1,2], M.F. da Silva[2]
[1] *Centro de Física Nuclear, Universidade de Lisboa, P-1699 Lisboa Codex, Portugal*
[2] *Instituto Tecnológico e Nuclear, P-2685 Sacavém, Portugal*

Abstract
The recrystallization and dopant redistribution of $LiNbO_3$ implanted with 50 keV Pt^+ ions to a fluence of 5×10^{16}cm^2 at room temperature was studied using RBS/channeling and optical absorption measurements. It was found that the redistribution mechanisms for the Pt implant strongly depend on the annealing temperature and duration as well as on the thermal history of the sample. Segregation of Pt was detected for annealing at 600ºC (independent of the annealing time) and at 800ºC (for annealing times up to 4 h), while diffusion was observed for longer annealing times at 800ºC and at 1000ºC. The Pt diffusion was strongly hampered in the case of the incrementally annealed sample. RBS/channeling and optical absorption measurements suggest the existence of Pt aggregates. The $LiNbO_3$ host lattice recovered partly during annealing at temperatures up to 800ºC, while full recovery is achieved only at after annealing at 1000ºC and is accompanied by a drastic reduction of the Pt concentration.

Nucl. Inst. and Meth. B (in press).

Lattice Site Determination of Cr in Low Doped Lithium Niobate Single Crystals Using PIXE/Channeling

A. Kling[1], J.C. Soares[1,2], M.F. da Silva[2], J.A. Sanz-García[3], E. Diéguez[3], F. Agulló-López[3]
[1] *Centro de Física Nuclear da Universidade de Lisboa, P-1699 Lisboa Codex, Portugal*
[2] *Instituto Tecnológico e Nuclear, P-2685 Sacavém, Portugal*
[3] *Departamento de Materiales, C-IV, Universidad Autónoma, E-28049 Madrid, Spain*

Abstract
The lattice sites of Cr have been determined in $LiNbO_3$:Cr doped with 0.1 mol% Cr^{3+} as well as in $LiNbO_3$:Cr,Mg doped with 0.1 mol% Cr^{3+} and 6.0 mol% MgO in melt using PIXE/channeling. In the case of $LiNbO_3$:Cr for the first time the occupation of both regular cation sites (60% on Li sites and 40% on Nb sites) has been observed for a trivalent impurity. For $LiNbO_3$:Cr,Mg a significant reduced fraction of Cr (20%) is located on Li sites. The major fraction (80%) occupies an interstitial site shifted 0.1 Å from the regular Nb site towards the octahedron.

Nucl. Inst. and Meth. B (in press).

Influence of the Sputtering Parameters on the Properties of Al_2O_3 and AlN Insulators in Spin Tunneling Junctions

T.S. Plaskett[a], P.P. Freitas[a,b], J.J. Sun[a], R.C. Sousa[a], F.F. da Silva[a], T.T.P. Galvão[a], N.M. Minho[a], S. Cardoso[a,c], J.C. Soares[c,d]
[a] *Instituto de Engenharia de Sistemas e Computadores, R..Alves Redol 9, 1000 Lisboa, Portugal*
[b] *Instituto Superior Técnico (IST), Dept. of Physics, Av. Rovisco Pais, 1096 Lisboa, Portugal*
[c] *Instituto Tecnológico e Nuclear (ITN), Estrada nacional 10, 2685 Sacavém, Portugal*
[d] *Centro de Física Nuclear da Univ.Lisboa (CFNUL), Av. Prof. Gama Pinto 2, 1699 Lisboa, Portugal*

Abstract
He role of the Sputtering parameters on the properties of thin Al_2O_3 and AlN insulator layers for spin dependent tunneling junctions was studied. Al_2O_3 insulators were prepared by rf sputtering from an Al_2O_3 target and also by plasma oxidation in an O_2 atmosphere of a thin Al layer. The thin Al layer was deposited by rf sputtering at room temperature. AlN was prepared by reactive sputtering from an Al target in a Ar-N_2 atmosphere. The index of refraction n measured by ellipsometry, was used to optimize the sputtering parameters, and chemical etch rates were used for information on pin-hole density. The aimed values were n=1.75 for Al_2O_3 (corundum structure) and n=2.2 for AlN. For reactively sputtered Al_2O_3, under optimized deposition conditions, n reaches 1.65 for films 300 Å thick. Al_2O_3 thin layers fabricated by plasma oxidation of a thin Al film were also prepared but the oxide layer was too thin for ellipsometry characterization. AlN insulating films show a constant n value of 1.92 from 400 Å down to 60 Å thick films. Rutherford backscattering analysis show that the ratio of Ar to N_2 must be less than 1 to avoid O_2 incorporation in the film. Junctions of the type glass/FM/insulator/FM, where FM is either Co or NiFe, were made by contact metal masks with a junction area of 0.25 mm^2. The Al_2O_3 deposited by sputtering from an Al_2O_3 target under conditions expected to give n>1.5 had a magnetoresistance at room temperature MR(RT) of 2.5 % with a junction resistance R_j of 2.2 MΩ and by fitting the I-V curve to existing tunneling theory gave an effective barrier thickness t_j of 20 Å and a barrier height Φof 2.2 eV. These occurred in only one sample (9 junctions) and were no reproducible. The Al_2O_3 insulators prepared by plasma oxidation of a thin Al layer gave a MR(RT) = 4%, t_j = 22 Å, R_j = 250 Ω and Φ = 1.4 eV. The AlN insulator with n = 1.9 gave a MR(RT) = 1.6 %, t_j = 20 Å, R_j = 4.8 Ω and barrier height 1.2 eV. In the last two cases junctions with MR at room temperature can be produced with reasonable reproducibility. Al_2O_3 samples showed a 15-20 % increase in MR as the temperature was lowered to 17 K, while AlN junctions show an increase of a factor of 2 in MR at 77 K.

Mat. Res. Soc. Symp. Proc. (in press).

Lattice Site Location of Hf in $LiNbO_3$: Influence of Dopant Concentration and Crystal Stoichiometry

J.G. Marques[1], A. Kling[1], J.C. Soares[1,2], L. Rebouta[2], M.F. da Silva[3], E. Diéguez[4], F. Agulló-López[4]
[1] *Centro de Física Nuclear da Universidade de Lisboa, P-1699 Lisboa Codex, Portugal*
[2] *Departamento de Física, Universidade do Minho, P-4719 Braga, Portugal*
[3] *Instituto Tecnológico e Nuclear, P-2685 Sacavém, Portugal*
[4] *Departamento de Materiales, C-IV, Universidad Autónoma, E-28049 Madrid, Spain*

Abstract
The influence of dopant concentration and crystal stoichiometry on the lattice site of Hf in $LiNbO_3$ single crystals was studied combining ion beam and hyperfine interaction methods. Two cases were studied a near-stoichiometric $LiNbO_3$ crystal doped with 1 mol% HfO_2 and a congruent crystal doped with 6 mol% HfO_2. In both cases it is shown that Hf occupies both Li and Nb sites, in contrast with the case of congruent crystals doped with a small amount of Hf, where it occupies only Li sites. The roles played by the stoichiometry of the crystal and the dopant concentration are discussed in the framework of the current models for the defect structure of lithium niobate.

Nucl. Inst. and Meth. B (in press).

Annealing Recovery of Neutron Irradiated $LiNbO_3$:Hf Single Crystals

J.G. Marques[1], A. Kling[1], C.M. de Jesus[1], M.F. da Silva[2], J.C. Soares[1,2], E. Diéguez[3], F. Agulló-López[4]
[1] *Centro de Física Nuclear, Universidade de Lisboa, P-1699 Lisboa Codex, Portugal*
[2] *Instituto Tecnológico e Nuclear, P-2685 Sacavém, Portugal*
[3] *Departamento de Materiales, C-IV, Universidad Autónoma, E-28049 Madrid, Spain*

Abstract
Hf doped $LiNbO_3$ single crystals were irradiated with thermal neutrons to a fluence of 4×10^{16} n/cm^2 and characterised by Rutherford Backscattering and Perturbed Angular Correlations after irradiation and after several annealing steps. It is shown that in crystals where Hf occupied both Li and Nb sites, the fraction in Nb sites is strongly reduced after irradiation as a result of the recoil induced by the (n,γ) reaction with Hf. Annealing at 700ºC restored the initial situation. The results are discussed in the framework of the Li and Nb vacancy models proposed in the literature for the defect structure of $LiNbO_3$.

Nucl. Inst. and Meth. B (in press).

SEMICONDUCTORS

Microscopic Studies of the Hydrogen Passivation in n-Type Silicon: A New Application of the ^{73}As γ-e$^-$ PAC Technique

J.G. Correia[1], J.G. Marques[1], A. Burchard[2], M. Deicher[2], R. Magerle[2], D. Forkel-Wirth[3] and the ISOLDE Collaboration

[1]*Centro de Física Nuclear da Univ. de Lisboa, Av. Prof. Gama Pinto 2, P-1699 Lisboa Codex, Portugal*
[2]*Fakultät für Physik, Universität Konstanz, D-78434 Konstanz, Germany*
[3]*CERN, CH-1211 Geneva 23, Switzerland*

Abstract

The hydrogen passivation of As donors in Si was studied using the -e ¯ Perturbed Angular Correlation technique. The samples were doped with ^{73}As by implanting the parent isotope ^{73}Se at room temperature and 60 keV energy, to a dose of 2×10^{13} at/cm^2. After removing the radiation damage, hydrogen was implanted with low energy (300eV), to a dose of 5×10^{15} at/cm^2. Two electric field gradients were observed and assigned to H correlated complexes formed at the As donors. These complexes dissociate at temperatures lower than 1070K.

in "Shalow Level Centers in Semiconductors", eds. C.A.J. Ammerlaan and B. Pajot, World Scientific, Singapore, 1997, pp. 363-368.

The Photoluminescence of Pt-implanted Silicon

E.Alves[a], J.Bollmann[b], M.Deicher[c], M.C.Carmo[d], M.O.Henry[c,e], M.H.A.Knopf[c], J.P.Leitão[d], R.Magerle[c], C.J.McDonagh[e]

[a] *Dep. de Física, Instituto Tecnológico e Nuclear, Estrada Nacional 10, P-2685 Sacavém, Portugal*
[b] *Humboldt Univrsitaet Berlin, Invalidenstrasse 110, D-10115 Berlin, Germany*
[c] *Fakultaet Physik, Universitaet Konztanz, D-78434 Konstanz, Germany*
[d] *Departamento de Física, Universidade de Aveiro, 3810 Aveiro, Portugal*
[e] *School of Physical Sciences, Dublin City University, Dublin 9, Ireland*

Abstract

Photoluminescence measurements on Pt-implanted n-type FZ silicon samples show that three vibronic bands are produced , with principal zero-phonon lines at ~1026, 884 and 777 meV. The thermal binding energies are 34.6, 5.2 and 11.2 meV, respectivly. Uniaxial stress data show that all centres are axial. None of the spectra can be identified with known transitions on the Pt donor or acceptor centres, and they do not correspond to any previously published PL or absorption spectra. Evidence for the involvement of iron and lithium in some of the centres is presented.

Mat. Sci. Forum **258-263** (1997) 473-478.

Direct Evidence for Stability of Tetrahedral Interstitial Er in Si up to 900°C

U. Wahl[1], J.G. Correia[2], G. Langouche[1], J.G. Marques[3], A. Vantomme[1] and the ISOLDE Collaboration[2]

[1] *Instituut voor Kern- en Stralingsfysica, University of Leuven, Celestijnenlaan 200 D, B-3001 Leuven, BELGIUM*

[2] *CERN-PPE, CH-1211 Geneva 23, SWITZERLAND*

[3] *CFNUL, Av. Prof. Gama Pinto 2, P-1699 Lisboa Codex, PORTUGAL*

Abstract

Conversion electron emission channeling from the isotope ^{167m}Er (2.27 s), which is the decay product of radioactive ^{167}Tm (9.25 d), offers a means of monitoring the lattice sites of Er in single crystals. We have used this method to determine the lattice location of ^{167m}Er in Si directly following room temperature implantation of ^{167}Tm, after subsequent annealing steps, and also in situ during annealing up to 900°C. Following the recovery of implantation damage around 600°C, about 90% of Er occupies near-tetrahedral interstitial sites in both FZ and CZ Si. While in FZ Si ^{167m}Er was found to be stable on these sites even at 900°C, the tetrahedral Er fraction in CZ Si decreased considerably for 10 min annealing at 800°C and above.

Mat. Sci. Forum **258-263** (1997) 1503-1508.

Lattice Sites and Damage Annealing of Implanted Tm and Er in Si

U. Wahl[1], J.G. Correia[2], J. De Wachter[1], G. Langouche[1], J.G. Marques[3], R. Moons[1], A. Vantomme[1*], and the ISOLDE Collaboration[2]

[1] *Instituut voor Kern- en Stralingsfysica, University of Leuven, Celestijnenlaan 200 D, B-3001 Leuven, Belgium, email: ulrich.wahl@fys.kuleuven.ac.be*

[2] *CERN-PPE, CH-1211 Genève 23, Switzerland*

[3] *CFNUL, Av. Prof. Gama Pinto 2, P-1699 Lisboa Codex, Portugal*

Abstract

We have studied the lattice sites of Er in CZ Si single crystals by using conversion electron emission channeling from the isotope ^{167m}Er (2.27 s) which is the decay product of radioactive ^{167}Tm (9.25 d). Following 60 keV implantation of ^{167}Tm at a dose of 4×10^{13} cm^{-2} and annealing at 600°C, >90% of ^{167m}Er is found close to tetrahedral interstitial (T) sites, giving evidence that Er can be incorporated on tetrahedral sites in Si. The tetrahedral fraction of ^{167m}Er decreases considerably for 10 min annealing at 800°C and above. We attribute this to the onset of diffusion of the parent ^{167}Tm and its trapping at other defects, presumably oxygen atoms or clusters of Tm/Er.

Mat. Res. Soc. Symp. Proc. **469** (1997) 407-412.

Selective Area Vapor-phase Epitaxy and Structural Properties of $Hg_{1-x}Cd_xTe$ on Sapphire

N.V. Sochinskii[a], V. Munoz[a], S. Bernardi[b], J.I. Espeso[c], E. Alves[d], M.F. da Silva[d], J.C. Soares[d], C. Marin[e] and E. Diéguez[e]

[a] *Departamento de Física Aplicada, Universitat de Valencia, c/o Dr. Moliner, No.50, E-46100 Burjassot, Valencia, Spain*

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

[c] *European Synchrotron Radiation Facility, BP 220, F-38043, Grenoble, France*

[d] *Dep. de Física, Instituto Tecnológico e Nuclear, Estrada Nacional 10, P-2685 Sacavém, Portugal*

[e] *Departamento de Física de Materiales, Universidad Autónoma de Madrid, E-28049 Madrid, Spain*

Abstract

Selective area (SA) $Hg_{1-x}Cd_xTe$/sapphire layers have been grown using the recently developed technique of the vapor-phase epitaxy (VPE) of $Hg_{1-x}Cd_xTe$ layers on CdTe/sapphire heteroepitaxial substrates (HS), which we have called "VPE on HS technique" (Sochinskii *et al*., J. Crystal Growth 149 (1995) 35; 161 (1996) 195). First, planar CdTe (111) 5-7 μm thick layers were grown on sapphire (0001) wafers by metalorganic vapor-phase epitaxy (MOVPE) at 340º C for 1-2.5 h using dimethylcadmium and di-isopropyltellurium as precursors. Second, CdTe/sapphire mesas were formed using standard photolithography in the form of alternating parallel linear arrays consisting of 500 × 70 μm^2 elements. Third, SA $Hg_{1-x}Cd_xTe$/sapphire 17-23 μm thick layers with the composition x = 0.3 were obtained by the VPE of HgTe on the CdTe/sapphire mesas at 530º C for 60-90 h which was followed by the interdiffusion of Hg and Cd. Results on the structure characterization of the SA $Hg_{1-x}Cd_xTe$/sapphire layers by scanning electron microscopy, synchrotron X-ray topography and Rutherford backscattering spectrometry are reported. The layers were found to have a good single-crystalline quality and uniform composition over the entire substrate area. The structural properties of the SA $Hg_{1-x}Cd_xTe$/sapphire layers are compared with those of the planar layers grown by the VPE on HS technique under similar experimental conditions.

Journal of Crystal Growth **179** (1997) 585-591.

Direct Evidence for Tetrahedral Interstitial Er in Si

U. Wahl [1)], A. Vantomme [1)], R. Moons [1)], J. De Wachter [1)], G. Langouche [1)], J.G. Marques [2)], J.G. Correia [2,3)], and the ISOLDE Collaboration

[1)] IKS, University of Leuven, Celestijnenlaan 200 D, B-3001 Leuven, Belgium

[2)] CFNUL, University of Lisboa, Av. Prof. Gama Pinto 2, P-1699 Lisboa Codex, Portugal

[3)] CERN-PPE, CH-1211 Geneva 23, Switzerland

Abstract

We report on the lattice location of Er in Si using the emission channeling technique. The angular distribution of conversion electrons emitted by the decay chain ^{167}Tm ($t_{1/2}$=9.25 d) → ^{167m}Er (2.27 s) was monitored with a position sensitive detector following room temperature implantation and annealing up to 950°C. Our experiments give direct evidence that Er is stable on tetrahedral interstitial sites in float-zone Si. We also confirm that rare earth atoms strongly interact with oxygen, which finally leads to their incorporation on low-symmetry lattice sites in Czochralski Si.

Phys. Rev. Lett. **79** (1997) 2069-2072.

Acceptor-Hydrogen Interaction in InAs

A.Burchard[1], J. G. Correia[2], M. Deicher[1], D. Forkel-Wirth[2], R. Magerle[1], A. Prospero[1], A. Stötzler[1], and the ISOLDE Collaboration[2]

[1] *Fakultät für Physik, Universität Konstanz, D-78457 Konstanz, Germany*

[2] *CERN / PPE, CH-1211 Geneva 23, Switzerland*

Abstract

The hydrogen passivation of the group IIB acceptor Cd in InAs was studied using the perturbed angular correlation spectroscopy. After low energy H+ implantation up to three, different hydrogen correlated complexes are formed at the Cd probe atom. After H+ implantation with an energy of 100 eV the well-known Cd-H pair, oriented along <111> lattice directions (υ_Q = 427 MHz, η= 0) and the complex C555 (υ_Q = 555 MHz, η=0. 19) were found. After H+ implantation at 1 keV the Cd-H pair and a different complex C577 (υ_Q = 577 MHz, η= 0.09) are detected. Both, C555 and C577 transmute into Cd-H and the corresponding transformation energy is ED = 1.0(1) eV. An interpretation of C555 and C577 in terms of Cd-H, or Cd-H-defect complexes is possible. Besides these hydrogen correlated complexes a fourth one, C168, appeared after He+ and hydrogen implantation. It is labeled by υ_Q = 168 MHz, η = 0.0 and its nature is not yet identified.

Mat. Sci. Forum **258-263** (1997) 1223.

Performance Degradation of Microcrystalline Silicon-Based p-i-n Detectors upon He^4 Irradiation

R. Schwarz[1], M. Vieira[2], F. Maçarico[2], S. Koynov[3], S. Cardoso[4], J.C. Soares[5]

[1] *Physics Department, Instituto Superior Técnico, 1096 Lisboa, Portugal*

[2] *ISEL, Instituto Superior de Engenharia de Lisboa, 1900 Lisboa, Portugal*

[3] *CL-SeNES, Bulgarian Academy of Sciences, 1784 Sofia, Bulgaria*

[4] *INESC, Instituto de Engenharia de Sistemas e Computadores, 1000 Lisboa, Portugal*

[5] *ITN, Instituto Tecnológico e Nuclear, 2685 Sacavém, Portugal*

Abstract

Microcrystalline Silicon p-I-n structures show a good responsivity in the visible and near infrared spectral region. We have used a 1.6 MeV He^4 beam to study the –degradation of the spectral response and the changes in the current-voltage characteristics of such detectors. All the layers in the detector structure were deposited by high-rate plasma-enhanced chemical vapor deposition. Under a fluence of 3×10^{15} cm^{-2} the short circuit current decreased by about a factor of 3. The decrease of bulk material recombination life time is evidenced by the shift of the peak energy of the spectral response to short wavelengths. The degradation effect is compared to reports on amorphous and monocrystalline silicon detectors.

Mat. Sci. Forum **258-263** (1997) 593-598.

Nucleation and Growth of Platelet Bubble Structures in He Implanted Silicon

P.F.P. Fichtner[1a], J.R. Kaschny[2], A. Kling[3], H. Trinkaus[4], R.A. Yankov[2], A. Mücklich[2], W. Skorupa[2], F.C. Zawislak[1b], L. Amaral[1b], M.F. da Silva[5], J.C. Soares[3,5]

[1] *Departamento de Metalurgia (a) / Instituto de Física (b), Universidade Federal do Rio Grande do Sul, P.O.B. 15051, 91501-970 Porto Alegre, RS, Brazil*

[2] *Research Center Rossendorf, D-01314 Dresden, Germany*

[3] *Centro de Física Nuclear, Universidade de Lisboa, P-1699 Lisboa, Portugal*

[4] *Institut für Festkörperforschung, KFA Jülich, P.O.B. 1913, D-5170 Jülich, Germany*

[5] *Instituto Tecnológico e Nuclear, P-2685 Sacavém, Portugal*

Abstract

He^+ ions were implanted into (100) Si at energies from 30 to 120 keV and fluences from 5×10^{15} to 1×10^{16} cm^{-2}. After implantation, pieces of these samples were subjected to rapid thermal annealing for 600 s at temperatures ranging from 300 to 700°C. The samples were analyzed by Transmission Electron Microscopy (TEM) and by Rutherford Backscattering and channeling spectrometry (RBS/C). The TEM observations were related to the RBS/C measurements and the results discussed in terms of a nucleation model to explain the formation of overpressurized bubbles in He implanted and annealed silicon.

Nucl. Inst. and Meth. B (in press)

Lattice Site Location of Tu and Er Implanted GaAs

E.Alves[1], M.F. da Silva[1], J.C. Soares[1,2], M.O.Henry[3], R.Gwilliam[4] , B.J. Sealy[4], K.Freitag[5], R.Vianden[5] and A. Stievenard[6]

[1]*- Instituto Tecnológico e Nuclear(ITN), EN 10, Sacavém, Portugal*

[2]*- Centro de Física Nuclear da Univ. de Lisboa, Av. Prof.Gama Pinto 2, 1699 Lisboa, Portugal*

[3]*- School of Physical Sciences, Dublin City University, Dublin, Ireland*

[4]*- Department of Electrical and Electronic Engineering, Univ. of Surrey, Guildford, GU25HX, U.K*

[5]*- Institut fur Strahlen und Kernphysik der Universitat Bonn, D-53115 Bonn, Germany*

[6]*- Institut Supérieur d´Electronic du Nord, 41 Boulevard Vauban, 59046 lille, France*

Abstract

The lattice site location of Tm and Er implanted in GaAs were studied using the RBS/channeling technique. For implanted doses up to 10^{14} cm^{-2} both elements show nearly 100% substitutionality in the GaAs lattice. Further increasing the Er concentration causes a fraction to occupy an interstitial regular site. Axial channeling measurements along planar directions show that Er is incorporated in the GaAs matrix forming ErAs precipitates. The angular scans are compared with Monte Carlo simulations and the Er fractions in the different lattice sites are calculated. With the incorporation of oxygen in the GaAs matrix the fraction of Er in substitutional sites increases. Taking into account these results the photoluminescence (PL) behavior of Er in these samples is discussed and results obtained with Er doped GaAs grown by Molecular Beam Epitaxy are understood.

Nucl. Inst. and Meth. B (in press)

RBS and SIMS Study of the Simultaneous Phase Growth of Iridium Silicides Formed by RTA in Vacuum

A. Almendra[1], J.J. Serrano[1], A. Kling[2], T. Rodríguez[1], J.M. Blanco[1], M.F. da Silva[3], J. Sanz-Maudes[1], M. Aguilar[1], J.C. Soares[2,3]

[1] *Departamento de Tecnologia Electrónica, E.T.S.I. de Telecomunicación, Ciudad Universitaria s/n, 28040 Madrid, Spain*

[2] *Centro de Física Nuclear, Universidade de Lisboa, P-1699 Lisboa, Portugal*

[3] *Instituto Tecnológico e Nuclear, P-2685 Sacavém, Portugal*

Abstract

In the present work, we report results on the quantitative analysis of the kinetics of growth of IrSi and $IrSi_{1.75}$ by RTA vacuum, taking into account that a layered silicide formation of the two compounds proceeds simultaneously. The composition and thickness of the different layers as a function of annealing temperature and time has been obtained by RBS measurements. We have used a multiphase system model based on the simultaneous growth of different phases, assuming that silicon is the moving reactant in the Ir/Si system, to obtain the interfacial reaction constant K and the diffusion coefficient D of silicon in both silicides as a function of annealing temperature. With the help of the layer thickness vs. annealing time plots at different temperature, we have calculated, using numerical analysis techniques, two pairs of constants related to K and D. SIMS characterization of the samples has been necessary to estimate the values of the silicon concentration gradients, so that we have been able to obtain the final values of K and D at different processing temperatures, and, as a consequence, to determine their activation energies.

Nucl. Inst. and Meth. B (in press).

Electron Emission Channeling with Position-Sensitive Detectors

U. Wahl[1], J.G. Correia[2], S. Cardoso[3], J.G. Marques[3], A. Vantomme[1], G. Langouche[1], and the ISOLDE collaboration[2]

[1] *IKS, University of Leuven, Celestijnenlaan 200 D, B-3001 Leuven, Belgium*

[2] *CERN-PPE, CH-1211 Geneva 23, Switzerland*

[3] *CFNUL, Univerity of Lisboa, Av. Prof. Gama Pinto 2, P-1699 Lisboa, Portugal*

Abstract

Electron emission allows direct lattice location studies of low doses of radioactive atoms implanted in single crystals. For that purpose the anisotropic emission yield of conversion electrons from the crystal surface is measured, most conveniently by use of position-sensitive detectors. We discuss characteristic features of this method, including quantitative data analysis procedures, which are achieved by fitting simulated two-dimensional emission distributions for different lattice sites to the experimental patterns. The capabilities of this approach will be illustrated by the results of recent lattice location studies of rare earth atoms (Er, Tm, Yb) in Si, where we were able to do lattice location experiments down to implanted doses which are 150 times lower compared to previous RBS studies.

Nucl. Inst. and Meth. B (in press)

RBS Studies of the Intercalation Compound Hg_xTiS_2: Morphology and Staging

T. Butz[1], R.-H. Flagmeyer[1], St. Jankuhn[1], T. Reinert[1], M.F. da Silva[2], J.C. Soares[2,3], W. Tröger[1]

[1] *Universität Leipzig, Fakultät für Physik und Geowissenschaften, Abteilung Nukleare Festkörperphysik, Linnéstr. 5, 04103 Leipzig, Germany*

[2] *Instituto Tecnológico e Nuclear, Est. Nac. 10, 2685 Sacavém, Portugal*

[3] *CFNUL, Univerity of Lisboa, Av. Prof. Gama Pinto 2, P-1699 Lisboa, Portugal*

Abstract

In order to study the intercalation process of Hg into the layered crystal TiS_2 we performed Rutherford backscattering (RBS) and channeling experiments on TiS_2 and on the intercalation compound Hg_xTiS_2 with variable uptake. After the intercalation of Hg in TiS_2 we observed a clear Hg-signal in the RBS-spectrum, increasing with reaction time and temperature. The variation of the Hg RBS-signal depending on the lateral position on the analysing beam can not be explained by vertical reaction fronts or by a spatially homogeneous intercalation process. We conclude from these measurements that the intercalation process proceeds parallel to the TiS_2 layers in concave reaction fronts. The empty TiS_2 crystal exhibits a good channeling effect ($\chi_{min} \approx 20\%$). Owing to Hg uptake, however, the ion channeling effect in the "misfit compound" Hg_xTiS_2 vanishes. We attribute this to the incommensurability of the Hg and TiS_2 sublattices as well as to a bending of the crystal induced by the intercalation process.

Nucl. Inst. and Meth. B (in press)

RBS/Channeling Characterization of GaSb and Ion-Implanted GaSb after Several Surface Treatments

Zhongning Dai[1], J.C. Soares[1], M.F. da Silva[2]

[1] *CFNUL, Univerity of Lisboa, Av. Prof. Gama Pinto 2, P-1699 Lisboa, Portugal*

[2] *Instituto Tecnológico e Nuclear, Est. Nac. 10, 2685 Sacavém, Portugal*

Abstract

Gallium antimonide surfaces are characterized by RBS/Channeling after several different treatments, including mechanical polishing, chemical etching, annealing, ion implantation and subsequently annealing after different doses' implantation of 100 keV $^{180}Hf^+$. We have found that the GaSb samples have much better crystalline quality both after chemical etching at a certain condition and annealing at 350º C. After 100 keV $^{180}Hf^+$ implantation at a dose of $5 \times 10^{14}/cm^2$ the sample is heavily damaged, and almost amorphized. However the GaSb sample can recover after 600º C annealing in a conventional furnace for 20 s. After 100 keV $^{180}Hf^+$ implantation at a dose of $5 \times 10^{16}/cm^2$, the GaSb crystal is almost completely amorphized. Annealing in a conventional furnace at 500º C in open air for 10 minutes result in the loss of Sb and Ga on both implanted and un-implanted sides of the sample. The possible reason is the oxidation of the sample, as we have found oxygen on the surface. Different annealing temperatures of RTA (Rapid Thermal Annealing) show there is no Sb or Ga loss or oxidation in the flowing N2 for 10 s, and there is almost no much more improvement at temperature higher than 350º C.

Nucl. Inst. and Meth. B (in press)

Epitaxial Regrowth of C- and N-implanted Silicon and α-Quartz

F. Harbsmeier[1], W. Bolse[1], M.R. da Silva[2], M.F. da Silva[3], J.C. Soares[2,3]
[1]*Physikalishes Institut and Sonderforschungsbereich 345 Universität Göttingen, Germany*
[2]*Centro de Física Nuclear, Univ. de Lisboa, Av. Prof. Gama Pinto 2, 1699 Lisboa Codex, Portugal*
[1]*Instituto Tecnológico e Nuclear, Estrada Nacional 10, 2685 Sacavém, Portugal*

Abstract
We have investigated the epitaxial regrowth and diffusion during thermal annealing of C and N implanted Si and α-quartz, utilising Rutherford Backscattering spectrometry in channeling geometry (RBS-C) and nuclear reaction analysis (NRA). In both materials solid phase epitaxial regrowth was observed and occurs at temperatures around 550º C (Si) and 1000º C (SiO2). Epitaxial regrowth in Si was found to be strongly retarded as compared to self ion implantation and the epitaxial recrystalization front stopped at about the depth of maximum implant concentration even after annealing at 900º C. In fact, the implanted N-concentration profile in Si was found to be unchanged up to this temperature. This point at a chemical reaction between Si and the implanted C- atoms and N-atoms, which obviously suppresses epitaxial recrystallisation. In SiO2 almost no nitrogen could be detected anymore in the N-implanted sample at T ≥ 900º C, i.e. no significant amount of impurity atoms was present during recrystallisation, which may hinder or suppress the regrowth process. A similar behaviour is expected for the implanted C-atoms. In fact, epitaxial recrystallisation in the C-implanted α-quartz sample was found to be almost complete, except for a small amount of defects in the recrystallised matrix.

Nucl. Inst. and Meth. B (in press)

Lattice Sites and Stability of Implanted Er in FZ and CZ Si

U. Wahl[1], J.G.Correia[2], G. Langouche[1], A. Vantomme[1], and the ISOLDE Collaboration[2]
[1] *Instituut voor Kern- en Stralingsfysica, University of Leuven, Celestijnenlaan 200 D, B-3001 Leuven, Belgium, email: ulrich.wahl@fys.kuleuven.ac.be*
[2] *CERN-PPE, CH-1211 Genève 23, Switzerland*

Abstract
We report on the lattice location of ^{167}Er in Si measured by conversion electron emission channeling. In both FZ (Float Zone) and CZ (Czochralski) Si, a high fraction of Er (>65%) occupies near-tetrahedral interstitial (T) sites directly following 60 keV room temperature implantation at doses of 6×10^{12} cm^{-2}. For higher doses, the as-implanted near-T fractions of Er visible by emission channeling are smaller, due to beginning amorphization. Following the recovery of implantation damage at 600°C, more than 70% of Er is found on near-T sites in both FZ and CZ Si. In FZ Si, Er exhibits a remarkable thermal stability and only prolonged annealing for several hours reduces the near-T fraction. On the other hand, annealing of CZ Si at 900°C for more than 10 minutes results in the majority of Er probes in sites of very low symmetry or disordered surroundings.

Mat. Res. Soc. Symp. Proc., (in press).

Electrical Properties of Erbium and Thullium Implanted GaAs Correlated with Photoluminescence and Rutherford Backscattering Results

D.Stiévenard[1], D.Deresmes[1], X.Wallart[1], K.Freitag[2], R.Vianden[2], S.E.Daly[3], M.O.Henry[3], E.Alves[4], J.C.Soares[4,5], R.G.Gwilliam[6], B.J.Sealy[6]

[1]- *IEMN-DepT. ISEN, UMR9929 (CNRS), 41 Bd Vauban, 59046 Lille Cédex (France)*

[2]- *Institut für Strahlen und Kernphysik der Universität Bonn, D-53115 Bonn(Germany)*

[3]- *School of Physical Sciences, Dublin City University, Dublin 9 (Ireland)*

[4]- *Instituto Tecnológico e Nuclear, Est. Nac. 10, 2685 Sacavém (Portugal)*

[5]- *Centro de Fisica Nuclear da Univ. de Lisboa, Av. Prof. Gama Pinto, 2, 1699 Lisboa (Portugal)*

[6]-*Dept. of Electrical and Electronic Engineering, University of Surrey, Guilford, GU25XH (U.K.)*

Abstract

Electrical characterisation (Capacitance - Voltage, Deep Level Transient Spectroscopy) of Erbium and Thullium implanted GaAs are presented. Electron traps are detected at Ec-0.55 eV and Ec-0.54 eV respectively for thulium and erbium implanted GaAs. A compensation of the free carriers is associated with these two defects. An hole trap at Ev + 0.36 eV is also detected in both cases. In order to identify these traps, we have performed Photoluminescence and Rutherford Backscattering (RBS). We propose that the electron traps are associated with substitutionnal rare earth (erbium or thulium) on gallium atomic site.

submitted to *Europhysics Letters*

METALS

Analysis of the Elements Sputtered During the Lanthanum Implantation in Stainless Steels

F.J. Ager[1], M.A. Respaldiza[1], A. Paúl[2], J.A. Odriozola[2], M.F. da Silva[3], L. M. Redondo[3] and J.C. Soares[3,4]

[1]*Departamento de FAMN, Universidad de Sevilla, PO Box 1065, Sevilla E-41080, Spain;*
[2]*ICMSE, Universidad de Sevilla-CSIC, PO Box 874, E-41071 Sevilla, Spain;*
[3]*ITN, E.N. 10, P-2685 Sacavém, Portugal;*
[4]*CFNUL, Av. Prof. Gama Pinto 2, P-1699 Lisboa Codex, Portugal.*

Abstract

The addition of lanthanum to the surface composition of AISI304 stainless steel has been proved to improve its resistance to high temperature oxidation. During the implantation process, the surface atoms, either from the steel matrix or already implanted, are pulled out of the surface. In previous implantation experiments in AISI304 performed by us, using 100 keV La^+ and doses comparable to those used in chemical deposition methods proved useful to improve the oxidation, EDX and optical microscopy showed the formation of iron oxide-rich nodules after the oxidation that lead to an inadequate behaviour. This could be related to the modification of the surface structure induced by the implantation process, appearing a new grain structure and a bcc-like phase, and/or to a differential sputtering that could enrich the relative amount of iron in the surface, preventing the formation of protective chromium oxides. Trying to find evidences of this differential sputtering, some AISI304 samples were placed in front of the other steel samples being implanted at 45º and shaded from the implantation beam, together with an almost pure carbon piece to collect the sputtering products. They were implanted with different fluencies and analysed after implantation by RBS and PIXE. This way, the sputtered atoms heavier than carbon taken from the steel surface can be clearly detected in the RBS and PIXE spectra and thus a quantitative analysis can be performed for such elements. After these analyses, the steel samples are annealed in an inert atmosphere to recover the structure and composition of the implanted layer, oxidised and studied again by RBS and by thermogravimetric methods.

Nucl. Instr. and Meth. B (in press).

Oxide Scale Depth Profiling of Lanthanum-Deposited AISI304: an Ion Beam Analysis

F.J. Ager[a], M.A. Respaldiza[a], A. Paúl[b], J.A. Odriozola[b], M.F. da Silva[c] and J.C. Soares[c,d]
[a]*Departamento de FAMN, Universidad de Sevilla, PO Box 1065, Sevilla E-41080, Spain;*
[b]*ICMSE, Universidad de Sevilla-CSIC, PO Box 874, E-41071 Sevilla, Spain;*
[c]*ITN, E.N. 10, P-2685 Sacavém, Portugal;*
[d]*CFNUL, Av. Prof. Gama Pinto 2, P-1699 Lisboa Codex, Portugal.*

Abstract
Rare earth deposits on stainless steels are known to improve the corrosion resistance at high temperatures, although the mechanism of this phenomenon is not yet completely understood. A detailed study of the composition of oxide scales formed at 1173 K onto lanthanum-deposited AISI304 specimens by means of a modified chemical vapour deposition method (CVD), PYROSOL, has been started with the help of ion beam analysis techniques such as Rutherford backscattering spectrometry (RBS) and nuclear reaction analysis (NRA). Complementary data on the protective effects has been obtained by means of other analytical techniques such as scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDX) and thermogravimetric measurements (TG).
Two sets of La-deposited AISI304 samples, one of them with a mill finishing and the other with a 2 minutes at 1173 K preoxidation treatment prior to lanthanum deposition, were oxidised at 1173 K in synthetic air together with a set of non-treated samples. TG measurements show that non-treated samples change from protective to non-protective behaviour after 10 hours in synthetic air stream, whereas the coated samples show a refractory behaviour for longer oxidation times (up to 50 h.), proving the effectiveness of the La deposition.
By combining RBS (H^+ and He^+) and NRA data ($^{52}Cr(p,\gamma)^{53}Mn$ and $^{55}Mn(p,\gamma)^{56}Fe$) oxide scale depth profiles, including O, La, Fe, Mn and Cr, have been obtained. This way IBA techniques are proven useful in determining the composition of the oxide layer grown onto the alloy surface helping in the understanding of the oxidation mechanisms at high temperature and the reactive element effect.

Nucl. Instr. and Meth. B (in press).

THIN FILMS

Magnetization and Magnetoresistance in Fe Ion-implanted Cu and Ag Thin Films

M.M. Pereira de Azevedo[1], J.A. Mendes[1], B.G. Almeida[1], M.S. Rogalski[1], Yu.G. Pogorelov[1], J.B. Sousa[1], I. Bibicu[3], L.M. Redondo[2], M.F. da Silva[2], C.M. de Jesus[4], J.G. Marques[4], J.C. Soares[2,4]

[1] *IFIMUP and Physics Dep. of Fac. of Sciences, University of Porto, P-4150 Porto, Portugal*
[2] *Institute of Nuclear Technology, P-2680 Sacavém, Portugal*
[3] *Institute of Atomic Physics, POBox MG-06, Bucharest, Romania*
[4] *CFNUL, University of Lisbon, 1699 Lisbon, Portugal*

Abstract
Diluted granular films of Cu-Fe and Ag-Fe (iron content ≤ 2%) were produced using ^{57}Fe ion implantation on Cu(Ag) films previously grown by laser ablation. Conversion electron Mössbauer spectroscopy shows that the implanted Fe forms either very small clusters (up to a few atoms) or large α - phase particles. These structural characteristics are directly reflected on the magnetisation, which exhibits ferromagnetic-like behaviour at room temperature (due to large clusters) superimposed by a significant paramagnetic contribution at low temperatures due to small clusters. We observe deviations from strict superparamagnetic behaviour due to non-negligible local anisotropy effects at low temperatures and low fields.
The condo effect is particularly enhanced in the Cu-Fe films which have higher concentration of isolated Fe atoms and small size clusters.
The magnetoresistivity $\Delta\rho/\rho$ of our films is dominated (for $0 \leq \mu_0 H \leq 15$ Tesla) by a linear term in H, attributed to GMR-like effect from spin dependent scattering when an electron passes between adjacent large and small clusters. At low fields we observe instead $\Delta\rho/\rho \propto H^2$, due to the usual GMR effect between large clusters, during the alignment of their easy axes. The relevant physical differences (structural, magnetic and magnetoresistive) observed in our ion-implanted diluted Fe films, with respect to the concentrated granular films are critically analysed.

J. Magn. Magn. Mat. **173** (1997) 230-240.

Changes in Structure and Composition of Silicon Oxide Thin Films Induced by Ultraviolet Illumination

E. G. Parada[1] , P. González[1], B. León[1], M. Pérez-Amor[1], M. F. Da Silva[2], J. C. Soares[2,3], A. Fernández[4], A. R. González-Elipe[4]

[1] *Departamento de Física Aplicada, Universidade de Vigo, Campus Lagoas-Vigo, Galicia, Spain*
[2] *Departamento de Física, Instituto Tecnológico e Nuclear, Sacavém, Portugal*
[3] *Centro de Física Nuclear, Universidade de Lisboa, Lisboa, Portugal*
[4] *Instituto de Ciencia de Materiales/CSIC Sevilla, Spain*

Abstract
The transformation of silicon oxide thin films at room-temperature by UV-photons provided by a Xe_2^* incoherent excimer lamp (λ = 172 nm, $\Delta\lambda$ = 14 nm) was studied. Films were produced

at low-temperature (T = 260 ºC) by ArF laser-induced CVD (LCVD) in parallel configuration from a silane/nitrous oxide/argon gas mixture. The silicon oxide films were irradiated in several consecutive steps to follow-up the modifications with the illumination time. Rutherford back-scattering (RBS), infrared (IR) and X-ray photoelectron (XPS) spectroscopies, ellipsometry, and elastic recoil detection analysis (ERDA) were used to characterize the effects of the irradiation on the structure, composition, density, and hydrogen content.
Under the UV illumination the as-deposited film evolves from a suboxide film ($SiO_{1.6}$) to a stoichiometric silicon dioxide (SiO_2), and its originally strained structure changes towards a relaxed tetrahedral configuration. The UV irradiation is able to anneal at room temperature the silicon oxide films breaking the Si-H bonds and incorporating new SiO and hydroxyl groups in a relaxed network. The hydrogen does not effuse out, but remains in the film as molecular hydrogen and/or forming silanol or water groups.

Mat. Res. Soc. Symp. Proc. **Vol. 441** (1997) 211.

Magnetic Properties and Structure of a New Multilayer $[FeTaN/TaN]_n$ for Recording Heads

S. Li[1], P.P. Freitas[1], M.S. Rogalski[2], M.M. Pereira de Azevedo[2], , J.B. Sousa[2], Z.N. Dai[3,4], J.C. Soares[3,4], N. Matsakawa[5], H. Sakakima[5]

[1] *INESC and IST, R. Alves Redol 9-1, 1000 Lisbon, Portugal*
[2] *IFIMUP, FCUP, R. Campo Alegre, 687, P-4150 Porto, Portugal*
[3] *Institute of Nuclear Technology, P-2680 Sacavém, Portugal*
[4] *CFNUL, University of Lisbon, 1699 Lisbon, Portugal*
[5] *Matsushita Electric Industrial Co. Ltd., Japan*

Abstract
In this article, the structure and soft magnetic properties of $[FeTaN/TaN]_n$ multilayers have been studied. Best multilayer films have coercivity of 1.2 Oe, which is further decreased to 0.5 Oe by annealing up to 400 ºC. The saturation magnetization is 19.5 kG. The initial permeability of multilayer films is 1200 and is constant up to 100 MHz. A predominant α-Fe(110) texture is observed by x-ray diffraction patterns and Mössbauer spectra (MS), but a small amount of ferromagnetic ε-Fe_xN ($x \geq 3$) and paramagnetic ε-Fe_xN ($2 < x < 3$) phases are identified by MS, which may account for the relatively large magnetostriction ($\sim 5 \times 10^{-6}$). The temperature dependence of MS and x ray show good thermal stability up to 430 ºC.

J. Appl. Phys. **81** (8) 1997) 4501-4503.

Structural and Compositional Studies of a-Si:C:H Thin Films Obtained by Excimer Lamp-CVD from Acetylene and Silane

X. Redondas[1], P. González[1], B. León[1], M. Pérez-Amor[1], J. C. Soares[2,3] and M. F. da Silva[3]
[1]*Dpto. de Física Aplicada, Univ. de Vigo, Lagoas-Marcosende 9, E-36200 Vigo, (Galicia, Spain)*
[2] *CFNUL, Av. Prof. Gama Pinto 2, Lisboa, (Portugal)*
[3] *Instituto Tecnológico Nuclear, Estrada Nacional 10, Sacavem, (Portugal)*

Abstract
Amorphous silicon-carbon thin films were obtained on silicon and steel substrates, by irradiating a gas mixture of Ar-diluted silane and acetylene by a Xe_2^* excimer lamp in a reaction chamber. The influence of the deposition parameters on film properties was analyzed by different techniques. Film composition was determined by Rutherford Backscattering Spectroscopy (RBS) and Elastic Recoil Detection Analysis (ERDA) and the structure of the material was studied by Infrared and Raman Spectroscopies, X-ray Photoelectron and Auger Electron Spectroscopies. Thickness and refractive index were evaluated by ellipsometry.
Structural studies reveal that carbon and silicon are present in the films mainly forming homonuclear and hydrogenated bonds. Compositional analyses indicate high carbon concentrations and only slight variations for dramatical changes of precursor gas mixture composition. Lamp power tuning affects the growth rate and film density. Moreover oxygen is incorporated when the layers are exposed to the atmosphere forming silicon oxides.

J. Vac. Sci. Technology (in press).

Microscopic Studies of Radioactive Hg Implanted in $YBa_2Cu_3O_{6+x}$ Superconducting Thin Films

V.S.Amaral[1], J.G. Correia[2,3], A.A. Lourenço[4], J.G. Marques[3], J.A. Mendes[1], M.A. Baptista[1], J.P. Araújo[1], J.M. Moreira[1], J.B.Sousa[1], E. Alves[5], M.F. da Silva[5], J.C. Soares[3,5] and the ISOLDE Collaboration[2]
[1]*IFIMUP, Dep. Física, Faculdade de Ciências, Rua do Campo Alegre 687, P-4150 Porto, Portugal*
[2]*Particle Physics Experiments Division, CERN, CH-1211 Geneva 23, Switzerland*
[3]*Centro de Física Nuclear, Univ. Lisboa, Av. Prof. Gama Pinto 2, P-1699 Lisboa Codex, Portugal*
[4]*INESC, R.Campo Alegre 687, P-4150 Porto, and Dep. Física, Univ. Aveiro, P-3800 Aveiro, Portugal*
[5]*Instituto Tecnológico e Nuclear, Estrada Nacional 10, P-2685 Sacavém, Portugal*

Abstract
$YBa_2Cu_3O_{6+x}$ (YBCO) superconducting thin films implanted with low doses of radioactive ^{197m}Hg and ^{199m}Hg isotopes were studied with a combination of nuclear and non-nuclear characterization techniques. We show that after implantation Hg lies on a unique site in the YBCO lattice and the critical temperature increases slightly with the increase of the ^{197}Au concentration from the nuclear transmutation of ^{197}Hg.

J. Magn. Magn. Mat. (in press).

Growth and Characterization of Carbon Nitride Thin Films Prepared by Laser Ablation

R. Soto[1], P. González[1], X. Redondas[1], E. G. Parada[1], J. Pou[1], B. León[1], M. Pérez-Amor[1], M. F. da Silva[2] and J. C. Soares[2,3]
[1]*Dpto. Física Aplicada, Univ. Vigo, Lagoas-Marcosende 9, E-36200 Vigo, Galicia, Spain*
[2]*ITN, E. N. nº 10, 2685 Sacavém, Portuga.*
[3]*CFNUL, Av. Prof. Gama Pinto 2, 1699 Lisboa, Portugal*

Abstract
Rutherford Backscattering Spectroscopy has been systematically applied to determine the composition of carbon nitride thin films prepared by laser ablation at different substrate temperatures. The films were deposited onto silicon and aluminium substrates by irradiating a glassy graphite target with an ArF excimer laser (193 nm) in a reactive ammonia atmosphere. The characterization was completed with the additional analysis techniques Energy Dispersive X-ray Spectroscopy (EDS), Fourier Transform Infrared Spectroscopy (FTIR) and profilometry.
The role of the processing substrate temperature on the film growth and properties is investigated. RBS measurements indicate that the concentration of nitrogen decreases from 23 to 9 at.%, as the temperature is increased up to 300 ºC. The infrared analyses show that nitrogen is bonded to carbon in different configurations (1300 - 1650 cm^{-1}) and the presence of hydrogen in the layers forming CH_x and NH_x groups.
For this new material, several calibration diagrams for often used techniques, such as EDS and FTIR, are presented. The inverse absorption cross section of the C=N vibration mode, as well as the calculation of the apparent activation energy of the process are reported.

Nucl. Instr. and Meth. B (in press).

High Flux $^{56}Fe^{+}$ and $^{57}Fe^{+}$ Implantations for GMR Applications

L.M. Redondo[1], C.M. de Jesus[2], J.G. Marques[2], M.F. da Silva[1], J.C. Soares[1,2], M.M. Pereira de Azevedo[3], J.A. Mendes[3], M.S. Rogalski[3] and J.B. Sousa[3]
[1] *Instituto Tecnológico e Nuclear, E.N. 10, P-2685 Sacavém, Portugal*
[2] *CFNUL, Universidade de Lisboa, Av. Prof. Gama Pinto 2, P-1699 Lisboa Codex, Portugal*
[3] *IFIMUP, Universidade do Porto, P-4150 Porto, Portugal*

Abstract
The magnetoresistive behaviour of granular thin films prepared by ^{56}Fe and ^{57}Fe ion implantation into Ag thin films, in doses up to $8x10^{16}$ at/cm^2 is reported. The implantation produced both small and large Fe clusters, with the large clusters being dominant for high doses. A significant magnetoresistive response of the films was obtained for doses above $6x10^{16}$ Fe/cm^2, reaching values of 1-2% at 10 K and low fields.

Nucl. Instr. and Meth. B (in press).

Ion Beam Studies of CdTe Films Epitaxially Grown on Si, GaAs and Sapphire Substrates

E.Alves[1], M.F. da Silva[1], J.C. Soares[1,2], N.V. Sochinskii[3] and S. Bernardi[4]
[1] *Instituto Técnologico e Nuclear, E.N 10, P-2685 Sacavém, Portugal*
[2] *Centro de Física Nuclear, Univ. de Lisboa, Av. Prof.Gama Pinto 2, P-1699 Lisboa, Portugal*
[3] *Dep. de Fisica Aplicada,Univ. de Valencia, c/ Dr. Moliner nº50, E-46100 Burjassot, Spain*
[4] *Consorzio C.R.E.O, Via Pile 60, I- 67100 L' Aquila, Italy*

Abstract
The epitaxial growth and the crystalline quality of CdTe films grown on different substrates were studied using Rutherford backscattering spectrometry combined with channeling. The films which were 2.5 to 20 μm thick, were grown by metalorganic vapor phase epitaxy (MOVPE). Despite the big lattice mismatch (>10%) between the CdTe and the Si, GaAs and Al_2O_3 substrates used, a good epitaxial growth of the films was observed. The results show nearly defect free films with the following epitaxial relationship with the substrates: (111)CdTe || (0001)Al_2O_3, (100)CdTe || (100)GaAs, (111)CdTe || (100)Si. The values of the minimum yields, measured along the main axial directions, indicate that the best films were obtained with sapphire substrates. The surface quality of the layers improves with a subsequent recrystallization induced by laser annealing. These results, combined with previous electron microscopy and Photoluminescence studies, give the possibility to optimize the growth conditions of large area CdTe layers used in the infrared technology.

Nucl. Inst. and Meth. B (in press).

Determination of the Strain Depth Profile in Solid-phase Epitaxially Grown SiGe Layers Using RBS/channeling

A. Rodríguez[1], T.Rodríguez[1], A. Kling[2], J.C. Soares[2,3], M.F. da Silva[3], C. Ballesteros[4]
[1] *Departamento de Tecnologia Electrónica, E.T.S.I. de Telecomunicación, Ciudad Universitaria s/n, 28040 Madrid, Spain*
[2] *Centro de Física Nuclear, Universidade de Lisboa, P-1699 Lisboa, Portugal*
[3] *Instituto Tecnológico e Nuclear, P-2685 Sacavém, Portugal*
[4] *Departamento de Física, E.P.S., Universidad Carlos III., E-28911 Leganés, Spain*

Abstract
The strain depth profiles in undoped and heavily boron-doped $Si_{1-x}Ge_x$ layers with x=0.21, 0.26 and 0.34 and thicknesses of 30-44 nm grown on (001) Si by solid phase epitaxy have been measured using RBS/channeling angular scans and the results have been correlated with the defect distributions observed in the samples by high resolution electron microscopy. The layers exhibit a partially relaxed defect free region next to the layer-substrate interface for which the strain is almost constant. for the top region of the layers a decrease of the strain, which correlates well with the observation of strain-relieving defects, is found. The defect-free regions are thicker and closer to coherency to the Si substrate in the doped layers than in the undoped ones, indicating a strain compensation due to the inclusion of boron atoms.

Nucl. Inst. and Meth. B (in press).

Modification of Silicon-Carbon Film Properties Under High Energy Ion Beam Irradiation

X. Redondas[1], P. González[1], R. Soto, B. León[1], M. Pérez-Amor[1], M. F. da Silva[2] and J. C. Soares[2,3]
[1]*Dpto. Física Aplicada, Univ. de Vigo, Lagoas-Marcosende 9, 36200 Vigo, Galicia, Spain*
[2] *ITN, E.N. nº 10, 2685 Sacavém, Portugal*
[3]*CFNUL, Av. Prof. Gama Pinto 2, 1699 Lisboa, Portugal*

Abstract
Changes induced on the properties of amorphous hydrogenated silicon-carbon layers by the 2 MeV $^4He^+$ ion beam irradiation during RBS and ERD measurements are reported. Thin films have been deposited by photo-CVD using a Xe_2^* excimer lamp at low temperatures ranging from 60 ºC to 300 ºC from different gas mixtures (acetylene, ethylene, silane and disilane). Additional techniques, such as ellipsometry and infrared spectroscopy have been employed to complete the film characterization.
Hydrogen is removed from the material while the samples are irradiated, and an exponential decrease of the ERD counts is observed with the number of ions impinging the surface. Ellipsometric measurements reveal that this effect depends on the film properties, since for films produced from ethylene and disilane, a linear relationship between the relative amount of hydrogen removed and the refractive index was found.
It is well known that this material presents an aging process when exposed to atmospheric conditions, consisting basically in a film oxidation and the consequent diminution of the refractive index values. This effect is reduced for the ion beam irradiated films exhibiting a lower decrease of the refractive index. Moreover, the diminution of the film thickness observed in the irradiated area can be interpreted as a consequence of a densification of the layer.

Nucl. Instr. and Meth. B (in press).

Interfacial Roughness of Multilayered TiN/ZrN Coatings

C.J. Tavares[1], L. Rebouta[1], E.J. Alves[2], B. Almeida[3], J. Bessa e Sousa[3], M.F. da Silva[2] and J.C. Soares[2,4]
[1] *Universidade do Minho, Departamento de Física, Campus de Azurém, 4810 Guimarães, Portugal*
Tel: +351-53-510151, Fax: +351-53-510153, E-mail: rebouta@fisica.uminho.pt
[2] *ITN, Departamento de Física, E.N.10, 2685 Sacavém, Portugal,*
[3] *Universidade do Porto, Departamento de Física, 4100 Porto, Portugal*
[4] *CFNUL, Av. Prof. Gama Pinto, 2, 1700 Lisboa, Portugal*

Abstract
TiN/ZrN multilayers with nominal modulation periods of 8.5, 12.5 and 16.5 nm and total thicknesses ranging between 60 nm and 1.5 μm were prepared by combined DC and RF magnetron sputtering. These coatings were deposited on polished Si substrates. The multilayer period was evaluated by X-Ray diffraction (XRD) in the low angle region. Several orders of multilayer reflections are present in these diffraction patterns, exhibiting a pronounced attenuation, which in turn indicates of a moderate degree of interfacial roughness. The interface roughness was studied as a function of the number of bilayers and of the modulation period by Rutherford backscattering spectrometry. An increase of the average interlayer roughness with the thickness and with the modulation period was observed

Nucl. Inst. Meth. B, (in press).

Improving the Thermal Stability of Photoresist Films by Ion Beam Irradiation

F.C. Zawislak[1], Irene T.S. Garcia[2], D. Samios[3], D.I. Baptista[1], P.F.P. Fichtner[4], E. Alves[5], M.F. da Silva[5] and J.C. Soares[5,6]

[1] - Institut de Física, UFRGS, Caixa Postal 15051-970 Porto Alegre, Brasil
[2] - PGCIMAT, UFRGS, Porto Alegre, Brasil
[3] - Instituto de Química, UFRGS, Porto Alegre, Brasil
[4] - Escola de Engenharia, UFRGS, Porto Alegre, Brasil
[5] - Instituto Tecnológico e Nuclear, Sacavém, Portugal
[6] - Centro de Física Nuclear, UL, Lisboa, Portugal

Abstract

The thermal stability of ion irradiated 1.7 μm thick AZ-1350J photoresist films was investigated using the RBS and ERDA techniques to measure the composition of the irradiated and annealed films. The films have been irradiated with He, N, and Ar ions at energies from 380 to 760 keV and fluences between 2×10^{15} and 10^{16} cm^{-2}. A considerable increase in the thermal stability of the He irradiated films is observed from ~200°C - when the non-irradiated film starts to decompose - to 400°C after irradiation. The FTIR spectroscopy and SEM observations were used to study the chemical structural changes and the surface morphology of the irradiated samples. The results are discussed ion terms of the energy density deposited by the ions, the large loss of H during irradiation, and the resulting increase in the cross-linking density.

Mat. Res. Soc. Symp. Proc. (in press).

Giant Magnetoresistance in Iron and Cobalt Implanted Silver Thin Films

C.M. de Jesus[1], J.G. Marques[1], J.C. Soares[1], L.M. Redondo[2], M.F. da Silva[2], M.M. Pereira de Azevedo[3], J.B. Sousa[3]

[1] *CFN, Universidade de Lisboa, Av. Prof. Gama Pinto, 2, P-1699 Lisboa Codex, Portugal*
[2] *Instituto Tecnológico e Nuclear, Estrada Nacional 10, P-2685 Sacavém, Portugal*
[3] *IFIMUP, Universidade do Porto, P-4150 Porto, Portugal*

Abstract

The magnetoresistive behaviour of granular thin films prepared by Fe and Co implantation in Ag thin films is reported. Ag thin films (~2000 Å) were implanted with Fe or Co at fluences up to $8x10^{16}$ at./cm^2. The magnetoresistive response obtained after implantation was found to increase with the implanted fluence. A further increase by a factor of 3-4 can be achieved annealing the films in a conventional furnace at 620 K under vacuum. The best value of the magnetoresistance obtained so far is 9% at 10 K for a film implanted with Co at a fluence of 8×10^{16} at./cm^2.

Mat. Res. Soc. Symp. Proc. (in press).

Physical and Mechanical Properties of $Ti_{1-X}Si_XN$ Films

F. Vaz*, L. Rebouta*, S. Ramos[#], A. Cavaleiro[#], M. F. da Silva[+] and J. C. Soares[+]
**Dep. Física, Universidade do Minho, Azurém, 4800 Guimarães, Portugal, Fax, 351-53-510153*
[#] Departamento Engª Mecânica, Universidade de Coimbra, 3030 Coimbra, Portugal
[+] ITN, Departamento de Física, E.N.10, 2685 Sacavém, Portugal.

Abstract
$Ti_{1-x}Si_xN$ coatings with $0 \leq x \leq 0.30$ and thicknesses ranging from 1.2 to 3.3 μm, were deposited onto polished high-speed steel substrates by r.f. reactive magnetron sputtering. The atomic composition of the samples was measured by Rutherford Backscatering Spectrometry (RBS) and the texture was determined by X-ray diffraction (XRD). Great improvements in hardness and adhesion behaviour were obtained when compared to TiN. Hardness results and adhesion behaviour as a function of the Si content will be shown and discussed. The $Ti_{.85}Si_{.15}N$ sample presented the best results with a hardness value of about 36 GPa and a critical load for total failure around 70 N.

Surf. and Coat. Techn., (in press).

The Potential of PIXE to Measure Multilayer Film Thickness Through Computer Simulation

Zhongning Dai[1], J.C. Soares[2,3], M.F. da Silva[3]
[1] *JAERI Takasaki, 1233 Watanuki, Takasaki, Gunma 370-12, Japan*
[2] *CFNUL, Univerity of Lisboa, Av. Prof. Gama Pinto 2, P-1699 Lisboa, Portugal*
[3] *Instituto Tecnológico e Nuclear, Est. Nac. 10, 2685 Sacavém, Portugal*

Abstract
In this paper a method concerning the measurement of film thickness using PIZE analysis trhough computer simulation is described. Because of continuous decreasing cross sections for X-ray production of irradiation with the energy of the incident beam decreasing, PIXE has some potentiality to measure film thickness through computer simulation. The ratios of intensities of X-rays are sensitive to the changes of energy and geometrical parameters, therefore by changing ion beam energy or the geometrical parameters; we can determine the film thickness to a good accuracy. This method doesn't require any kind of absolute calibration of experimental parameters, as only the ratios of X-ray intensities are taken into consideration. Therefore the inaccuracy in the procedure is only of statistical origin. A titanium film on molybdenum substrate is used as an example to the method. Preliminary result on a very thin Cr/Nd/Fe multilayer film on silicon substrate is presented to show the advantages and possible use of the method. More complex samples, such as multilayer samples with several lements in each layer, can be dealt with same means too.

Nucl. Inst. Meth. B, (in press).

The Effect of the Addition of Al and Si on the Physical and Mechanical Properties of Titanium Nitride

F. Vaz[*], L. Rebouta[*], M. Andritschky[*], S. Ramos[#], M. F. da Silva[+] and J. C. Soares[+],
[*] *Dept. Física, Universidade do Minho, Azurém, 4800 Guimarães, Portugal, Fax xx-351-53-510153,*
[#] *Dept. Engª Mecânica, Universidade de Coimbra, 2030 Coimbra, Portugal,*
[+] *ITN, Dept. Física, EN 10, 2685 Sacavém.*

Abstract
In this work it will be shown that the addition of Al and Si improve not only the oxidation resistance of TiN but also the adhesion behaviour. For this, $Ti_{1-x}Si_xN$ and $Ti_{1-y}Al_yN$ coatings with $0 \le x \le 0.3$, $0.38 \le y \le 0.81$ and thicknesses ranging from 1.2 to 5.6 μm, were deposited onto polished high-speed steel substrates by DC and RF reactive magnetron sputtering. The samples were oxidised in air for temperatures between 500 ºC and 900 ºC. The atomic composition of the samples was measured by Rutherford Backscatering Spectrometry (RBS) and the texture by X-ray diffraction (XRD). Adhesion behaviour was characterised by scratch test experiments. The results show a great improvement in oxidation resistance for both $Ti_{1-x}Si_xN$ and $Ti_{1-y}Al_yN$ systems when compared with that of TiN. For the $Ti_{1-y}Al_yN$ system, higher oxidation resistance was observed for y = 0.65 which is the composition were the best results for critical loads were observed. In the $Ti_{1-x}Si_xN$ system, best oxidation performance was observed for x = 0.3, but the highest critical loads were obtained for x = 0.15. The discussion of the failure mechanisms as a function of the x and y values will also be presented and discussed in some detail.

Comunication to: *International Conference on Advanced Materials and Processing Technologies, AMPT'97*, 21-25/7/97 Guimarães, Portugal,

---

Characterization of Titanium Silicon Nitride Films Deposited by PVD

F. Vaz[*], L. Rebouta[*], R. M. C. da Silva[#], M. F. da Silva[#] and J. C. Soares[#,+]
[*] *Dept. Física, Universidade do Minho, Azurém, 4810 Guimarães, Portugal*
[#] *ITN, Dept. de Física, E.N.10, 2685 Sacavém, Portugal*
[+] *CFNUL, Av. Prof.Gama Pinto, 2, 1700 Lisboa, Portugal*

Abstract
In recent years, nitride coatings have found widespread applications for tool and other hard surfaces. In this work the (Ti,Si)N system was investigated and some of its properties characterised. For this, (Ti,Si)N films with thicknesses ranging from 1 to 3.3 μm and different contents of Ti and Si were deposited onto silicon wafers and polished high-speed steel substrates by r.f. reactive magnetron sputtering technique. The atomic composition of the samples were measured by Rutherford Backscattering Spectrometry (RBS). $Ti_{1-x}Si_xN$ samples with 0×0.37 were produced. Regarding the structural properties, two cubic crystallographic structures were found, with lattice parameters of about a = 4.29 Å and 4.18 Å. The grain size evaluated by Fourier analysis of X-ray peaks ranges from 5 nm to 34 nm. Concerning the adhesion results, the $Ti_{.70}Si_{.30}N$ and $Ti_{.87}Si_{.13}N$ sample presented the best results in adhesion with a critical load for total failure around 115 N and 105 N, respectively.

Comunication to: *2nd European Topical Conf. on Hard Coatings*, 22-24/9/97, Lisboa, Portugal.

GMR Behavior of Granular Fe and Co Implanted Ag Thin Films

J.C. Soares[1,2], L.M. Redondo[2], C.M. de Jesus[1], J.G. Marques[1], M.F. da Silva[2], M.M. Pereira de Azevedo[3], J.A. Mendes[3], M.S. Rogalski[3] and J.B. Sousa[3]

[1] *CFNUL, Universidade de Lisboa, Av. Prof. Gama Pinto 2, P-1699 Lisboa Codex, Portugal*

[2] *Instituto Tecnológico e Nuclear, Estrada Nacional 10, P-2685 Sacavém, Portugal*

[3] *IFIMUP, Universidade do Porto, P-4150 Porto, Portugal*

Abstract

The magnetoresistive behaviour of granular thin films prepared by Fe and Co implantation in Ag thin films is reported. Ag thin films (~2000 Å) were deposited by evaporation or laser ablation onto Si and SiO_2 substrates and implanted with Fe or Co at fluences up to 10^{17} at./cm^2. The magnetoresistive response obtained after implantation was found to increase with the implanted fluence. An increase of the magnetoresistive response by a factor of 3-4 can be achieved after annealing the films in a conventional furnace at 620 K under vacuum. The best value of the magnetoresistance obtained so far is 9% at 10 K and 1.5% at room temperature for a film implanted with Co at a fluence of 8×10^{16} at./cm^2 and annealed at 620 K.

J. Vac. Sci. Tech. (submitted).

Giant Magnetoresistance Induced by Fe Ion Implantation in Ag Thin Films

M.M. Pereira de Azevedo[1], J.A. Mendes[1], M.S. Rogalski[1], J.B. Sousa[1], L.M. Redondo[2], M.F. da Silva[2], C.M. de Jesus[3], J.G. Marques[3], J.C. Soares[2,3]

[1] *IFIMUP and Physics Department of Fac. of Sciences, University of Porto, P-4150 Porto, Portugal*

[2] *Institute of Nuclear Technology, P-2680 Sacavém, Portugal*

[3] *CFNUL, University of Lisbon, 1699 Lisbon, Portugal*

Abstract

We report the observation of magnetoresistive behaviour in granular thin films prepared by Fe ion implantation in Ag films. These Ag films were previously deposited by pulsed laser ablation and evaporation onto glass and silicon substrates from 99.99 at.% pure Ag targets using a high vacuum chamber with a base pressure of $5\text{x}10^{-9}$ mbar. Laser fluences of 5 to 10 J/cm^2 were obtained from a KrF/Ne filled excimer laser (λ=248nm) operating at 50 Hz, resulting in low deposition rates of 0.5 to 2 Å/s to minimize local inhomogeneities in our films. The thicknesses of the deposited films (in the range 2000Å - 3000Å) were determined with a profilometer and confirmed by Rutherford Backscattering Spectrometry (RBS). These films were subsequently implanted with Fe ions, at doses of 1-6 x 10^{16} at/cm^2 with ion energy from 50-180 keV, using a Danfisyk 1090 High Fluence Ion Implanter. Iron ions were produced in a CHORDIS ion source, using a sputter ion target. The magnetoresistive response was found to be enhanced by increasing the ion implantation dose. High angle X-ray diffraction patterns for the ion implanted films show the (111) and (200) Bragg reflections typical of fcc Ag crystal lattice, slightly shifted towards larger angles (with respect to the standard spectra for bulk samples) which indicate the presence of compressive strain in such metallic matrices. Mössbauer conversion electron spectroscopy (CEMS; local probe) shows the formation of magnetic and non-magnetic Fe clusters. Magnetisation data reveal a dominant superparamagnetic behaviour of the Fe clusters consistent with the CEMS results. A significant magnetoresistive response (GMR) of these films, was obtained by increasing the implantation doses reaching values of ~1-2 % at T=10K.

J. Mat. Proc. Tech. (submitted).

OTHERS

Characterisation of Alpha Sources Prepared by Direct Evaporation Using Rutherford Backscattering Spectrometry

P. Blanco[a], A. Martín[a], F. Vera[a], J.L. Ferrero[b], C. Roldán[b,c], D. Juanes[b], F.J. Ager[d], M.F. da Silva[e], J.C. Soares[e,f]

[a] *Dep. de Física, Universidad de Extremadura, Av. de Elvas s/n, 06071 Badajoz, Spain*
[b] *ICMUV, Universitat de València. Av. Dr Moliner 50, 46100 Burjassot, Valencia, Spain*
[c] *Dep. de Física Aplicada, Univ. de València. Av. Dr Moliner 50, 46100 Burjassot, Valencia,Spain*
[d] *Dep. de Física Atómica Molecular y Nuclear, Univ. de Sevilla. P.O. BOX 1065, 41080 Sevilla,Spain*
[e] *ITN, Estrada Nacional 10, 2685 Sacavém, Portugal*
[f] *CFNUL, Universidade de Lisboa, Av. Prof. Gama Pinto 2, 1699 Lisboa, Portugal*

Abstract
Standardization of solutions containing alpha emitting nuclides by direct evaporation onto metal supports is a widely used technique due to its simplicity in providing good quantitative results. In order to avoid inhomogeneity in the deposition surface, polished stainless steel disks and a spreading agent are generally used. These sources are usually measured by alpha spectrometry using passivated implanted silicon detectors. The resolution of the source is a measure of the thickness and homogeneity of the evaporated layer. Rutherford backscattering of He+ and H+ was here used to measure directly this thickness and homogeneity. The results were in agreement with semiconductor detectors alpha spectrometry.

Nucl. Instr. and Meth. ***B*****132** (1997) 501-506.

Semipermeable Membrane to Retain Platinum Atoms in the Electrodeposition Process of Alpha Spectrometry Sources

J.L. Ferrero[a], A. Martín[b], C. Roldán[a,c] , F. Vera[b], M. F. da Silva [d], J. C. Soares [d,e], D. Juanes[a]

[a] *ICMUV, Universitat de València. Av. Dr Moliner 50, 46100 Burjassot, Valencia, Spain*
[b] *Dep. de Física, Universidad de Extremadura, Av. de Elvas s/n, 06071 Badajoz, Spain*
[c] *Dep. de Física Aplicada, Univ. de València. Av. Dr Moliner 50, 46100 Burjassot, Valencia,Spain*
[d] *ITN, Estrada Nacional 10, 2685 Sacavém, Portugal*
[e] *CFNUL, Universidade de Lisboa, Av. Prof. Gama Pinto 2, 1699 Lisboa, Portugal*
[f] *Dep. de Física Atómica Molecular y Nuclear, Univ. de Sevilla. P.O. BOX 1065, 41080 Sevilla,Spain*

Abstract
In earlier work alpha sources electrodeposited on stainless steel backings were analyzed by X-ray fluorescence (XRF) and Rutherford backscattering spectrometry (RBS) finding that during the electrodeposition process large quantities of platinum from the anode were deposited on the cathode surface jointly with the actinides. In the present work, a method to retain platinum atoms using an electrodeposition cell with a semipermeable membrane located between anode and cathode is proposed and tested. The XRF and RBS of alpha sources electrodeposited using this method show that there is less platinum on the stainless steel backing, thereby improving the quality of sources to be measured by alpha spectrometry.

Appl. Radiat. Isot. (in press).

Ion Beam Analysis and Alpha Spectrometry of Sources Electrodeposited on Several Backings

J.L. Ferrero[a], F. Vera[b], A. Martín[b], C. Roldán[a,c] ,M. F. da Silva [d], J. C. Soares [d,e], F.J. Ager[f], D. Juanes[a], P. Rubio[b]

[a]*ICMUV, Universitat de València. Av. Dr Moliner 50, 46100 Burjassot, Valencia, Spain*
[b]*Dep. de Física, Universidad de Extremadura, Av. de Elvas s/n, 06071 Badajoz, Spain*
[c]*Dep. de Física Aplicada, Univ. de València. Av. Dr Moliner 50, 46100 Burjassot, Valencia,Spain*
[d]*ITN, Estrada Nacional 10, 2685 Sacavém, Portugal*
[e]*CFNUL, Universidade de Lisboa, Av. Prof. Gama Pinto 2, 1699 Lisboa, Portugal*
[f]*Dep. de Física Atómica Molecular y Nuclear, Univ. de Sevilla. P.O. BOX 1065, 41080 Sevilla,Spain*

Abstract

Alpha sources of several activities were prepared by electrodeposition of natural uranium onto four different backings: stainless steel, Ni, Mo and Ti. The influence of the activity, the type of backing, and the process of heating the source on the energy resolution of the spectra were investigated using alpha spectrometry and Rutherford backscattering spectrometry (RBS) techniques. Diffusion profiles of the radioactive deposits in the backings were obtained from RBS and related to the results using alpha spectrometry.

Nucl. Instr. and Meth. B (in press).

Current Work

Metastable Alloys in Al-Cr System Produced by Ion Implantation and Laser Processing

L. Prudêncio[1], L.M. Redondo[1], J. Rocha[1], R.C. da Silva[1], O. Conde[2], R. Vilar[3]
[1] *Instituto Tecnológico e Nuclear, Estrada Nacional 10, P-2685 Sacavém, Portugal*
[2] *Dep. Física, FCUL - Ed. C1, Campo Grande, 1700 Lisboa, PORTUGAL*
[3] *Dep. Materiais - IST, Av. Rovisco Pais 2, 1700 Lisboa, PORTUGAL*

Metastable Al-Cr surface alloys were produced by high fluence Cr implantations into polycrystalline aluminium, under different conditions of temperature and fluence. The implantations were performed with 140 keV Cr ions to fluences of 1, 2 and 5×10^{17}/cm^2, at RT, and with 2×10^{17}/cm^2 at 250 and 510 ºC.

In order to compare ion implanted and laser alloyed systems, Cr was also deposited by vacuum evaporation onto Al discs.

The samples were characterised by Rutherford backscattering spectrometry (RBS) and Glancing incidence X-ray diffraction, (GIXRD).

At present, RBS and GIXRD results show that:

- doses of 5×10^{17} Cr^+/cm^2 readily produce an extended layer of the $Al_{13}Cr_2$ intermetallic compound, more than 0.5 μm in thickness (Al equivalent);
- for doses of 2×10^{17} Cr^+/cm^2 the $Al_{13}Cr_2$ compound does not form up to temperatures of 250 ºC, but it is already formed at 510 ºC;
- the $Al_{13}Cr_2$ compound is not detected after vacuum annealings of the 1 and 2×10^{17} Cr^+/cm^2 implanted Al, at temperatures of 300 ºC, but is detected after annealing of the 2×10^{17} Cr^+/cm^2 implanted Al at 400 ºC;
- the presence of other intermetallic compounds, richer in Cr - *e.g.* $Al_{11}Cr_2$ or $Al_{84}Cr_{16}$ - has also been detected along side $Al_{13}Cr_2$, but in very much lower concentrations. The relative amount has been found to change through annealings, the Al richer $Al_{13}Cr_2$ growing at the expense of the Cr richer phases.
- GIXRD spectra recorded while the samples are rotating around an axes perpendicular to the sample surface, and through its center, differ from spectra taken under static conditions. This points to the the existance of texture in the intermetallic compound layer.

Work is currently under way in order to determine the formation temperature of $Al_{13}Cr_2$, and its relation to the implantation dose.

Also, the stability and transformation mechanisms of the different alloys as a function of temperature are currently under study.

Finally, an assessment of the mechanical properties and of the wear and corrosion resistance of these materials will be made in order to evaluate possible applications.

Characterisation of Metal-oxide Silicate-glass Multilayers by Ion Beam Analysis

P.R. Gordo, E. Alves, L.C. Alves, R.C. da Silva, A. P. Gonçalves, J. Marçalo, M.F. Araújo and A. Pires de Matos
Instituto Tecnológico e Nuclear, Estrada Nacional 10, P-2685 Sacavém, Portugal

Surface coating of glass with metal oxides has many useful applications. Among them we can refer the sun-shielding glass, heat mirrors, antireflection layers, laser light frequency splitters, colour conversion filters, rear-view mirrors, dichroic glasses used in art applications, etc.. The alternate deposition of thin layers of oxides with different refraction indexes gives origin, by interference, to glasses of selective transmission. Examples of metal oxides used are TiO_2, ZrO_2, V_2O_5, Nb_2O_5 and Ta_2O_5. Multiple layers of silicate glass-metal oxides are being deposited onto glass, to produce colours that change with the angle of observation and depend on the refraction index of the materials.
One of the techniques used for multilayer preparation is vacuum deposition of the metal oxides using an electron beam. This allows the thickness of each layer to be easily controlled. In order to check the success of the deposition and to help optimise it several analytical methods have been used.
The analysis of dichroic glass samples made by multilayer deposition of two different metal oxides onto glass, showed us that whereas X-ray fluorescence and Laser Ablation Fourier transform ion cyclotron resonance mass spectrometry can give a quick answer about the elemental analysis of the major oxides deposited, Rutherford Backscattering combined with PIXE analysis allows the determination of the composition and thickness of the different layers.
With these techniques we can obtain the correlations between modulation period, composition and spectral response (i.e. colour), and thus finely tune the production of n-fold double layered structures with colours covering the full wavelength range of the optical spectrum.

Characterisation of Mg Surfaces Implanted with Reactive Elements Fe, Y, and La

C.M. Lobo, R.C. da Silva
Instituto Tecnológico e Nuclear, Estrada Nacional 10, P-2685 Sacavém, Portugal

Although magnesium has approximately the same elasticity-to-density ratio as aluminium, its chemical reactivity is much higher, namely in what concerns the oxidation behaviour. This imposes severe limitations to its widespread use, *e.g.* in the transportation industries (terrestrial, in motor vehicles, aeronautical, etc.). In an effort to help understanding the oxidation behaviour of Mg surfaces, the reactive elements Fe, Y and La were introduced by ion implantation with doses in the range 10^{16}-5×10^{17}/cm^2. The evolution in air of the resulting concentration profiles and O distributions were followed by RBS.

In order to understand the transport behaviour of the implanted species, annealing experiments were also conducted.

The results obtained so far show that all the changes detected in the depth profiles can be correlated with oxygen intake. However a similar behaviour of Fe and Y was found, as opposed to that for La. The RBS profiles indicate that Fe and Y implanted Mg suffer extensive

oxidation at room temperature, along with extended diffusion of the implanted elements. In contrast, no signicant diffusion of La in the Mg lattice was found, even after annealings up to 450 ºC.

Study of the Formation of Al-Fe Intermetallic Compounds by High Fluence Ion Implantation of Fe into Al

L.M. Prudêncio, R.C. da Silva and C. Waerenbourgh
Instituto Tecnológico e Nuclear, Estrada Nacional 10, P-2685 Sacavém, Portugal

Polycrystalline Al was implanted with $3\times10^{17}/cm^2$ Fe^+ ions of 150 keV energy, and analysed by Rutherford Backscattering (RBS) technique. RBS spectra showed a flat topped Fe profile, extending down to 0.20 µm with a concentration ratio, consistent with a homogeneous composition of the type $Al_{13}Fe_4$ or Al_7Fe_2. In order to identify if such a intermetallic compound has formed, $10^{16}/cm^2$ $^{57}Fe^+$ ions of the same energy were then implanted, allowing to use the Mössbauer Spectroscopy (MS) technique to check for the existance of Al-Fe intermetallics. Although the MS spectra obtained indicates that $Al_{13}Fe_4$ exists in the implanted region, examination by Glancing Incidence X-Ray Diffraction (GIXRD) failed to confirm this.
The aluminium was examined by PIXE (Proton Induced X-ray Spectrometry) in order to detect low level concentrations of natural Fe in the bulk, that might contribute to the MS spectrum, namely through $Al_{13}Fe_4$ pre-existing inclusions. The concentration levels found were of the order of 0.2%, and the results obtained with unimplanted aluminium indicate that there is no significant bulk Fe contribution for the MS spectra recorded. New experiments are being planned in order to clarify these findings.

Characterisation of Thin YBaCuO:PrBaCuO Multilayers by Means of RBS under Large Tilts

R.C. da Silva, E. Alves and G. Bonfait
Instituto Tecnológico e Nuclear, Estrada Nacional 10, P-2685 Sacavém, Portugal

Stacks of very thin layers of $\{Y_1Ba_2Cu_3O_7{:}Pr_1Ba_2Cu_3O_7\}$ high-T_c supercondutors were analysed by RBS under large tilts in order to measure layers thicknesses and deviations to the stoichiometry relations. The intent of the experiments is to have an analysis tool that will serve to control and optimize the routine deposition of thin YBaCuO:PrBaCuO high-T_c superconductor multilayers.

By using very high incidence angles we are able to routinelly resolve up to 10 individual double layers of $\{Y_1Ba_2Cu_3O_7{:}Pr_1Ba_2Cu_3O_7\}$ with typical thicknesses of 20 nm.

The Ion Microprobe Facility

M.R. da Silva[1,2], M. Breese[1], L.C. Alves[2], L. Redondo[2], M. F. Silva[2], A. Melo[1], J.C.. Soares[1,2]
[1]*Centro de Física Nuclear, Univ. Lisboa, Av. Prof. Gama Pinto 2, P-1699 Lisboa Codex, Portugal*
[2]*Instituto Tecnológico e Nuclear, Estrada Nacional 10, P-2685 Sacavém, Portugal*

The Ion Beam Laboratory, located at Sacavém, is one of the several Laboratories of the "Centro de Processamento e Caracterização de Novos Materiais" created under the CIENCIA programme, involving the existing collaboration between two institutions – CFNUL and ITN. The upgrading of this Laboratory continued in 1997, with the construction of an ion microprobe to be installed in one of the 3 MeV Van de Graaff Accelerator beam lines.

The aim of this project is to provide a versatile instrument with a spatial resolution of about 1μm in order to allow the study of problems in a wide variety of fields. Many of its uses are for the analysis of microelectronics devices and study of crystalline quality and defects of materials.

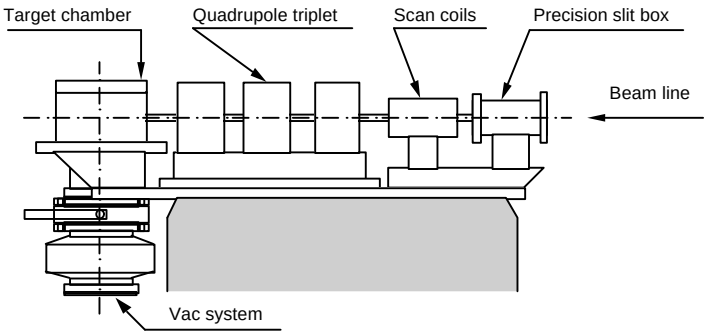
The microprobe with the set up shown bellow in the block diagram is presently being assembled.

Some of the components – scan coils quadrupole triplet and x-y-z manipulator were acquired directly from a firm in Oxford.

The other hardware components, namely the vacuum connections, chamber, flanges, detectors holders, slit boxes, concrete table were locally designed and built.

The vacuum tests were already carried out successfully.

Preliminary tests on the quality and size of the beam spot will start soon.



Silicides Formation by Ion Implantation

A.R. Ramos[1], M.R. da Silva[1], M.F. da Silva[2], J.C. Soares[1,2], A.A. Melo[1]
[1]*Centro de Física Nuclear, Univ. Lisboa, Av. Prof. Gama Pinto 2, P-1699 Lisboa Codex, Portugal*
[2]*Instituto Tecnológico e Nuclear, Estrada Nacional 10, P-2685 Sacavém, Portugal*

Ion implantation into semiconductors for doping is a well-established and technologically important process in microelectronics. Presently, a novel technique -Ion Beam Synthesis- has been under investigation and development, and in some cases is already successfully employed for device fabrication (for instance, IBS of insulating layers: SIMOX process). Recent work has demonstrated that high quality, epitaxial buried silicides can be fabricated by ion beam synthesis.

In order to investigate the process window for the formation of different silicides we have implanted several ions with different energies and doses into monocrystalline silicon ((111) and (100) oriented) held at different temperatures.
In particular, we have studied the formation of iridium, platinum, hafnium, tungsten and cobalt silicides. The results have been monitored by RBS/Channelling as well as by surface resistivity analysis.
Figure 1 shows typical channelled and random spectra of Co implanted silicon. High current density during implantation (7 $\mu A/cm^2$) resulted in good epitaxial quality for the cobalt disilicide layer immediately after implantation. Resistivity measurements show an excellent 37.7 $\pm$ 5.3 $\mu\Omega$cm value for the as implanted samples, which decreases to 21.8 $\pm$ 3.0 $\mu\Omega$cm after annealing. We have shown that with RBS/Channelling we can find no difference in the silicide quality between the two common annealing procedures: 600ºC for one hour + 1000ºC for 1/2 hour or 900ºC for 1 hour.
Figure 2 shows random and channelled spectra of W implanted silicon. W concentration is sub-stoychiometric (~20%), resulting in higher resisitivity and possibly polycrystalline island forming. Resisitivity measurements and annealing procedures are under way.

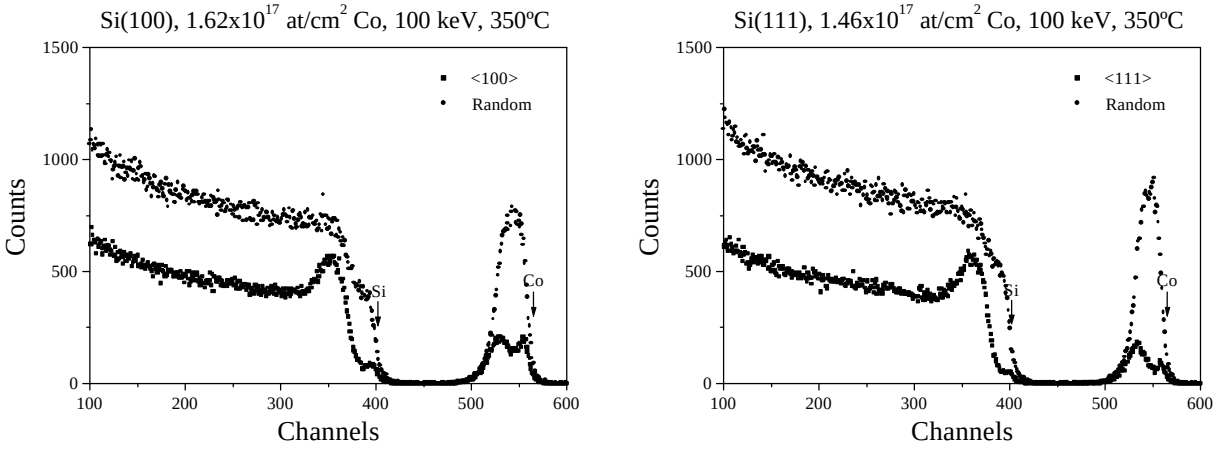


Fig.1 Random and channelled spectra for Co implanted silicon (as implanted samples).

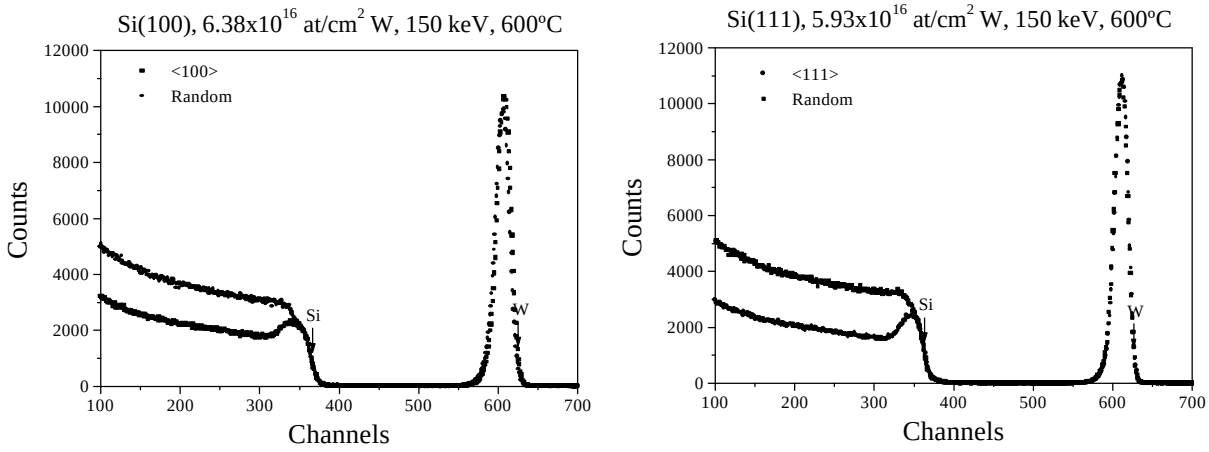


Fig. 2 Random and channelled spectra for W implanted silicon (as implanted samples).

This year we hope to finally tune the process for hafnium and tungsten silicide formation and begin investigating other silicides (chromium, lanthanum).