Nuclear Solid State Physics Using Ion Beams

Introduction

The activity of the group was carried out with a team from ITN and from the Nuclear Research Centre of Lisbon University.

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

Some of the achievements in 1998 are:

The upgrading of the Ion Beam Laboratory with the installation of the ion beam microprobe;

The work in the the European Fusion Program through the sub-task: “Be binary pebble bed electrical resistivity measurements” and “Precise Ion Beam Analysis of advanced materials for nuclear fusion reactors”;

The European completion of the CECA project “Optimisation of the High Temperature Oxidation Behaviour of Conventional Stainless Steel by Surface Applied Rare Earth Elements”. Four different modification procedures were used to enhance the oxidation resistance of AISI-304 stainless steels: spray coating, pyrolisis of aerosols, ion implantation and ion exchange. All the procedures lead to improvements in the oxidation resistance at 1173 K using Ce and La as reactive elements. Mass gain during oxidation was reduced by one order of magnitude;

The up-grade of the e^--γ spectrometer that allows for the first time e^--γ PAC experiments at temperatures as low as 10 K;

The research on high T_C Superconductors: it was observed that implanted Hg goes into unique lattice sites in YBCO, being stable under vacuum annealing up to 450 ºC.
Non-stoichiometric oxygen doping has been also studied at an atomic scale, being further discovered that a microscopic charge asymmetric transition along the O(2)-Hg-O(2) rows occurs below 100 K;

New channeling results, partly in combination with hyperfine interaction studies, on the $LiNbO_3$ system improved the understanding of the intrinsic defect structure and the interaction between dopants and the lithium niobate host lattice;

The GaN studies: Er implantation in low dose replaces Ga in the GaN lattice and the amorphization level is reached for a dose of 5×10^{15} Er^+/cm^2. Coimplantation with O increases the optical activity of erbium;

The doping of semiconductors: ion implantation of phosphorus ions to dope amorphous and micro crystalline silicon with the aim of developing a low temperature, self-aligned process for forming n^+ contacts to top-gate thin film;

The sapphire studies: Platinum implanted into sapphire forms crystalline metallic precipitates aligned along <0001> axis during the annealing in oxygen atmosphere;
The damage production during the implantation of Fe in sapphire and its recovery are influenced by the crystal orientation. This anisotropic behaviour is also observed for the Fe ions during the annealing;

The rutile studies: hot implantations (870 K) of rutile (TiO_2) with W^+ ions results in a dramatic increase in RT conductivity of the implanted layer. Similar results were obtained for vacuum annealed samples. This gives evidence that more than one transport mechanism is active.

Research Team

Researchers –	10 *	(10 PhD)
Research Students –	5	
Undergraduate Students –	2	
Technicians –	2	

* 3 from university, 2 post-doctoral and 1 post-doctoral from RPI.

Publications

Journals –	36	and 17 in press
Proceedings –	8	and 4 in press

Theses:

MSc. –	1
Lic. –	1

10^3 PTE

Expenditure:	**14 236**
Missions:	2 133
Other Expenses:	4 795
Hardware & Software:	322
Other Equipment:	6 986

10^3 PTE

Funding:		**19 338**
OE/ITN	OF	2 195 (1)
	PIDDAC	1 347
External Projects:	1997	6 150 (2)
	1998	5 800
Others		3 846

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(1) Grants 1.379

(2) Funding not used in 1997

INSULATORS

Large Tunneling Magnetoresistance Enhancement by Thermal Anneal

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Abstract

Spin tunnel junctions with tunneling magnetoresistance of 36.5% ± 0.5%, resistance-area product of 35 ± 6 kΩ × μm^2, and junction area between 6 and 75 μm^2 were fabricated. The barrier height is 2.5 ± 0.3 eV and the barrier thickness is 7.7 ± 0.3 Å. Large tunneling magnetoresistance (TMR) values are obtained by vacuum anneal (at temperatures from 100 to 240 ºC for over 5 h) of junctions prepared with as-deposited TMR of 21% ± 1.7%, and an as-deposited resistance- area product of 25 ± 6 kΩ × μm^2. Two regimes occur during anneal. The first one occurs for anneals up to 200 degrees C where TMR and junction resistance increase, but the barrier parameters are unaltered. The second occurs above 200 degrees C, where TMR increases faster, together with an increase in barrier height. At 240 degrees C, TMR starts to decrease. Rutherford backscattering analysis indicates an asymmetry in the oxygen distribution in the as-deposited barrier. The oxygen distribution becomes homogeneous for anneals above 150 ºC.

Appl. Phys. Lett. **73** (1998) 3288-3290.

Photoluminescence and Rutherford Backscattering Spectrometry Study of Ion-Implanted Er^{3+}-doped $LiNbO_3$ Planar Waveguides

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(4) *Department of Chemistry, Physics and Environmental Science, University of Sussex, Brighton, United Kingdom*

Abstract

In this work we present a study of Er^{3+}-doped $LiNbO_3$ ion-implanted planar waveguides by laser spectrometry and Rutherford backscattering spectrometry (RBS). RBS measurements for both random and channeling conditions were carried out in order to investigate the crystal quality of the original crystals and their respective waveguides. The angular dependences of the Nb and Er yields were compared and a non-substitutional fraction of Er^{3+} ions was found in both the bulk crsytal and the waveguide. Some deterioration of the crystal quality in the waveguide compared to the bulk was also detected. Laser spectroscopy was used to obtain information about the optical properties of the Er^{3+} ions within the waveguides and to compare

them with the properties of the Er^{3+} ions in the bulk. The results obtained show that the spectroscopic properties of the waveguide Er^{3+} ions are essentially the same as those of the Er^{3+} ions in the bulk, except a slight broadening of the bands and some changes in the relative intensities.

J. Phys.: Condens. Matter **10** (1998) 3275.

Sapphire (α-Al_2O_3) Behaviour under Heavy Ion Implantation

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Abstract

The surface region of sapphire was modified by the implantation of heavy ions. Single crystalline α-Al_2O_3 samples were implanted at room and liquid nitrogen temperatures with energies in the range of 100 keV to 200 keV. Several fluences have been implanted in order to study the evolution of the implantation damage and the formation of new microstructures. The amorphization and the damage recovery was observed to be strongly dependent on the chemical nature of the implanted ions. For example, while fluences of 1×10^{15} Hg^+/cm^2 were enough to amorphize the implanted region we need to implant a fluence one order of magnitude higher of Pt to obtain the same result. The implanted metallic ions occupy manly substitutional Al sites in the sapphire corundum structure while the rare earth ions go to the free octahedral site along the c-axis. Post-implantation annealing at high temperature allows the complete recrystallization of the amorphous region and induces the redistribution of the implanted ions decreasing the maximum peak concentration. During this process the formation of metallic precipitates epitaxially aligned with the c-axis of the α-Al_2O_3 structure are observed.

Proceedings of *OECD/NEA Workshop on Ion and Slow Positron Beam Utilization*, Costa da Caparica, 15-18 Sept. 1998.

Combined Ion Beam and Hyperfine Interaction Studies of $LiNbO_3$ Single Crystals

J.G. Marques(1,3), A. Kling(1), L. Rebouta(2), M.F. da Silva(3), J.C. Soares(1,3)
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Abstract

A review of recent studies of $LiNbO_3$ crystals doped with Hf and Mg,Hf combining high precision RBS/channeling, PIXE/channeling and hyperfine interaction techniques is presented. The lattice location of Hf was found to depend strongly on the dopant concentration, crystal stoichiometry and Mg-codoping level. At low concentrations Hf occupies Li sites in congruent crystals, while it occupies both Li and Nb sites for higher doping levels or in near-stoichiometric crystals. Co-doping with Mg also forces a split location of Hf in Li and Nb sites and when the MgO amount exceeds 4.5 mol% Hf occupies only Nb sites. Neutron irradiation of these crystals displaces Hf from its initial lattice site and leads to a strong decrease of the Nb

site fraction. The results are discussed in the framework of the Li and Nb vacancy models currently proposed in the literature for the defect structure of $LiNbO_3$.

Proceedings of *OECD/NEA Workshop on Ion and Slow Positron Beam Utilization*, Costa da Caparica, 15-18 Sept. 1998.

Electrical Conductivity in Ion Implanted TiO_2 Single Crystals

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Abstract

Rutile with a band gap of 3 eV and a conductivity of $\sigma < 10^{13}\Omega^{-1}cm^{-1}$ in the pure state can readily be n-doped to about $10^{19}/cm^3$ with many cation impurities including Nb, Ta, W, etc. which are substitutional (on Ti-lattice sites) under oxidation conditions. The conductivity can be changed by orders of magnitude in a reducing atmosphere or by radiation damage. In the following study we compare the effects of Xe- and W-ion implantation on the electrical conductivity of TiO_2 to determine if the increase of σ is due to defect doping and/or impurity doping.

The conclusion can be drawn that the electrical conductivity at room temperature is mainly controlled by the induced damage in the rutile structure. If there were any contribution of the extra charge to the electrical conductivity, it would be very weak. The extra charge is mainly compensated by oppositely charged defects in the rutile structure, which are created during the implantation process.

Proceedings of *Int. Conf. on Ion Implantation Technology, IIT'98,* Kyoto, Japan, 1998.

Double Barrier Spin Dependent Tunnel Junctions with an Intermediate Ferromagnetic Layer

R.C. Sousa(1), P.P. Freitas(1,2), T.T. Galvão(1,2), J.J. Sun(1), V. Soares(1), A. Kling(3), M.F. da Silva(4) and J.C. Soares(3)

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Abstract

A new spin dependent tunnel junction configuration using two insulator barriers with an intermediate ferromagnetic layer is described. The junction structure is bottom electrode/insulator/intermediate ferromagnetic/insulating/top electrode. The electrodes are CoFe layers pinned by TbCo or MnRh exchange films. The insulator is Al_2O_3 obtained by RF plasma oxidation of a thin Al film. Co, NiFe and CoFe intermediate ferromagnetic layers were used. In one of the three types of fabricated structures spin dependent tunneling is observed through the two Al_2O_3 insulating barriers. The measured tunneling magnetoresistance (TMR)

was 7%. for most double junctions studied however, only one of the barriers seems to be active, either the top or bottom one, TMR values up to 18%. Rutherford Backscattering (RBS) analysis is used to check the oxidation level of the two barriers. For the case where double tunneling was observed, the TMR falls to half its value for a bias above 500 mV and an asymmetry in the forward and reverse bias behavior was found. Conductance measurements at room temperature (RT) exhibit the usual parabolic curve as for single barrier junctions.

Mater. Res. Soc. Symp. Proc., in press.

Formation of Coherent Precipitates of Platinum in Sapphire

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(3) *CFNUL, Av. Prof. Gama Pinto, 1600-Lisboa, Portugal*

Abstract

The surface region of sapphire was modified by implantation of Pt ions. Single crystalline α-Al_2O_3 samples were implanted at room temperature with 160 keV Pt ions to fluences from 1×10^{14} to 5×10^{16} $Pt^+/cm^{.2}$. For doses up to 10^{15} Pt^+/cm^2 80% of the implanted ions occupy substitutional sites in the Al sublattice. The increase in dose makes the implanted region highly damaged. The amorphization is observed for doses higher than 10^{16} Pt^+/cm^2. During vacuum annealings at 1200 ºC the regular fraction of Pt decreases and most of the damage is recovered. Annealing of 5×10^{16} Pt^+/cm^2 implanted α-Al_2O_3 at the same temperature but in an oxidizing atmosphere allows the complete recrystallization of the amorphous region and induces the formation of Pt precipitates as found from glancing incidence X-ray diffraction (GIXRD) measurements. Detailed angular scans through the main axial and planar directions show that the metallic precipitates are aligned with the c-axis of the α-Al_2O_3 structure.

Nucl. Instr. and Meth. B, in press.

Structural Defects in Congruent and Near-Stoichiometric $LiNbO_3$

J.G. Marques**1)*****, A. Kling*****1)*****, J.C. Soares*****1)*****, M.F. da Silva*****2)*****, R. Vianden*****3)*****, K. Polgár*****4)*****, E. Diéguez*****5)** ***and F. Agulló-López*****5)**
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Abstract

A comparative study of $LiNbO_3$ single-crystals with congruent and near-stoichiometric compositions was performed using hyperfine interactions and ion beam techniques. Our data show that the structure of congruent $LiNbO_3$ cannot be completely described by the models which assume only vacancies and Nb antisites. Additional disorder has to be considered both at the Li and Nb sites. The additional defects are cation stacking inversions, in which the cation

sequence coincides locally with the one of ilmenite-like $LiNbO_3$. We suggest that these stacking inversions are associated with the Nb antisites necessary for charge compensation in Li-deficient material.

Radiation Effects and Defects in Solids, in press.

Lattice Location and Annealing Behaviour of Pt and W Implanted Sapphire

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Abstract

Sapphire (α-Al_2O_3) single crystals were implanted with different doses of Pt and W ions in the range of 1×10^{14} at/cm^2 to 5×10^{16} at/cm^2 at room temperature. Detailed angular scans through the main axial directions show that up to10^{15} at/cm^2 fluences about 80% of the W and Pt ions are incorporated into substitutional or near substitutional lattice sites. Below the amorphization threshold implantation damage show a double peak structure which anneals out partially at low temperature (800 ^{o}C). Amorphization of the implanted region starts for doses of the order of 1×10^{16} at/cm^2. The amorphous layer regrowths epitaxially in vacuum at 1100 ^{o}C, with a velocity of 3 Å/min and stops when the crystalline/amorphous interface reaches the region of maximum Pt concentration. When the annealing is done at ambient atmosphere the damage recovers completely at 1100 ^{o}C even for doses of the order or 5×10^{16} Pt^+/cm^2 leading to the formation of Pt precipitates.

Nucl. Instr. and Meth. B, in press.

Effects of Crystal Orientation on Damage Accumulation and Post-Implantation Annealing for Iron Implantation Into Sapphire

Carl J. McHargue[1], E. Alves[2], M. F. da Silva[2] and J. C. Soares[2]
[1]*- University of Tennessee, Knoxville, TN, USA,*
[2]*- Nuclear and Technological Institute, Sacavem. Portugal*

Abstract

Some studies have suggested that the rate of damage accumulation is significantly affected by the orientation in strongly anisotropic crystals such as sapphire. However a direct comparison of orientation effects on post-implantation annealing kinetics has not been reported.
In the current study, damage accumulation and post-implantation recovery of sapphire samples having the a-axis normal to the surface were compared to previously reported results for samples having the c-axis normal. Iron was implanted at room temperature to fluences of 1x10^{16}, 4x10^{16} 1x10^{17} and 5x10^{17} ions/cm^2 with an energy of 160 keV. Lattice disorder at the peak damage position determined from Rutherford backscattering-channeling measurements increases linearly with the fluence up to 1x10^{17} ions/cm^2 without amorphization. This indicates an higher amorphization threshold in terms of implanted fluence compared to the c-cut samples. The implanted region became completely disordered with the implantation of 5x10^{17} ions/cm^2 and electrical measurements indicated that a metallic-like surface layer had formed.

Damage recovery was studied after annealing for one hour in an oxidizing atmosphere at 800 and 1100° C. For fluences up to $4x10^{16}$ ions/cm^2 most of the damage was removed during the annealing at 1100° C and no change was observed in the Fe profile contrary to what is reported for (0001) samples. With the increase of the fluence the damage recovery is strongly inhibited, after the first annealing and the Fe starts to outdiffuse forming a double peaked RBS spectrum. The implanted region of the sample with $5x10^{17}$ ions/cm^2 turns into a Fe oxide layer with the complete segregation of aluminum.

Nucl. Instr. and Meth. B, in press.

Incorporation of Hexavalent Impurities into $LiNbO_3$

***A. Kling*[1]*, J.G. Marques*[1]*, M.F. da Silva*[2]*, M.D. Serrano*[3]*, E. Diéguez*[3]*, J.A. Sanz-García*[3]*, J. García-Solé*[3]*, F. Agulló-López*[3] *and J.C. Soares*[1]**

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Abstract

The influence of the valence state on the lattice location of impurities in lithium niobate $LiNbO_3$ crystals was investigated using hexavalent dopants (W^{6+} and U^{6+}) introduced by doping of congruent melt. The distribution coefficients were determined by Rutherford Backscattering Spectrometry to be k=0.2 for W and k=0.07 for U. Channeling studies showed that the hexavalent impurities are located on the Nb site in both cases. The preferential occupation of Nb sites suggests that the formation of Li vacancies is favored. This result is a strong evidence for the Li-vacancy defect model for congruent $LiNbO_3$.

Radiation Effects and Defects in Solids, in press.

Simulation of Channeling in Crystals with Defects Using the CASSIS Code

***A. Kling*[1]*, J.C. Soares*[1] *and M.F. da Silva*[2]**

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[2] *Instituto Tecnológico e Nuclear, Sacavém, Portugal*

Abstract

The concepts for the introduction of defects into the Monte Carlo simulation code CASSIS are discussed. The feasibility of the code to describe correctly effects on the channeling of light ions in cubic crystals containing point defects and dislocations is demonstrated for several examples. Calculations for intrinsic defects in the complex $LiNbO_3$ structure indicate that channeling measurements combined with Monte Carlos simulations yield a valuable contribution to the solution of the problem of stoichiometry related defects in this material.

submitted to *Nucl. Instr. Meth. B*

Electric Field Gradients at the ^{111}In Site in the Ferroelectric and Paraelectric Phases of $LiTaO_3$

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[2) *Institut für Kern- und Strahlenphysik, Universität Bonn, Bonn, Germany*

Abstract

The temperature dependence of the electric-field gradient of ^{111}Cd in single crystalline $LiTaO_3$ was studied from room temperature to 1040 K in the ferroelectric and paraelectric phases. The data taken at room temperature show unambiguously the presence of two quadrupole interaction frequencies ν_{Q1} = 230 MHz and ν_{Q2} = 242 MHz, with non-zero asymmetry parameters, while above the Curie temperature (T_C = 878 K) the data are well described by a unique frequency. The electric field gradient shows an usual temperature dependence, increasing approximately in a linear fashion until T_C and then decreasing faster. The initial increase is explained mostly by the lattice expansion, while above T_C it is necessary to consider Li and O displacements.

submitted to *Hyperfine Interactions*

Temperature Dependence and Annealing Effects on Spin Dependent Tunnel Junctions

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Abstract

The temperature and annealing effects on junctions with high (10 to 13 $M\Omega \times \mu m^2$) and low (25 to 30 $k\Omega \times \mu m^2$) resistance-area products were studied. Junction tunneling magneto-resistance (TMR) is almost unchanged and above 20% up to 200°C. A sharp and reversible TMR decrease is observed between 200 and 220ºC and is due to the exchange loss in the pinning layer. Junction TMR increases from 22 to 26% in high resistance-area product samples (resistance decreases a factor of 2), and from 22 to 37% (resistance increases 30%) in low resistance-area product samples, upon anneal up to 200-230°C. Rutherford Backscatering (RBS) analysis of the oxygen distribution in as-deposited samples indicates oxygen asymmetry in the barrier. This asymmetry and asymmetry in barrier parameters, found in as-deposited samples, disappear after anneal at 200°C. Two regimes for the TMR dependence on anneal are proposed. The first up to 200°C, where TMR increases, as barrier is homogenized and polarization near the top electrode increases. The second, above 200°C in low-resistance junctions, where TMR increase is related with barrier height increase.

submitted to *J. Appl. Phys.*

Optical and Structural Properties of Chromium Implanted Lithium Niobate - Cluster Formation and Substitutional Incorporation

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Abstract

In order to study the possibility of Cr doping of $LiNbO_3$ by ion implantation Y-cut congruent lithium niobate samples were implanted with 100 keV Cr^+ ions at room temperature to fluences of 1×10^{16} cm $^{-2}$ and 5×10^{16} cm $^{-2}$. Annealings were performed in a conventional furnace at 600, 800 and 1000ºC for 15 min to 240 min under a flowing wet oxygen atmosphere. The optical absorption of the samples was studied in the spectral range from the ultraviolet (300 nm) to the infrared (3.5 μm). Rutherford backscattering spectrometry and Particle Induced X-ray emission studies were performed in order to investigate the lattice recovery and on the incorporation of the Cr dopant. Annealings at 600ºC and 1000ºC yielded a virtually complete lattice recovery and the Cr dopant was incorporated substitutionally in the same way as in melt-doped crystals. On the other hand at 800ºC the annealing was hampered by the formation of clusters.

submitted to *Nucl. Instr. Meth. B.*

SEMICONDUCTORS

Radioactive Isotope Identifications of Au and Pt Photoluminescence Centres in Silicon

M.O. Henry(a), E. Alves(b), J. Bollmann(d), A. Burchard(e), M. Deicher(f), M. Fanciulli(g), D. Forkel-Wirth(e), M.H. Knopf(f), S. Lindner(d), G. Margerle(f), E. MCGlynn(a), K.G. McGuigan(h), J.C. Soares(c), A. Stotzler(f) and G. Weyer(g)

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(h) *Department of Physics, Royal College of Surgeons, Dublin 2, Ireland*

Abstract

Photoluminescence (PL) measurements on silicon samples implanted with radioactive Hg isotopes which decay fully or partly through the series Au/Pt/Ir are reported. Results obtained with ^{193}Au isotopes (half-life 17 h) confirm earlier work concerning the involvement of Au in the 735 meV centre first observed in Fe-diffused silicon. A PL spectrum consisting of a series of doublets originating at 780/782 meV is found to be Au-related. An important new result is that the 1066 meV trigonal centre, commonly attributed to Fe-B pairs, is found to involve Au atoms. For Pt-related ^{191}Pt isotopes (half-life 2.9 days) provide conclusive proof of PI involvement in three PL centres, centres with zero-phonon lines at 1026, 884 and 777 meV. There is not sufficient evidence from the changes observed in the spectra to make any conclusive deductions concerning the occurrence of Ir-related PL spectra in our samples.

Physica Status Solidi B - Basic Research **210** (1998) 853-858.

Ion Beam and Photoluminescence Studies of Er and O Implanted GaN

E.Alves[1], M.F. da Silva[1], J.C.Soares[2], R.Vianden[3], J.Bartels[3], A.Kozanecki[4]

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Abstract

The annealing behavior and the lattice site location of Er and Er+O (only Er was localized) implanted into GaN single crystalline epilayers were studied with the RBS/channeling and photoluminescence techniques. After implantation the results show that for doses of 6x10^{14} Er^{+}/cm^{2} the Er ions occupy substitutional sites in the GaN lattice as revealed by detailed angular scans along the <10$\bar{1}$1> and <0001> axes. An increase of the dose to 6x10^{15} Er^{+}/cm^{2} produces a continuous amorphous layer. The damage produced by the implantation starts to anneal at 600^{o}C. For the samples implanted with the lower dose the recovery is significant after 2 min annealing at 900^{o}C. During the annealing process Er remains in substitutional sites. The

presence of O seems to slightly increase the substitutional fraction after annealing at 900°C. Further the photoluminescence signal is more intense for the sample co-doped with oxygen.

Nucl. Instr. and Meth. B, in press.

Annealing Behavior and Lattice Site Location of Hf Implanted GaN

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Abstract

The defect recovery and lattice site location of Hf implanted into GaN single crystalline epilayers were studied combining RBS/Channeling and Hyperfine Interactions measurements. The RBS/Channeling measurements performed after implantation of $5\text{x}10^{14}$ Hf^+/cm^2 at 100 keV show that nearly all the implanted ions were incorporated into substitutional sites of the GaN lattice. The damage produced by the implantation recovers almost completely after one hour annealing at 900 °C and all the Hf ions then occupy substitutional sites. The hyperfine interaction measurements were performed with the radioactive $^{181}\text{Hf}/^{181}\text{Ta}$ probe, after implantation of ^{181}Hf to a fluence of 5×10^{12} Hf^+/cm^2 with 80 keV. These measurements show that the defect recovery occurs in the 600-800 °C annealing temperature range.

Mat. Sci. & Eng. B, in press.

RBS Lattice Site Location and Damage Recovery Studies in GaN

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Abstract

Erbium was implanted with 160 keV at doses between $5\text{x}10^{14}$ and $5\text{x}10^{15}$ at/cm^2 into (0001) epitaxial GaN on sapphire and annealed at various temperatures between 600 and 1000 °C. The RBS/Channeling technique was used to analyze the damage recovery during different annealing steps and to determine the lattice location of the implanted Er. For a sample implanted with $5\text{x}10^{14}$ and annealed for 30 min at 600 °C a complete overlap of the Er and Ga angular scans across the <0001> axis was observed, indicating that 100% of Er occupies substitutional sites. Measurements along the $<10\bar{1}1>$ channel show that Er is located on Ga sites. The damage recovery was slightly better for the samples co-implanted with the same dose of Oxygen in an overlapping profile (E=25 keV). However, a complete recovery of the damage caused by the implantation was not achieved. Samples implanted with higher Er and O doses ($5\text{x}10^{15}$ at/cm^2) and at the same energies as above were annealed at 600° for 30 min and at 900, 1000 °C for 120 s using a proximity cap. The higher dose caused the complete

amorphisation of the surface layer. After annealing indications of epitaxial regrowth were observed, however, the substitutional fraction remains substantially lower and the damage recovery is less complete.

MRS - Internat. J. Nitride Semicond. Res., in press.

Ion Implantation of Microcrystalline Silicon for Low Process Temperature Top Gate TFTs

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Abstract

Ion implantation of phosphorus was used to dope amorphous and microcrystalline silicon with the aim of achieving a low-temperature, self-aligned process for forming n^{+} contacts to top-gate thin-film transistors. Amorphous and microcrystalline films made with both RF glow discharge and hot-wire chemical vapor deposition were implanted. The effect of the dose, energy and implantation temperature and subsequent annealing at increasing temperatures on the dark conductivity, activation energy and photoconductivity were studied. Lowering the energy (15 keV) while increasing the dose (10^{15} cm^{-2}) and the implantation temperature (300 ^{o}C) resulted in the highest after anneal (300 ^{o}C) dark conductivity for both hot-wire (0.3 Ω^{-1} cm^{-1}) and RF (0.2 Ω^{-1} cm^{-1}) microcrystalline films.

Thin Solid Films , in press.

Optoelectronic and Structural Properties of Amorphous Silicon/carbon Alloys Deposited by Low-Power Electron-Cyclotron Resonance Plasma-Enhanced Chemical-Vapor Deposition

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Abstract

The optoelectronic and structural properties of hydrogenated amorphous silicon-carbon alloys (a-SiC:H) are studied over the entire compositional range of carbon content. The films are prepared using low-power electron-cyclotron resonance (ECR) plasma-enhanced chemical vapor deposition. The carbon content was varied by using different methane (or ethylene-) –to-silane gas phase ratios and by introducing the methane (or ethylene) either remotely into the plasma stream or directly through the ECR source, together with the excitation gas

(hydrogen). Regardless of the deposition conditions and source gases used, the optical, structural and transport properties of the a-SiC:H alloys followed simple universal dependencies related to changes in the density of states associated with their structural disorder. The deep defect density from photothermal deflection spectroscopy, the ESR spin density, the steady-state and the transient photoluminescence, the dark and photoconductivity, the temperature of the hydrogen evolution peaks and the bonding from infrared spectroscopy are correlated to the Urbach tail energy, the B factor of the Tauc plot and E_{04} (defined as the energy at which the absorption coefficient is equal to 10^4 cm^{-1}). Silicon-rich and carbon-rich regions with very different properties, corresponding approximately to carbon fractions below and above 0.5, respectively, can be distinguished. The properties of the ECR a-SiC:H alloys are compared with those of alloys deposited by RF glow discharge.

submitted to *J. Appl. Phys.*.

Incorporation of the Transition Metal Hf into GaN

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Abstract

The perturbed angular correlation (PAC) technique was applied to study the incorporation of the transition metal Hf in to GaN after implantation. To this end the PAC probe ^{181}Hf(^{181}Ta) was implanted into epitaxial Wurtzite GaN layers (1.3 µm on sapphire) with an energy of 160 keV and doses of 7×10^{12} at/cm^2. PAC spectra were recorded during an isochronal annealing programme, using RTA and furnace annealing, in the 300-1000 °C temperature range. After implantation the spectra show a damped oscillation corresponding to a quadrupole interaction frequency (QIF) of ν_Q = 340 MHz for 30% of the probe nuclei. Annealing up to 600 °C reduces the damping of this frequency without an increase of the probe atom fraction in these sites. Above 600 °C the quadrupole interaction becomes rapidly better defined until after the 900 °C RTA step more than 80% of the Hf probes experience a well defined QIF due to the incorporation of Hf on undisturbed sites of the hexagonal GaN Wurtzite lattice. An interaction frequency of ν_Q = 340 MHz is derived. RTA and furnace annealing yield similar results for annealings up to 800 ^{o}C, where the undisturbed fraction reaches about 60%. Then RTA at higher temperatures increases this fraction, while furnace annealing leads to a decrease down to 30% after annealing at 1000 ^{o}C. To our knowledge this is the first time that a transition metal probe like Hf is incorporated to such a large extent into a semiconductor lattice.

submitted to *Hyperfine Interactions*.

METALS

The Behaviour of Ion-implanted Tungsten Species During Anodic Oxidation of Aluminium

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Abstract

Amorphous anodic oxide films have been formed at high efficiency on aluminium implanted with 3.0×10^{20} W ions m^{-2} in order to study the behaviour of tungsten during film growth. The initial film is composed mainly of alumina because the outer layer of aluminium above the main implanted region of the substrate is oxidized. During this period, tungsten atoms, present in low concentrations in the aluminium, accumulate in a thin metal layer just beneath the anodic film. Subsequently, the main tungsten-implanted region is oxidized, with incorporation of tungsten and aluminium species into the anodic film at the metal-film interface in proportion to their concentrations in the metal. The incorporated tungsten species migrate outwards in the anodic film at about 0.34 times the rate of Al^{3+} ions. After oxidation of the main tungsten-containing region, more dilute regions of metal containing about 1 at% W are consumed, with oxidation of aluminium and tungsten in the presence of a highly tungsten-enriched metal layer. The enrichment is initially equivalent to 15 ± 4 at% W, assuming that the enriched layer is 2 nm thick. However, rater, as the metal-film interface reaches regions of metal containing about 0.1 at% VV, the enriched layer contains significantly more tungsten than is usual for such dilute metal regions, indicating that tungsten is transported with the metal-film interface from metal regions of higher prior tungsten concentration as the film thickens.

J. Phys. **D31** (1998) 2083-2090.

Electrical and Metallographic Characterization of Beryllium Pebbles

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Abstract

To confirm the conservative assumptions about the electrical resistivity of the beryllium pebble bed in the course of the electromagnetic analyses for the Reference ITER Breeding Blanket, this property has been actually measured. In case of a single size 2 mm pebble bed, the resistivity of the bed decreases drastically to about 10^{-4} Ωm by applying an external pressure. After this first drop, the resistivity shows an almost linear decrease with the applied pressure. The same trend appears for a single size 0.1-0.2 mm pebble bed, but the resistivity values are about one order of magnitude higher than in the case of 2 mm pebbles. At room temperature, the lowest resistivity values were found for the case of a binary pebble bed. Results from the pebbles metallographic structure analyses by means of optical microscopy as well as BeO measurements using the Rutherford backscattering technique are also presented.

Proceedings of 20th Symposium on Fusion Technology, **Vol. 2** (1998) pp.1365.

Modification and Characterisation of Al Surfaces Implanted with Cr Ions

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Abstract

In this work metastable Al-Cr surface alloys were produced by high fluence Cr implantations into polycrystalline aluminium, under different conditions of temperature and fluence.

Clear evidence for the direct formation of $Al_{86}Cr_{14}$ and $Al_{13}Cr_2$ intermetallic phases by ion implantation is reported, as well as investigations on the influence of implantation temperature, fluence and annealing treatments on the formation and evolution of Al-Cr intermetallic compounds. It was also found that the $Al_{86}Cr_{14}$ phase forms first and is transformed into $Al_{13}Cr_2$ by a proper thermal annealing.

Proceedings of *OECD/NEA Workshop on Ion and Slow Positron Beam Utilization,* Costa da Caparica, 15-18 Sept. 1998.

Optimisation of the High Temperature Oxidation Behaviour of Conventional Stainless Steels by Surface Applied Rare Earth Elements

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Abstract

In this work we have performed an extensive survey of modification procedures of the stainless steel surface in order to enhance the resistance to high temperature oxidation of commercial AISI-304 grade stainless steels. Both lanthanum and cerium modifiers have been used in an attempt to increase the in service temperature of this stainless steel family. Four different modification procedures were utilised: spray of dilute nitrate solutions, pyrolisis of aerosol nitrate solutions, ion exchange of cerium cations over wet oxidised stainless steel surfaces and ion implantation. The effect of the modification procedure on the steel surface prior to the oxidation treatment is carefully studied. Whatever the modification procedure the in service temperature under synthetic air was increased up to 1173 K. However, the results obtained indicated that lanthanum modification is much more effective than cerium one. For the same operating conditions the weight gain and hence the scale thickness is about twice thicker for cerium than for lanthanum. The formation of a lanthanum perovskite in the case of lanthanum modified surfaces has been assessed by XRD, SEM-EDX and TEM. In the case of cerium the most stable oxidation state of this element prevents the formation of the perovskite phase, CeO, particles are detected by XRD. As both lanthanum and cerium enhances the oxidation resistance at high temperatures it has to be claimed more than one mechanism for the so-called reactive element effect. The oxide scale structure points to an effect of lanthanum consisting in the selective blocking of the outwards diffusion of cations and to slowing diffusion rates for the inwards diffusion of anions. This was also suggested from molecular dynamics simulations of model grain boundaries containing lanthanum.

Proceedings of the *European Conference "Information day on Stainless Steel"*, ECSC, D. Naylor ed., Sevilla 6th October 1998.

High Temperature Oxidation of AISI-304 Stainless Steel after Cerium Ion Implantation

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Abstract

100 KeV Ce^+ ion implantation of AISI-304 type stainless steels have been carried out with a nominal dose of 0.85x 10^{16} at.cm^{-2}. Implantation was performed on two sets of samples: 2B finished AISI-304 stainless steel and synthetic air oxidised samples (2 minutes at 1173 K). In order to test possible damages induced by the implantation, one set of samples was annealed at 1325 K after Ce^+ implantation.

Samples were isothermally oxidised at 1173 K for 22 hours in synthetic air in a thermogravimetric device. Mass gain was continuously monitored as a function of time. Results were compared with a reference non-implanted AISI-304 stainless steel. Oxide scales were characterised by SEM-EDX and XRD.

TG data show that implanted specimens have an enhanced oxidation resistance at 1173 K. Total mass gains after 22 hours oxidation were of 1 mg cm^{-2} for as-implanted samples (6.4 µm scale thickness), 0.5 mg cm^{-2} for implanted and annealed samples (3.2 µm scale thickness) and 4 mg cm^{-2} (25 µm scale thickness) for untreated sample. A three-term model including the effects of parabolic, linear and quadratic rate laws can fit the kinetic curve for implanted samples:

$$Dw = k_l t + k_q t^2 + k_p t^{1/2}$$

Results show that the parabolic rate constant is similar for implanted and implanted/annealed samples while for the latter the linear and quadratic rate constants are smaller, indicating a better protective behaviour of the samples after the annealing treatment. Possibly by reducing surface defects induced by implantation.

SEM-EDX analysis indicate that the scale formed over the implanted steels is composed mainly of Cr_2O_3 and Cr-Mn spinels while in the untreated sample the oxide layer is formed by Fe_20_3 and Fe-Cr oxides. Implanted/annealed samples present the same composition of the oxide scale than as-implanted samples but the morphology of the scale is slightly different: in as-implanted samples numerous oxide nodules form on the surface that do not appear on annealed samples.

XRD experiments confirm SEM-EDX results. They show the formation of a scale rich in Cr-Mn spinels and Cr_2O_3. Also a martensite layer formed in the alloy/scale interface is detected.

Communication (accepted) to: *International Conference on Stainless Steel '99 — Science & Market*. Assoziazione Italiana de Matallurgia. Sardinia, 7-9 June 1999.

THIN FILMS

Physical, Structural and Mechanical Characterization of $Ti_{1-x}Si_xN_y$ Films

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Abstract

Within the frame of this work, $Ti_{1-x}Si_xN_y$ hard coatings with $0 \leq x \leq 0.37$ and thicknesses ranging from 1.2 to 3.5 μm, were prepared by r.f. reactive magnetron sputtering in an Ar/N_2 gas mixture. X-ray diffraction and Fourier analysis of X-ray profiles were used to investigate the structure and grain size, and its correlation with hardness behaviour, as a function of the Si content, bias voltage and working gas (argon) flow rate. In this respect, the results show that a double cubic phase of NaCl type was developed with lattice parameters of 4.18 and 4.30 Å, revealing the (111) orientation for low Si content ($x = 0.05$), (220) for intermediate Si contents ($0.13 \leq x \leq 0.22$) and (200) for the highest Si contents ($0.30 \leq x \leq 0.37$). Regarding the results of ultramicrohardness tests, and although all samples with $0.05 \leq x \leq 0.30$ present a hardness value higher than 30 GPa, the $Ti_{.85}Si_{.15}N_{1.03}$ revealed the highest hardness value, around 47 GPa, which is more than twice as high as that of common TiN. Furthermore, the study of hardness as a function of the applied bias voltage revealed that best results are achieved between –50 V and 0 V. The variation in hardness as a function of the argon flow showed that best results in hardness are obtained when working with flow rates around 110 cm^3/min.

Surface and Coatings Technology **108-109** (1998) 236-240.

(p,p) non-Rutherford Backscattering Analysis of Silicon Carbide

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Abstract

In this paper, (p,p) non-Rutherford elastic backscattering (NBS) measurements with 1.6 MeV proton beams have been used to determine the areal density and C/Si stoichiometric ratio in 300-1000 nm $SiC_x(H_y)$ films deposited on silicon substrates by Chemical Vapour Deposition (CVD) using two different gas sources, methane (CH_4) and ethylene (C_2H_4). At the same time, oxygen is also found in some of these samples. The results show that (p,p) NBS can be used to determine the areal density and C/Si ratio with a reasonable accuracy, especially when samples are thick or the terminal energy of the accelerator is low for analysis using a helium beam.

Ncl. Instr. and Meth. **B142** (1998) 571-577.

Study by Rutherford Backscattering Spectroscopy of the Heterostructure of Lead Titanate Thin Films

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Abstract

A depth profile analysis of modified lead titanate thin films was performed by means of Rutherford Backscattering Spectroscopy (RBS). These films were deposited from sol-gel synthesized solutions onto platinized silicon substrates and crystallized by thermal treatments at temperatures of about 650 ºC. The chemistry of the solution and the thermal treatment for crystallization affect the heterostructure of the resulting films. Losses of lead and formation of substrate-film interfaces are produced during the crystallization of the films. These film characteristics determine their ferroelectric response.

Journal of Sol-Gel Science and Technology **13** (1998) 843-847.

Solid-phase Crystallization of Amorphous SiGe Films Deposited by LPCVD on SiO_2 and Glass

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Abstract

The crystallization kinetics and film microstructure of poly-SiGe layers obtained by solid-phase crystallization of amorphous SiGe with Ge fractions appropriate for the fabrication of thin film transistors have been studied in detail. Amorphous SiGe layers were deposited by LPCVD at 450ºC on thermally oxidized Si wafers and 7059 Corning glasses, using Si_2H_6 and GeH_4 as gas sources. The films were 110 nm thick and the Ge fraction (x) was in the 0–0.42 range. The samples were annealed at 550ºC for times up to 400 h at low pressure (below 9 Pa) to crystallize the amorphous layers. The evolution of the crystallization and the resulting film microstructure were characterized by X-ray diffractometry and transmission electron microscopy. The experimental results on growth kinetics fit the Avrami's model. The characteristic crystallization time decreases slowly for $x<0.3$ and more abruptly for higher values of x. The transient time depends exponentially on x in all the interval. The recrystallized films have a (111)-preferred orientation for low values of x and evolve to a more randomly oriented polycrystal as x increases. The grain size in the fully crystallized layers is lognormally distributed and decreases with increasing x. The results are similar for the films deposited on silicon dioxide and glass.

Thin Solid Films, in press.

Strain Relaxation Mechanisms in $Si_{1-x}Ge_x$ Layers Grown by Solid-Phase Epitaxy: Influence of the Growth Temperature

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Abstract

The influence of the composition and growth temperature on the strain and defect structure of $Si_{1x}Ge_x$ layers of $0.21 \leq x \leq 0.34$ grown on (001) Si wafers by solid phase epitaxy is presented. The strain in the layers was measured by Raman spectroscopy and RBS/channeling angular scans. The defects were analyzed using high resolution electron microscopy. Three different relaxation mechanisms have been identified and characterized. The first mechanism occurs at the layer-substrate interface of the samples by the introduction of isolated defects. It is found to be thermally activated with an activation energy of E_a = 0.16 eV and a prefactor that depends on the Ge content of the layer. This mechanism produces partial relaxation of the layers and hinders the growth of fully strained layers. The second relaxation mechanism emerges at a distance from the interface which depends on the stress in the crystallized portion of the layer. In this case the strain relaxation is caused by stacking faults that nucleate when they are energetically feasible and propagate towards the surface of the sample during growth. At low growth temperatures the defects are confined to the upper part of the epitaxial layers at a distance from the interface that agrees with the theoretical predictions based on the equilibrium critical layer thickness. The third relaxation mechanism is introduced at high growth temperature and is based on the gliding of the stacking faults towards the layer-substrate interface. As a result of this mechanism the stress in the layers is reduced compared to the stress in the layers grown at lower temperatures and approaches the equilibrium value corresponding to the whole layer thickness. This behaviour indicates that the layers grown at low temperature, where the stacking faults are confined to the upper part, are to some extent metastable.

J. Electronic Mater., in press.

GMR in High Fluence Ion Implanted Granular Thin Films

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Abstract

We report on the magnetic and magnetoresistive (MR) properties of granular materials obtained by implantation of high doses (from 10^{16} to 1.8×10^{17} ions/cm^2) of Fe and Co ions into Cu and Ag thin films (about 2000 Å). The local microstructure vs implantation dosis and posterior thermal treatment is discussed, indicating optimum conditions to reach MR values of practical relevance. MR measurements up to very high fields (32 T) are presented, supporting

recent theoretical work on the role of short-range magnetic correlations in MR for granular materials.

J. Mag. and Mag. Mat. (in press).

High-Tc Superconductors Studies with Radioactive Ion Beams at ISOLDE

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Abstract

New applications of radio isotopes for research in solid state physics are steadily growing, due to the big variety of intense radioactive ion beams which are provided by the on-line coupling of high resolution isotope separators to particle accelerators, such as the ISOLDE facility at CERN. There new experiments are performed by an increasing number of solid state researchers which combine nuclear spectroscopic techniques such as Mössbauer, Perturbed Angular Correlations and Emission Channeling with the traditional non-radioactive techniques like Deep Level Transient Spectroscopy and Hall Effect measurements. Recently isotopes of elements not available before were successfully used in new PAC experiments, and the first Photoluminescence (PL) measurements, where the element transmutation plays the essential role on the PL peak identification, have been performed.

In this paper we shortly review the ISOLDE facility, and present a new approach to investigate the oxygen doping and structural point defects in High-Tc Superconductor materials (HTcS). For this purpose radioactive ion beams are used to dope HTcS thin films and pellets, which are then studied by combining complementary techniques such as Perturbed Angular Correlation, Emission Channeling, and Electrical/Magnetic Measurements. As examples we will discuss the most recent studies of Hg doping high quality $YBa_2Cu_3O_{6+x}$ thin films, and first results obtained in the characterization of the order/disorder of Hg in the Hg-planes of the HTcS family $Hg_1Ba_2R_{(n-1)}Cu_nO_{(2n+2+\delta)}$ (T_C >130 K).

Proceedings of *OECD/NEA Workshop on Ion and Slow Positron Beam Utilization*, Costa da Caparica, 15-18 Sept. 1998.

On-line Perturbed Angular Correlation Studies with the Short Lived ^{127}Cs Probe

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Abstract

On-line Perturbed Angular Correlation (PAC) experiments were performed on the 66.0 keV excited state of ^{127}Cs, using the γ (114.7 keV) – e^-_L (66.0 keV) cascade from the decay of the short-lived ^{127}Ba ($T_{1/2}$=13 min) isotope produced at the ISOLDE facility at CERN. The PAC experiments were performed with an optimized γ-e^- spectrometer coupled to the ISOLDE beam line, which allowed simultaneous implantation and measurement. The optimization of the experiment is described and the first results on metallic foils and single crystals of Al, Be, Ga, Zn, and Ni are presented and discussed. The derived nuclear moments of the 66.0 keV excited state of ^{127}Cs are $|\mu| = 2.9(2)\ \mu_N$ and $|Q| = 0.58(12)$ b. Applications of this new PAC isotope are outlined.

Nucl. Instr. and Meth. B, in press.

The Influence of O on the Lattice Sites of Rare Earths in Si

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Abstract

We have used conversion electron emission channeling to investigate the lattice sites of ^{167m}Er following implantation of the radioactive isotope ^{167}Tm into CZ Si and FZ Si at varying doses (6×10^{12} - 5×10^{13} cm^{-2}). For annealing temperatures up to 600°C no influence of O on the Er lattice sites could be detected, but isothermal annealing at 900°C showed up characteristic differences between FZ and CZ Si. While in both cases prolonged annealing at 900°C caused Er to leave its preferred near-tetrahedral sites in favour of random lattice sites, this process occurred by orders of magnitude faster in CZ Si. Besides, in FZ Si the incorporation of Er on random lattice sites was fastest in samples implanted with high doses of Tm/Er, in CZ Si it was fastest for low dose implantations. Using a numerical model for O and Tm/Er diffusion and precipitation, we are able to consistently explain this behaviour by the preferential formation of rare earth silicide precipitates in FZ and rare earth oxide precipitates in CZ Si. Our results therefore support the interpretation that small Er oxide precipitates might be involved in the enhancement of Er luminescence which is usually observed in O co-implanted samples annealed at 900°C.

Nucl. Instr. and Meth. B, in press.

Stability and Diffusion Studies of Hg Implanted $YBa_2Cu_3O_{6+x}$

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Abstract

The ^{197m}Hg radioactive isotope was implanted with low fluences of 10^{13} at/cm^2 and 60 keV energy into $YBa_2Cu_3O_{6+x}$ (YBCO) superconducting thin films at ISOLDE/CERN. We report on the Hg dynamics and stability inside the YBCO lattice as a function of annealing temperature up to 890 K under vacuum and O_2 atmospheres. The perturbed angular correlation (PAC) technique was used for probing at an atomic scale the Hg behavior, while by monitoring in-situ the sample's activity the Hg outdiffusion was studied.

Nucl. Instr. and Meth. B, in press.

Lattice Site and Stability of Hg Implanted $YBa_2Cu_3O_{6+x}$ Thin Films

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Abstract

High quality $YBa_2Cu_3O_{6+x}$ (YBCO) superconducting thin films were implanted with the radioactive ^{197m}Hg ($T_{1/2}$=24 h) isotope to low fluences of 10^{13} at/cm^2 and 60 keV energy. The lattice location and stability of the implanted Hg were studied combining the Perturbed Angular Correlation and Emission Channeling techniques. We show that Hg can be introduced into the YBCO lattice by ion implantation into a unique regular site. The EC data show that Hg is located on a highly symmetric site on the YBCO lattice, while the PAC data suggests that Hg occupies the Cu(1) site. Annealing studies were performed under vacuum and O_2 atmosphere and show that Hg starts to diffuse only above 653 K.

Nucl. Instr. and Meth. B, in press.

Characterisation of Titanium Silicon Nitride Films Deposited by PVD

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Abstract

In recent years nitride coatings have found widespread applications for tool and other hard surfaces. In this work (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 was measured by Rutherford backscattering spectrometry (RBS). $Ti_{1-x}Si_xN$ samples with $0 \le x \le 0.37$ 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 $Ti_{0.70}Si_{0.30}N$ and $Ti_{0.87}Si_{0.13}N$ samples presented the best results, with a critical load for total failure around 115 N and 105 N respectively.

Vacuum, in press.

OTHERS

Multilayer Surface Coating of Glass with Metal Oxides: Characterisation by Rutherford Backscattering, X-Ray Fluorescence and FTICR Mass Spectrometry

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(see Inorganic and Organometallic Chemistry)

Proceedings of XVIII International Congress on Glass, San Francisco, California, USA, July 5-10, 1998. [Westerville, Ohio, USA]: The American Ceramic Society, principal editors M.K.Choudhary, N.T. Huff and C.H. Drummond III, 1998. "C4:pp.13-18, Coatings, Surfaces and Surface Analysis". [cd-rom]

Nuclear Microbeam Study of Advanced Materials for Fusion Reactor Technology

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Abstract

The Oxford Scanning Proton Microprobe was used to study SiC fibres, SiC/SiC ceramic composites and Be pebbles, which are some of the most important materials for fusion technology. For the SiC materials, although the results reveal a high degree of homogeneity and purity in the composition of the fibres, some grains containing heavy metals were detected in the composites. Rutherford backscattering analysis further allowed establishing that at least some of these grains are not on the surface of the material but rather distributed throughout the bulk of the SiC composites. The two different types of Be pebbles analysed also showed very different levels of contaminants. The information obtained with the microbeam analysis is confronted with the one resulting from the broad beam PIXE and RBS analysis.

Submitted to *Nucl. Instr. and Meth. B.*

Current Work

Nuclear Microprobe at ITN

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The upgrading of the ion beam laboratory has been accomplished with the installation of a nuclear microprobe in the (p,γ) beam line of the 2.5 MeV Van de Graaff accelerator. Conditions for optimum performance of the microprobe have been established. This has involved the construction of a small room with air conditioning to maintain a stable temperature. As already mentioned in the 1997 annual report, some of the components were bought direct from a company in Oxford. Many others have been designed and manufactured at ITN. The tests of microprobe started at the end of 1998 and will continue through 1999 in order to fully optimised its performance. The spatial resolution attainable to date has been measured by recording the focused beam energy loss as it scans over a 2000 mesh copper grid (period of 12.7 μm). A resolution of <1 μm has been measured using a very low beam current of less than 1 pA of 2 MeV protons. At a beam current of 1-10 pA, the minimum required for PIXE and backscattering analysis, the measured spatial resolution so far is ≈1.5 μm. This is extremely good considering that the system has only been commissioned in the last week.

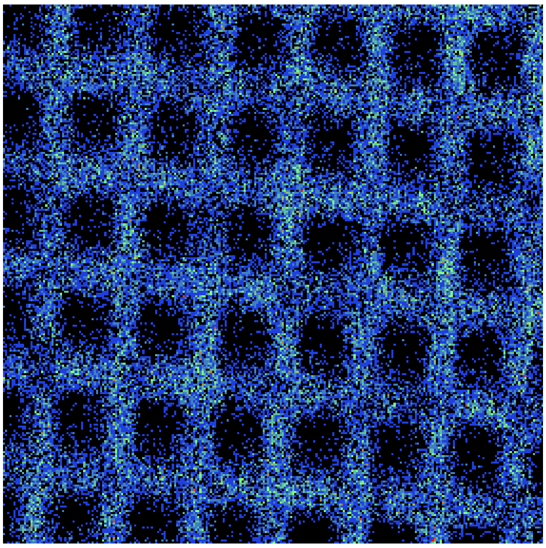

Fig. 1 – Microprobe scan image (80*80 μm) of a copper grid (period of 12.7 μm).

Incorporation of Iodine into Gallium Arsenide after Implantation

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Single crystalline samples of (100)-cut GaAs were implanted with 160 keV I^+ ions to a fluence of 3×10^{15} cm^{-2} at room temperature. The projected range is 48 nm and the straggling 19 nm according to TRIM calculations. In order to avoid outdiffusion of arsenic during the annealing process the samples were capped with a 40 nm thick silicon nitride layer. Annealings were performed at 700ºC (sample R7) and 800ºC (sample R8) for 30 s using Rapid Thermal Annealing.

For the analysis by Rutherford backscattering spectrometry the capping had to be removed before starting the measurements using fluoric acids. Fig. 1 shows RBS spectra for samples annealed at 700 and 800ºC recorded using a 1.6 MeV He^+ ion beam. The I-RBS yield in the aligned direction is reduced pointing out that a large fraction of the iodine has been incorporated into the host lattice.

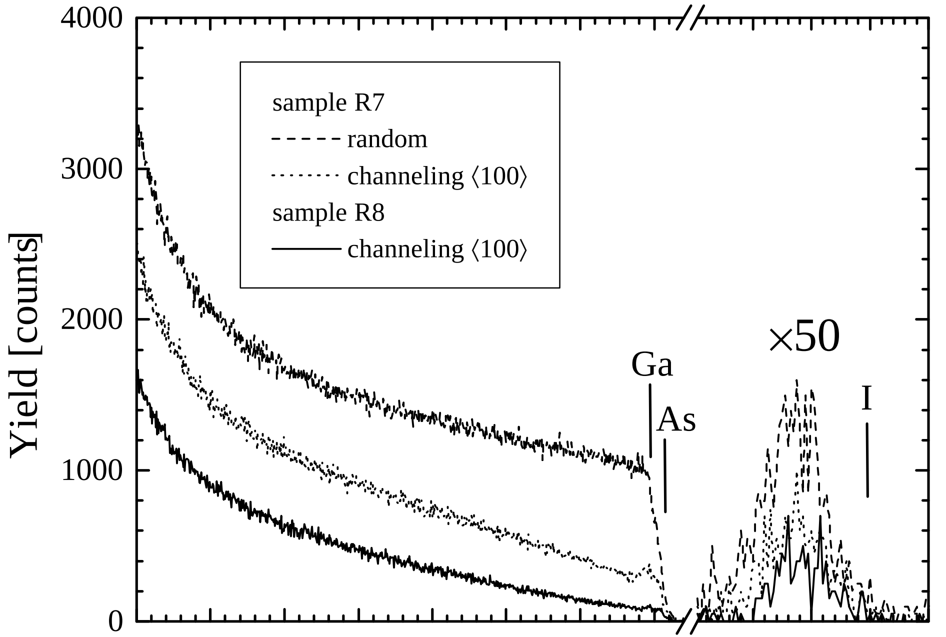
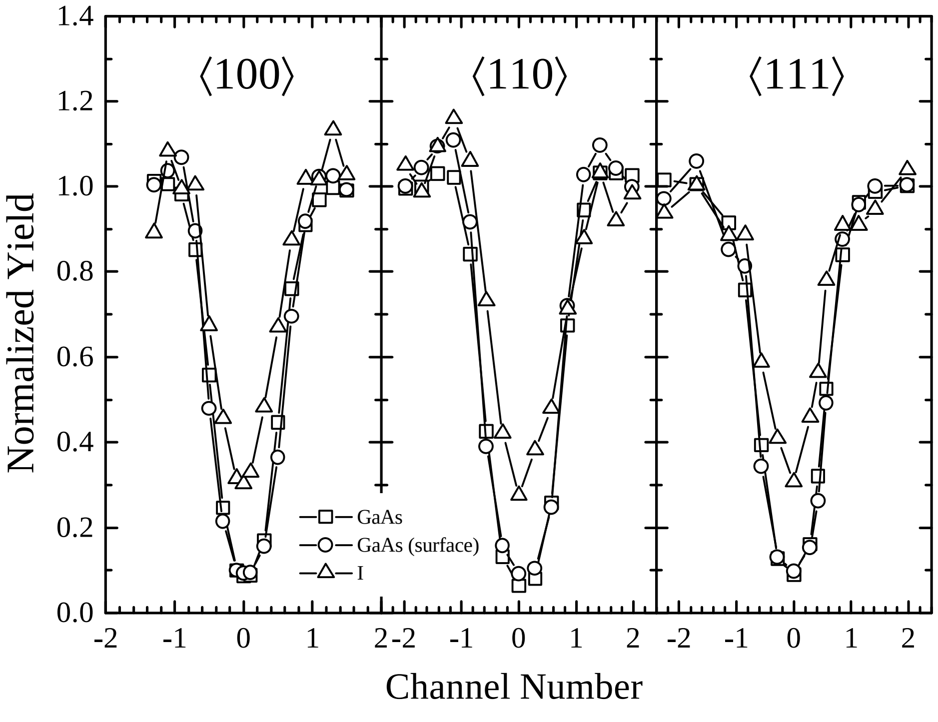


Fig. 1: RBS spectra (random and aligned) of GaAs samples implanted with 160 keV I to a fluence of 3×10^{15} cm^{-2}

In order to obtain more detailed information on the incorporation mechanism of iodine into GaAs angular scans were performed for the three major axial direction of the zinc blende crystal structure of GaAs. The results for the sample R8 are depicted in Figs. 2. Since Ga and As cannot be separated well in the RBS spectra a common yield for both elements was determined. Two regions were selected: one near the surface (0-70 nm) corresponding to the layer in which the iodine is found and a second one between 70 and 140 nm where the host lattice is virtually undisturbed by implantation damage. For the I-RBS only one region (0-70 nm) was selected. The clear dips observed for the I-RBS signals and their similar width compared to the GaAs-RBS confirm that there is a significant substitutional fraction of the dopant. In order to check if there is a fraction of iodine dopants located on a defined interstitial lattice site (e.g. tetrahedral site) the minimum yields χ of GaAs and iodine were compared using the following formula

$$f = \frac{1 - c_{I}}{1 - c_{GaAs}}$$

It was found that for all axes in both samples the values for f are lying close to 0.8. Therefore it can be excluded that a measurable fraction of iodine atoms is located on a defined interstitial site and the non-substitutional fraction is distributed randomly in the lattice. By inserting the minimum yields for a perfect host lattice (between 2 and 3% depending on the axis) into the previous formula one obtains a good estimate for the substitional dopant fraction with values of 70(±5)% for sample R8.



Fig, 2: Angular scans for GaAs implanted with I and annealed at 800ºC for 30 s.

Silicide Formation by Ion Implantation: Characterisation by RBS/Channeling and Resistivity Measurements

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We have been developing Ion Beam Synthesis (IBS) as a novell technique for silicide formation, namely $IrSi_3$ and $HfSi_2$. The first is one of the few trisilicides known and the one with the highest Schottky barrier to silicon. The second is a refractive silicide with an orthorhombic structure, which would, in principle, allow it to grow epitaxially on silicon.

Our results show that it is possible to form the hafnium disilicide immediately after implantation. Upon adequate annealing, we are able to form a low resistivity buried layer with some epitaxial quality (~76%). We also demonstrate the crucial role played by the range of the implanted profile. Only the samples implanted in a channeling direction, that is, samples with higher Hf concentrations at greater depths and less implantation damage, result in a channeled profile after annealing.

In the case of the iridium trisilicide, we have been successful in forming surface continuous polycrystalline silicide layers, with good electrical characteristics. Our results show that only after annealing is the silicide phase formed, which contradicts earlier results by other authors. For these, however, the formation of the silicide phase was mostly assumed, and no XRD analyses were made. Temperature control in these early experiments might also have been deficient.

We are now interested in using other analytical techniques such as Perturbed Angular Correlations (Hf silicides) and in implanting new samples with higher energies and in channeling directions.

Chromium Silicide Formation on Porous Silicon

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Porous silicon is an interesting material because of its promising technological applications. It is hoped that one can take advantage of it when fabricating nanostructures and that electroluminescence in porous silicon might lead to light–emitting elements integrated into Si based microcircuits.

Our preliminary results show that it is possible to form chromium disilicide immediately after high temperature implantation in both columnar and spongy porous samples. The pre-oxidised porous samples have lower resistivity, similar to the normal Si ones. This particular treatment, performed before implantation, is of great importance, as the sputter coefficient and the process of chromium diffusion seem to be different in samples subjected to it.

The first annealing treatments show that the stability of the chromium profiles varies between porous and normal silicon samples. Different heat treatments have, therefore, to be studied and performed.

This year we intend to determine the correct process window for chromium silicide formation in different kinds of porous silicon.

Modification and Characterisation of Alumino-silicate Crystals by Nuclear Physics Techniques: Production and Characterisation of Colour Centres in Topaz

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Pure topaz is an alumino-silicate that is transparent throughout the visible region of the spectrum. It is well known that most topaz samples become coloured when bombarded with high-energy electrons, gamma-rays or neutron beams. It is also known that a controlled annealing of the defect structures produced upon irradiation can develop a stable blue colour in the samples. This process has been used to enhance the commercial value of the mineral.

An annealing protocol to relief tension and improve crystalline quality has been established. After this conditioning annealing step samples have been irradiated with ^{60}Co photon beam (to several kGy) or with thermal neutrons, or, as recently, ion implanted with different doses of 140 keV Cr^+ or W^+ ions.

Prior to and after irradiation the optical transmission is evaluated by means of a UV-vis.-IR spectrophotometer, and the crystalline quality checked by RBS-C.

Subsequent annealing of the irradiated samples has proved successful in producing stable blue colours.

These samples have been studied by RBS, RBS-C, X-ray diffraction, SEM and TEM, EPR and NMR as well as micro-Raman spectroscopy.

The recourse to these latter techniques fits within steps taken to establish the framework of collaboration with the Physics Department of Universidade de Aveiro.