Nuclear Solid State Physics Using Ion Beams

The research activity of the Nuclear Solid State Physics Group is focused mainly in the processing and characterisation of advanced materials using ion beam based techniques.

The main output of this activity are the publications in international journals and the training of young researchers and students.

The group is responsible for the operation of a large infrastructure called the **Ion Beam Laboratory (IBL)** in Sacavém, with the 3.1 MeV van de Graaff accelerator and the 210 KV High fluence Ion Implantor and **the two hyperfine interactions laboratories** located in Lisbon at the Nuclear Research Centre and in Geneva (ISOLDE/CERN), respectively.

The IBL is unique in Portugal and until last year was unique in the Iberian Peninsula.

There is collaboration with other researchers located mainly at the universities (UL, UNL, UC, UP, UA, UM) and it is expected and desirable to increase this collaboration through joint research groups.

Also, considerable collaboration exists with foreign research teams, namely from the universities of Seville, Madrid, Surrey, Bonn, Knoxville (USA), Budapest amongst others. Most of these collaborations started through bilateral contracts and the participation in international projects.

The strategy has been to place the IBL as a **national facility** opened to the universities and other research institutions. The group has critical size to explore and keep the facility running and available for training and advanced training in Nuclear Physics and its applications. Concerning the teaching role this facility is currently used for tutorials of the Nuclear Techniques course of the 4th year of the Physics and Engineering licenciature of the Faculty of Sciences of the University of Lisbon. Also, the group collaborates in the Master degree course of Applied Nuclear Physics of that Faculty of Sciences. A few students are doing their last year theses, MSc or PhD using this infrastructure.

The on-going activity is shortly described in the following pages. The research work covers a wide range of materials being studied using the ion beam based techniques. In 1999 about 32 papers were published in International Journals and 24 were accepted for publication. These include on-going studies on insulators such as $LiNbO_3$ and Al_2O_3, continuing research started a few years ago and on which new interesting results were obtained.

It is worth mentioning the potential of ion beam techniques for studies of thin films and multilayers. Relevant work is being carried out in the characterisation of magnetic thin films for spin valves.

The techniques installed in the IBL together with other techniques available at ITN, like neutron activation analysis and X-ray fluorescence, are also a powerful tool for research in the field of Environment, Biology, Biomedicine, Eco-Toxicology, Geology, and so on. Extensive work has been already developed in some of these areas as presented elsewhere in this annual report. It is urgent to attract more researchers and graduate students to develop more work in those areas.

The natural development of the Ion Beam Laboratory will be the installation of a higher energy accelerator, of 3 or 5 MV at the terminal. The possibility of producing multiple charged heavy ions would be a major expansion of our capabilities. Light elements can be fully separated with heavy ion Elastic Recoil Detection Analysis, leading to detection limits in heavier substrates around 0.1 at.%. Also, the composition of systems in which we have a long-standing interest, such as GaN, can be determined with much higher precision than with conventional RBS. Furthermore, heavy ion RBS leads to an enhanced mass resolution, enabling close-by elements such as Fe and Co to be separated, which is important in many systems we study, such as magnetic heterostructures.

Relevant work on Archaeology could also be carried out with the installation of an experimental beam line dedicated to the Accelerator Mass Spectrometry (AMS) technique.

Formation of metallic nanocrystals in Sapphire and rutile by ion implantation*

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Objectives

The formation of metallic nanocrystals in α-Al_2O_3 and TiO_2 using the ion implantation technique and its influence in their optical and electrical properties will be studied. The metallic ions Fe, Pt, Au and Er will be implanted in these oxides to concentrations well above its solid solubility. The non-equilibrium state produced in the implanted region will return to a thermodynamically equilibrium state by the precipitation of the implanted ions or through the formation of new compounds. The structural changes in the implanted layer and its relationship with the optical and electrical properties will be studied with the Rutherford backscattering, Channeling and Glancing X-ray techniques, optical absorption spectroscopy as well as resistivity measurements. The results will allow us to understand the physical processes involved in the formation of the nanocrystals and its influence in the optical and electrical properties of sapphire and rutile.

Results

Single crystalline α-Al_2O_3 was implanted at room temperature with Fe, W, Pt and Er. For the heavier elements, fluences of the order of $1\times10^{16}cm^{-2}$, create a continuous amorphous layer through the implanted region. When the implanted doses are below the amorphization threshold the Fe and Pt ions are incorporated into Al substitutional sites, while the W ions occupy displaced Al sites [1,2]. The Er goes to free octahedral sites. The annealing behaviour below the amorphization threshold is similar for all the implanted species. Above this value the recrystallization of the samples is influenced by the chemical nature of the implanted ions and annealing atmosphere. These results clearly confirm the trend that the recrystallization processes in sapphire are influenced both by the implanted atomic element and the crystallographic direction [3,4].

A large increase in the PL intensity of the samples implanted with Er was found after annealing at 825 ^oC. An increase of a factor of 4 in the intensity was found for the 1.54μm emission on the c- crystals implanted with 6×10^{14} Er^+/cm^2 [5]. Implantation of high fluences of iron in sapphire leads to the formation of metallic precipitates during the implantation.

Above 2×10^{17} Fe^+/cm^2 a layer with metallic characteristics develops at the surface, leading to a strong ferromagnetic behavior. The microstructures developed during annealing are strongly dependent on the substrate orientation and annealing atmosphere [6].

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Further work

The research on the precipitation kinetics studies of Co and Pt implanted sapphire is currently being done. The role of the annealing environment and implanted dose on the formation of the nanoprecipitates are under study. Simultaneously we are studying the influence of the new structures on optical and magnetic properties of samples implanted with Fe and Co. The research on this field will continue with the aim to development new properties with technological interest.

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Redistribution and incorporation of metal ions implanted into $LiNbO_3$

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Objectives

Ion implantation is a useful method for the doping of optical materials. Compared to the doping in melt and by indiffusion the impurity can be introduced in a very controlled way. Also implantation enables the doping with elements that are insoluble under normal conditions. However, processes to anneal the damage caused by the implantation process have to be developed and their impact on the redistribution and incorporation into the material has to be studied.

Results

In order to study the feasibility of the formation of thin Cr-doped layer in lithium niobate for the formation of waveguide laser $LiNbO_3$ single crystals were implanted with x keV Cr^+ ions to doses ranging from 5×10^{15} cm^{-2} to 5×10^{16} cm^{-2}. Annealings were performed at 600ºC, 800ºC, 1000ºC in a conventional furnace with flowing wet oxygen atmosphere. Samples annealed at 600ºC and 1000ºC showed after 15 min a strong Cr diffusion and a partly or complete recrystallization, respectively. In contrast, in the sample annealed at 800ºC the damage is not removed even after annealing times up to several hours [1]. PIXE/channeling investigations [2] of the sample annealed at 1000ºC showed that the lattice sites of the implanted Cr coincides fully with that observed for melt-doped material . The assignments for the lattice sites were performed using the CASSIS program (Kling, A*., Nucl. Instr. Meth.* ***B*** **102** (1995) 141-144) in its latest version [3].

The incorporation of Ir and Pt - impurities that can be incorporated during the growth process from the crucible material - was also investigated by implanting these noble metals into $LiNbO_3$ at doses up to 5×10^{16} cm^{-2}. Due to the large ionic radius of these two noble metals it was expected that no substitutional incorporation should take place. While Pt showed – as expected - only a moderate diffusion at high temperatures (1000ºC) and a tendency to form oriented clusters (Kling, A., Soares, J.C., da Silva, M.F., *Nucl. Instr. Meth*. ***B*** **141** (1998) 436-440).Ir rather readily diffuses into the bulk forming a homogenous distribution with a low concentration and exhibits a partial incorporation into the host lattice [4]. The different behaviour of the two noble metals is rather surprising since their size and chemical properties are quite similar.

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Further work

Optical waveguides are planned to be formed by He^+ implantation in the Cr-implanted samples so that the optical properties of the implantation-doped layer can be investigated with regard to luminescence and absorption.

Combined Ion Beam and Hyperfine Interaction Studies of $LiNbO_3$ and $LiTaO_3$

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Objectives

Lithium niobate ($LiNbO_3$) and lithium tantalate ($LiTaO_3$) are widely used in optoelectronics and integrated devices. However, some basic questions remain open, namely the fine details of their defect structure and the interplay of the factors that determine the lattice site of dopants. Ion beam and hyperfine interaction methods are powerful tools in these fields. A review of the work performed recently by our group can be found in ref. [1].

Results

$LiNbO_3$ and $LiTaO_3$ are normally Li-deficient and this deficiency must be compensated by a deviation from the ideal structure to maintain charge neutrality. Stoichiometric $LiNbO_3$ crystals grown in Budapest and Madrid were compared with congruent crystals. Our data show that the structure of congruent $LiNbO_3$ cannot be completely described by the models that assume only cation vacancies and Nb antisites. The additional defects are cation stacking inversions, in which the sequence coincides locally with the one of ilmenite-like $LiNbO_3$. We have proposed that these inversions are associated with the Nb antisites necessary for charge compensation [2]. Studies of the lattice site of hexavalent impurities showed that they are exclusively located on Nb sites indicating that only Li vacancies can exist in $LiNbO_3$ [3].

Another interesting aspect of these oxides is their transition to the paraelectric phase. The phase transition occurs at a significantly lower temperature for $LiTaO_3$ (~900 K) making more accessible a study in this oxide. The neutron scattering data of Abrahams (S.C. Abrahams et al., J. Phys. Chem. Sol. 34 (1973) 521) are the bases of all theories modeling the transition as an order-disorder mechanism with the Li ions hopping among two sites. Hyperfine Interaction measurements with the ^{111}In probe (which replaces Li) were made up to 1040 K. The observed temperature dependence of the electric field gradient (efg) is unusual, first increasing up to T_C and then decreasing. Calculations of efg using a point charge model show that the thermal expansion of the lattice explains the temperature dependence up to T_C. In contrast, the data above T_C cannot by explained by the movement of the Li ions alone. The model of the phase transition recently proposed by Inbar and Cohen (I. Inbar, R.E. Cohen, Phys. Rev. B 53 (1996) 1193) in which the phase transition is driven by coupled displacements of Li and O could explain the dependence above T_C but more detailed calculations are necessary [4].

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Further work

Future studies will include the phase transition in $LiNbO_3$ and the influence of technologically important codopants like In and Sc on the defect structure.

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Defect evolution in He^+ implanted $LiNbO_3$

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Objectives

Implantation of He^+ into $LiNbO_3$ is an established technique for the production of optical waveguide in this material (Townsend, P.D., Nucl. Instr. Meth. **B 46** (1990) 18-25). The production of extended defects in the end-of-range region of implantation is generally assumed to be responsible for the optical barrier formation but has not been proven so far. A combination of ion beam analysis and electron microscopy studies has been started in order to unveil the details of the defect structure of this system.

Results

Implantations of He^+ into $LiNbO_3$ single crystals with an energy of 20 keV and a dose of 1×10^{16} cm^{-2} have been performed at the Universidad Rio Grande do Sul, Porto Alegre, Brasil. RBS/channeling investigations of virgin, as-implanted and annealed samples show that the damage induced by the He implantation itself is small but increases significantly during heat treatment at temperatures up to 270ºC. This behavior is analogue to that observed during bubble formation in Si (Fichtner, P.F.P., Kaschny, J.R., Kling, A., Trinkaus, H., Yankov, R.A., Mücklich, A., Skorupa, W., Zawislak, F.C., Amaral, L., da Silva; M.F., Soares, J.C., Nucl. Instr. Meth. **B136-138** (1998) 460-464). The amount of damage seems to be independent from the heat treatment time. At higher temperatures an annealing reduces the amounts of defects.

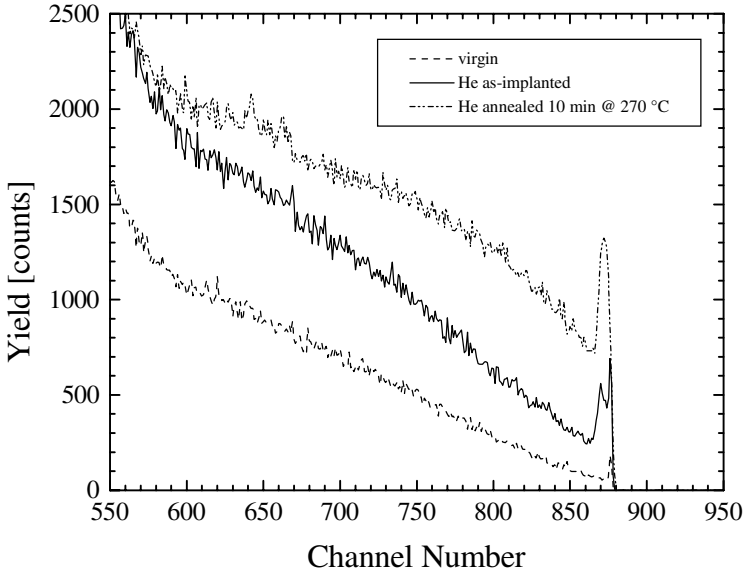

Fig. 1: RBS/channeling spectra of virgin, as-implanted and heat-treated $LiNbO_3$ for 4 MeV He^{++}

Further work

Investigations using transmission electron microscopy are in progress in order to study the occurrence of extended defects, especially of bubbles or cavities in the $LiNbO_3$ single crystals. Further the dependence of defect generation on parameters like implantation dose and energy are planned. Also of interest is the interaction of He with other implants.

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Relationship between growth domains, impurities and gray tracks of $KTiOPO_4$ grown in a high temperature flux

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Objectives

The induced coloration of non-linear optical $KTiOPO_4$ (KTP) single crystals (often called gray tracks) is a major drawback for optical applications. This damage can be produced by electrical currents flowing through the crystal, photon irradiation and by annealing in reducing atmospheres. The susceptibility of KTP for gray tracking is influenced by the growth method, either hydrothermal or flux methods, by the incorporation of OH^- radicals or impurities and by annealing in oxygen rich atmospheres. In this study the relationship of different properties with the gray track formation is investigated using a combination of EPR, optical absorption, Rutherford backscattering spectroscopy and channeling for crystals grown by top seeded solution growth (Martín, M.J., Zaldo, C., Díaz, F., Solé, R., Bravo, D., López, F.J., Rad. Eff. Def. Sol. 136 (1995) 243; Solé, R., Nikolov, V., Koseva, I., Peshev, Ruiz, X., Zaldo, C., Martín, M.J., Aguiló, M., Díaz, F., Chem. Mat. 9 (1997) 2745).

Results

Optical damage (gray tracks) has been induced into undoped and doped c-cut KTP crystals by low intensity ($I_0 \leq 100$ mW/cm^2) continuous-wave illumination with ultraviolet light (λ=364 nm) from an Ar^+ laser. The presence of two crystal regions with different sensibility to the optical damage has been observed. The region with lower induced coloration limits with [100] lattice direction and that with the highest one with [010] direction. These regions have been ascribed to growth domains which may be also observed after thermal reduction of the sample and chemical etching.

The domain more susceptible to damage incorporates more impurities as could be easily detected by RBS. This higher defect density leads also to a reduced channeling effect along the major crystallographic directions of KTP and to a reduction of the chemical etching efficiency. The optical damage consists of a broad band in the visible region (λ_{max}=590 nm) which may be bleached thermally and optically by 488 and 514 nm light. Impurities shift the optical absorption edge of KTP towards lower energies, they induce a minor absorption band at about 488 nm and they enhance the stability of the induced damage at room temperature. The damage kinetics have been found to saturate at a level which depends on the ultraviolet irradiation light density. Donor centers are related to the absorption edge shifting. Some impurities (e.g. W) change their valence due to ultraviolet irradiation. A model considering electron donors and traps has been developed to describe qualitatively the kinetics of the optical damage and its erasure (Zaldo, C., Carvajal, J., Solé, R., Díaz, F., Bravo, D., Kling, A., Relationship between growth domains, impurities and gray tracks of $KTiOPO_4$ grown in a high temperature flux, submitted to J. Phys: Condens. Matter).

Further work

Impurities in $KTiOPO_4$ are of high importance for many applications (e.g. lasers) and therefore investigations on their properties and lattice location will be a wide field for future investigations on this material.

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Optical and Electrical Doping of GaN by Ion Implantation

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Objectives

GaN is a wide band-gap semiconductor that is becoming rapidly important in many areas of the semiconductor technology, mainly optoeletronics. Epitaxially grown GaN is already available commercially and is widely used for the production of blue LEDs. However, as in many areas of semiconductor technology the basic understanding of doping processes and the behaviour of dopants and their interaction with defects still have some open questions. Although the considerable progress made in recent years, several problems still remain. An outstanding one is difficulty to achieve reproducible high level doping by incorporation of dopants during growth because of the strong interaction between dopants and the growth system hardware. Ion implantation is an attractive alternative method of dopant incorporation. It offers the advantages of precise control of dopant concentration, lateral and depth distribution, and is free from limitation imposed by solubility consideration. Although the initial progress, there is still argument about doping of GaN by ion implantation mainly due to the difficulty to remove the implantation damage in order to achieve the complete activation of the dopants. In our studies it is expected that the comparison of detailed information about the microscopic environment of the dopants obtained with hyperfine interaction techniques using radioactive dopants combined with the results of Photoluminescence and RBS/Channelling will lead to a better understanding of the incorporation mechanisms of various impurities. The results will also contribute to improve our understanding of defects on group III nitrides considerably and will aid to develop processes that minimise the influence of defects and optimise the activation of dopants after ion implantation.

Results

After implantation into epitaxial GaN Er and Hf ions are incorporated into substitutional Ga sites. The implantation damage starts to anneal above 600^{o}C as revealed by the hyperfine interaction measurements with the ^{181}Hf probe [1,2]. The increase of the annealing temperature up to 900^{o}C is not enough to remove completely the damage. At this temperature both Er and Hf remains stable in Ga sites and the thickness of the amorphous layer is reduced, indicating the occurrence of some epitaxial regrowth. A further furnace annealing at 1000^{o}C with a proximity cap results in the dissociation of the GaN surface with the formation of a metallic Ga layer. The threshold dose for the amorphization of the GaN films studied is close to 5×10^{15} Er^{+}/cm^{2}, a relatively high value compared with other III-V compounds [4]. The presence of low doses of O in the implanted layer does not influence the annealing process significantly but increases the amount of Er in Ga sites after the annealing at 900^{o}C. At the highest annealing temperatures the presence of O seems to stabilise the Er in substitutional lattice position. Erbium incorporated in the films during the growth also go to Ga sites and display visible luminescence in the green region [5,6].

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Further work

The research in this area will proceed in the next years in order to reach the proposed objectives. A project will be submitted to the PRAXIS programme to get founds that support some of the costs allowing the possibility to offer a post-doc position.

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e^--γ Perturbed Angular Correlations and Emission Channeling at ISOLDE/CERN*

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Objectives

We apply the Electron-Gamma Perturbed Angular Correlations (e^-–γ PAC) and electron Emission Channeling (EC) techniques to study the behaviour of point defects and the lattice site location of elements implanted in high-Tc superconductors and semiconductors. Our work is centred on three approved proposals at CERN/ISOLDE scientific committee: "Studies of High-T_C Superconductors Doped with Radioactive Isotopes" -IS360, "Test of New PAC Probes for Hyperfine Interaction Studies at ISOLDE" -ISC/P66- and "Lattice Location of Transition Metals in Semiconductors" -IS368-.

Results

Studies on the $Hg_1Ba_2R_{(n-1)}Cu_nO_{(2n+2+\delta)}$ high T_C Superconductors (T_C >130 K). It is our aim to characterize at the atomic scale the non-stoichiometric oxygen doping (O_δ) that regulates the injection of charge carriers into the superconducting (CuO_2) planes and to follow microscopic structural transitions along the apical O(2)-Hg-O(2) rows. By using the PAC technique we have shown that the ^{199m}Hg nuclei are highly sensitive to the presence and concentration of the non-stoichiometric oxygen O_δ,. A small structural/electronic change, which occurs in the neighbourhood of the apical oxygen-Hg chain, was found at low temperature below T_C. The atomic behavior of Hg/Au in high quality $YBa_2Cu_3O_{6+x}$ (YBCO) thin films is also being studied, since it has been reported that doping with Hg and Au improves the lattice stability and increases T_C. The simulations of the conversion electron EC data, obtained from the decay of ^{197m}Hg implanted high-quality $Y_1Ba_2Cu_3O_{6+x}$ (YBCO) thin films, have been finished. Together with the PAC data, obtained on the same isotope, they contributed to identify the Hg lattice site to be the Cu(1) site. Further studies are being performed which aim to dope the Infinite Layer Cuprates ($R(CuO_2)$, R = Ca, La, Sr) by ion implantation of alkaline and earth-alkaline elements. These compounds are the most basic family of superconducting cuprates where the charge carrier reservoirs for superconductivity are poorly identified. The work performed on the field of semiconductor doping is centred at applications of the EC and PAC techniques at ISOLDE, which we are using to learn about the lattice site location of implanted Er, Yb, Fe, Cu, As, Ca in Si, GaN and diamond. At Sacavém / ITN, the Rutherford backscattering/Channeling technique (RBS/C,) is applied to study the lattice recovery of implantation defects.

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Further work

New PAC experiments on the local dynamics of Cd in $La_xCd_{1-x}MnO_3$ have started. It is intended to learn if the local electron symmetry and density around the Cd dopant changes during the paramagnetic to ferromagnetic transition. First combined experiments with the high-pressure research center, UNIPRESS, Warsaw, have been done. There, ^{73}As implanted GaN samples have been annealed under thermodynamic equilibrium conditions at high temperature/high nitrogen pressure. After annealing first successful PAC spectra could be obtained that probe the As electronic environment in GaN. New EC experiments are being performed looking forward for determining the lattice site of implanted Fe and Cu in Si, as a function of temperature. Finally, education and training, and excellent and unique results, are the main deliverables of this project.

* Funding: PRAXIS/CERN, 13×10^6 PTE.

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Investigation of iridium diffusion in silicon

A. Rodríguez[1], G. González[1], T. Rodríguez[1], A. Kling, M.F. da Silva, J.C. Soares

Objectives

The diffusion of Ir in Si has so far only been determined from the vapour phase in the temperature range 950-1250º (Azimov, S.A., Umarov, B.V., Yumusov, M.S., Sov. Phys.-Semicond. 10 (1976) 842). For these conditions, the Ir concentration in Si is several orders of magnitude lower than necessary for a silicide formation. In order to investigate this concentration range crystalline and preamorphized Si wafers were implanted with Ir^+ at room temperature with an energy of 130 keV, the projected range being 55 nm. The dose was 3×10^{16} cm^{-2}, which yields a maximum Ir concentration of 8×10^{21} cm^{-3} which is slightly below the value that leads to $IrSi_3$ formation.

Results

The samples were annealed at 550 ºC for 3600 s to regrow the amorphized region of the substrate by solid phase epitaxy. These annealing conditions are sufficient to complete the substrate crystallisation. Further annealings were down by RTA at temperatures ranging from 800-1000ºC. The depth profile was measured using 1.6 MeV He^+ RBS. The diffusion process has been analysed using the solution of Fick´s second law corresponding to an infinite source with a constant diffusion coefficient (concentration and depth independent). Fig. 1 shows as an example the fit of the results to the solution of the diffusion equation for 900ºC for different annealing times. The diffusion coefficient and the activation energy were determined to be $D=2.61\times10^{-14}$ cm^2s^{-1} and E_a=3.3 eV, respectively [2].

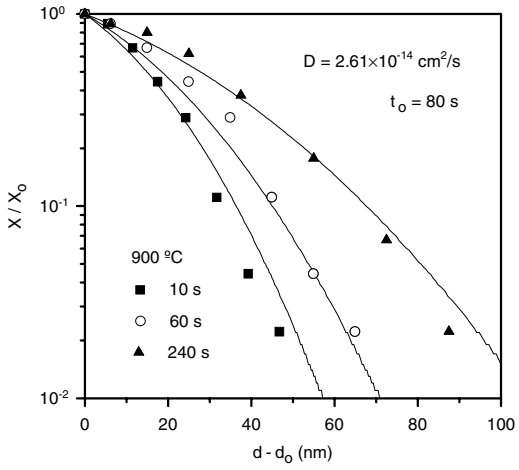

Fig. 1: Fit of Ir depth profiles of samples annealed at 900ºC for different anneal times.

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Further work

In the future samples will be investigated with transmission electron microscopy and X-ray diffraction methods in order to check for the existence of small silicide cluster that may have formed during the diffusion process.

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Optoeletronic properties of amorphous silicon-carbon alloys

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S. Arekat[2], A. Federov[3], M.N. Barberan-Santos[3], F. Giorgis[4], C.F. Pirri[4]

Objectives

Amorphous hydrogenated silcon-carbon alloys (a-$Si_{1-x}C_x$:H) are wide-band gap, thin film semiconductors. Alloys with a low carbon content are used as transparent layers in solar cells and photodetectors while for alloys with high carbon content the emission of visible photoluminescence has triggered interest in the application of this material in large-area electroluminescent devices. The aim of this work was to give a comprehensive overview on the optoelectronic and structural properties of a-$Si_{1-x}C_x$ over the entire compositional range by the application of various experimental methods: photothermal deflection spectroscopy, ESR, the photoluminescence, dark and photoconductivity measurement hydrogen evolution and infrared spectroscopy. Rutherford an resonant elastic backscattering were used in order to gain the crucial information on the stoichiometry of the samples investigated.

Results

The films were prepared using low-power electron-cyclotron resonance 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 source, together with the excitation gas (hydrogen).

The actual composition of the film was determined by conventional RBS and by the use of the broad elastic proton scattering resonance at 1.75 MeV which allows also the acurate determination of very low carbon contents. Regardless of the deposition conditions and source gases used, the optical, structural and transport properties of the a-SiC:H alloys follow simple universal dependencies related to the 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 [1].

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Ion Beam Synthesis of Silicides for Microelectronics Applications*

A.R. Ramos[1], M.R. da Silva[2], J.C. Soares, M.F. da Silva

Objectives

Silicides have been intensively studied for use as low resistivity gates, interconnections and ohmic contacts. Their synthesis through ion implantation offers certain advantages over the more conventional techniques (e.g., it is a "clean" process -isotopic separation of elements- and it allows the formation of certain phases at lower temperatures). We have studied the ion beam synthesis of silicides on normal and porous silicon.

Results

In spite of the high impurity level of porous silicon we were able to form a good quality chromium silicide in most samples. Best results were obtained with high temperature implantations, in which case the impurities were partially expelled from the forming silicide. Further annealing did not seem to help. Very high oxygen contents inhibited silicide formation [1].

Channeled implantation was used to form a stochiometric continuous surface layer of $HfSi_2$. A multi-sep annealing procedure (800/900/1000ºC) was found to favour layer stability compared to a two-step anneal (700/1000ºC). Minimum resistivity values of 60 μΩcm (~bulk values) were obtained after the 1000ºC annealings [2].

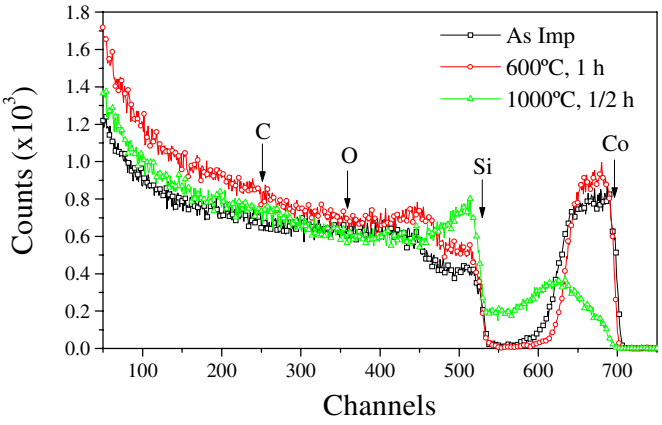

Fig. 1 RBS results of a porous Si sample implanted with $2x10^{17}$ at/cm^2 Co at 350ºC and annealed at different temperatures (600ºC e 1000ºC).

Further work

The annealing and implantation conditions for optimal quality $HfSi_2$ layers need further investigation. In order to study the exact mechanisms at work during silicide formation on porous silicon, other implantations (namely Co) are under way.

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* PRAXIS/2/2.1/FIS/348/94, 36 × 10^6 PTE for three years, ITN, **CFNUL**, INESC.
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Analysis of growth, defects and optical activity of SiC

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Objectives

Silicon carbide is a wide bandgap semiconductor promising for applications in high temperature, high-power devices. Apart of its excellent properties as a material for devices working at extreme conditions, SiC is also a very good luminescent material emitting in the visible spectral range from the blue to yellow. Er doped 6H SiC is also one of the semiconductors interesting for applications in light sources emitting at 1.5 μm, that could be utilised in optical telecommunication systems.

We have deposited high conductivity SiC containing large concentrations of β-SiC crystallites using chemical vapour deposition (CVD), and in a separate study have also used ion implantation to introduce Er as an impurity into 6H SiC. We study the growth, defects, and optical activity of the samples.

Results

We have produced by CVD highly conductive SiC films with very low concentrations of crystalline and amorphous Si. The films generally consist of amorphous and crystalline SiC with varying concentrations of C-C related material. Incorporation of this additional carbon in the films is enhanced at high temperatures. We also find that increased concentrations of CH_4 in the plasma also results in carbon incorporation in the form of C-C bonds in the film having a potentially detrimental effect on film conductivity [1,2].

For the ion implanted samples [3], we have shown that implantation of 6H SiC with Er at 350°C extends the range of useful doses of Er to obtain intense luminescence of Er^{3+} to at least 10^{14} /cm^2. The second very important conclusion is that annealing temperatures may not exceed 1300°C, when amorphisation is prevented. Owing to this relatively low annealing temperature the outdiffusion and losses of Er from the surface and the formation of other polytypes of SiC are also suppressed.

Evidence has been presented that N donors are indirectly involved in excitation of Er^{3+} ions, mediating the transfer of energy from the recombining excitons bound to N-donors. It is also suggested that N atoms are not involved directly in the formation of the dominant luminescent Er-centres.

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Be Binary Pebble Bed Electrical Resistivity Measurements*

E. Alves, A.A. Melo[1], J.C. Soares, M.F. da Silva, M. R. da Silva[2], F. Scaffidi-Argentina[3]

Objectives

In the present ITER breeding blanket design the beryllium is used in form of a binary bed of large (diameter $\Phi = 2 \pm 0.2$ mm) and small pebbles (diameter $\Phi = 0.1$- 0.2 mm), which allows to achieve a bed packing factor of about 80%. Although beryllium has no structural function in the blanket, microstructural and electrical properties are important, as they might influence the material behaviour during both normal and off-normal reactor operating conditions like plasma disruptions. To confirm the conservative assumptions about the electrical resistivity of the beryllium pebble bed made in the course of the electromagnetic analyses for the Reference ITER Breeding Blanket, this properties has been actually investigated.

Results

The resistivity measurements show that in the case of the single size 2 mm pebble bed the resistivity of the bed decreases drastically to about 10^{-4} Ωm by applying an external pressure. The same trend was observed for the single size 0.1-0.2 mm pebble bed but, because of its larger BeO content, 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. However, for low external pressure values the resistivity of the binary bed is coincident with that of the single size 2 mm pebble bed (Electrical and metallographic characterisation of beryllium pebbles, F. Scaffidi-Argentina, E.Alves, M.F. da Silva, A. A. Melo, J.C.Soares, Proc. of the 20th Symposium on Fusion Technology (1998), vol.2, pag.1365). This is due to the fact that in the mixed pebble bed the small pebbles just fill the empty spaces, but they result in additional paths for the current flow only after the electrical contact between small and large pebbles has formed. Above room temperature (~15°C) the resistivity starts to increase. The resistivity follows a linear dependence with the temperature in the pressure and temperature range studied (up to 550°C). After the heating cycle there was a shift of the resistivity to higher values. This increase of the resistivity can be related with an increase of the BeO layer of the pebbles during the measurements at high temperature, which was observed by the RBS measurements [1].

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Future work

Since in a Breeding Blanket the pebbles are accommodated within a Helium purge gas atmosphere, and exposed to intense neutron and gamma irradiation, the gas is expected to get ionised, and this phenomenon might change the electrical resistivity of the complete bed. Therefore, further experiments shall be performed with the pebble bed exposed to an adequate radiation source simulating the actual irradiation conditions as closely as possible. The impact of the radiation level shall be quantified in comparing the results with those obtained without irradiation. This task was approved for financing during 2000.

* EFDA Technology Workprogramme 2000 (EU Tasks V60/3).

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Precise ion beam analysis of advanced materials for nuclear fusion reactors*

E. Alves, L.C. Alves, J.C. Soares, M.F. da Silva

Objectives

SiC composites are one of the advanced materials that are expected to be used in fusion reactor technology. In order to avoid the formation of long-lived isotopes, the knowledge of the concentration levels of all the impurities present in these materials is a very important issue. Lithium compounds will be used for generating tritium. The chemical stability of those Li compounds with the SiC composites in fusion relevant conditions was also investigated.

Results

Macrobeam and microprobe analysis was performed in the SiC fibers used to reinforce the SiC composites and they were found to be very pure (1). On another hand SiC composites were found to contain several impurities. Furthermore, microprobe analysis showed that at least some contamination was present in the form of grains. Those grains contained mainly Fe and were not at the surface but rather distributed inside the matrix, revealing contamination during the manufacturing process.

The exposure of SiC materials to Lithium orthosilicate and Lithium titanate in fusion relevant conditions induces strong solid state reactions (2). The complete surface alteration topography is evident from the microprobe Si X-ray maps and the RBS analysis reveals the formation of an oxide layer as well as a diffusion of Li in samples exposed to both the compounds. An increase of C inside the SiC was also found for the sample exposed to Lithium titanate. XRD results show the surface formation of Li_2SiO_3 in both samples and the presence of SiO_2 only in the sample exposed to Lithium titanate. No energy corresponding to the C-Si bound could be found in the XPS analysis confirming the C surface depletion on both samples. The microprobe PIXE analysis show the presence of metallic Ti and Cr precipitates for the composites exposed to Lithium titanate (3).

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Further work

The surface depletion of C in the SiC composites is going to be further investigated as well as a more detailed study on the surface chemical reactions suffered by the Lithium compounds.

* Funding: Associação EURATOM/IST – SiC/SiCf ceramic composites TTMA-001- ITN sub-contract: 3×10^6 PTE.

U-doped standards for low activation metallic materials

A. Paúl[1] and L. C. Alves

Objectives

Some elements that are always present in the alloys to be used as structural materials in nuclear fusion reactors are undesired impurities that can adversely affect the reduced activation properties of these materials. Those impurities need to be measured accurately with adequate standards. One of the most important elements is uranium due to its easy activation with neutrons and the resultant radioactive family. To measure low levels of uranium, standards of several ppm (between 1 and 100) were fabricated. The alloying route of U in iron-base materials is not easy as U tends to segregate to the slags. An alternative route, based in some preliminary laboratory results, is the fabrication of U intermetallics that can then be used to alloy U in an easier way. Good candidates are binary and ternary U intermetallic with Al, Mn, Si, Sn, Sb and Bi. The final objective of this work is to develop U-doped standards with 1-100 ppm and to test the detection limits for U with PIXE.

Results

Some standards of F-82H and LA-12 alloys were prepared by direct alloying the compounds. In order to obtain the desired U composition metallic U was directly added to the raw materials in the adequate percentage (1, 10 and 100 ppm), then the mixture was melted in an induction furnace under Ar atmosphere and centrifugally casted into 30×15 mm (∅×h) cylindrical ingots. A total of 8 alloys were prepared with this base composition and the expected U concentration. To determine the U concentration in the alloys, PIXE experiment were conducted with broad beam and in the nuclear microprobe with transmission specimens. The alloys with a higher expected U proportion were examined for presence of U. None of the studied specimens presented measurable quantities of U. The fact that no U is present in the alloys is due to the segregation of U to the slag so that the efficiency for alloying U in these steels is so low that the proportion obtained is below detection limits. In order to test the efficiencies of U, two new alloys were prepared and also the slag was analysed for the presence of U. The spectra show the presence of U in the alloys and the slag. Deconvolution of the spectra allows to calculate the percentages of U and the detection limits.

Table 1.U concentration and detection limits in alloys and slags.

Material	F-82H 0.5U	Cr-0.5U	Slag of F-82 0.5UH	Slag of Cr-0.5U
U (ppm)	56	23	10730	9350
D. L	4	3	723	827

Further works

Fabrication of new alloys with lower U contents in order to test the detection limits for this element. These alloys will be made in three ways. First, direct addition of U to the alloy using the efficiency obtained for alloy F-82H. Second, supposing a efficiency of 100% for U already alloyed, to use the F-82H-0.5U alloy to add the desired quantity of U and third alloying of U starting from Al, Mn, Si, Sn, Sb and Bi binary and ternary intermetallics. Microstructural study of the alloy to see if the U is segregating to inclusions or intermetallic phases.

[1] PRAXIS Post-Doctoral.

Metastable alloys in Al-Cr system, produced by ion implantation and laser processing*

O. Conde[1], R.C. da Silva, L. Prudêncio[2], M.F. da Silva, R. Vilar[3], J.C. Soares[1, 4]

Objectives

The objectives centered on the production and study of the microstructure and stability of Al-based surface alloys made by alloying Cr into aluminium substrates, using ion implantation and laser alloying.

Ion implantation was used to produce surface layers containing fine dispersions of Al-Cr intermetallic compound particles in an α-Al supersaturated solid solution. The structure and properties of these materials were studied; the equilibrium concentration distributions of Cr after high fluence implantation, for an extended range of concentrations, were determined; the crystalline structure and lattice parameter of the intermetallic compounds of Al-Cr were studied by advanced electron microscopy and X ray diffraction methods; the thermal stability and transformation mechanisms of these alloys were investigated.

The Al-Cr surface alloys were produced by high fluence ion implantation and laser processing to Cr concentrations well above its solid solubility in Al. Characterisation was performed by glancing incidence X-ray diffraction, transmission electron microscopy and Rutherford backscattering.

Results

High purity polycrystalline Al discs were implanted with Cr fluences in the range $1\text{-}5\times10^{17}/cm^2$. In order to develop knowledge about the conditions leading to the direct formation of Al-Cr intermetallic compounds, implantations of 140 keV Cr^+ ions at different substrate temperatures and also with higher energy, 300 keV, were studied. This was carried along with an extensive annealing programme in order to study the stability and evolution of the so formed phases.

It was found that by ion implantation only the $Al_{86}Cr_{14}$ and $Al_{13}Cr_2$ intermetallic compounds are formed, as opposed to laser alloying which result in the formation of $Al_{13}Cr_2$, $Al_{11}Cr_2$ and Al_4Cr, but not $Al_{86}Cr_{14}$. The conditions for the direct formation of the $Al_{86}Cr_{14}$ and $Al_{13}Cr_2$ intermetallics by ion implantation were established. The stability and transformation of $Al_{86}Cr_{14}$ and $Al_{13}Cr_2$ was accessed, and the temperature domains of stability for each of these intermetallics were identified.

Depending on implantation dose and substrate temperature either one or both phases form in the implanted material, evolving from a solid solution of Cr in Al at low values of the process parameters to the formation of the intermetallics $Al_{86}Cr_{14}$ and $Al_{13}Cr_{12}$ dispersed in the Al matrix as those values increase. GIXRD measurements showed that both compounds form as embedded crystalline precipitates which grow and transform by annealing. The $Al_{86}Cr_{14}$ containing phase forms first and is transformed into $Al_{13}Cr_2$.

The results show that temperature is the main factor determining the formation of the observed $Al_{86}Cr_{14}$ and $Al_{13}Cr_2$ intermetallic compounds by ion implantation. At these high doses, energy and dose will determine the size distribution of the precipitate populations.

For temperatures below 200 ºC no intermetallic compounds of any type are detected: all Cr is retained in the Al host matrix, either dissolved or in Cr-rich aggregates, too small to be detected by GIXRD.

For temperatures ≥ 400 ºC $Al_{13}Cr_2$ is the predominant stable compound.

For temperatures in the range ≥ 250 ºC to < 400 ºC the predominant compound that forms is $Al_{86}Cr_{14}$, which transforms by annealing at temperatures ≥ 400 ºC in the stable form $Al_{13}Cr_2$.

Higher doses result in larger precipitates, ~ 20 nm for $Al_{86}Cr_{14}$, formed by implantation with $2\times10^{17}Cr/cm^2$, to ~200 nm for $Al_{13}Cr_2$, formed by implantation with $5\times10^{17}Cr/cm^2$).

From the project resulted 1 post-graduation (Masters) thesis.

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Further work

The research contract is closed. TEM in the Cr implanted Al samples to access the morphology and size distribution of the precipitates.

* PRAXIS/PCEX/P/FIS/2/96, 10.6 × 10^6 PTE for two years, ITN, **CFNUL**.
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Metastable alloys in Ti-Cr system, produced by ion implantation and laser processing*

R.C. da Silva, L. Prudêncio[1], A.D. Sequeira, O. Conde[2], R. Vilar[3]

Objectives

To develop wear and corrosion resistant Ti based light alloys by alloying Cr into titanium and titanium α-alloy substrates, using ion implantation and laser alloying and to study their microstructure and properties in order to obtain:

- detailed knowledge of the formation of Ti-Cr surface alloys produced by ion implantation and laser processing and of the precipitation of Ti-Cr intermetallic compounds such as $TiCr_2$, or others;
- detailed knowledge of the transport behaviour of Cr in Ti and in the Ti alloys under scrutiny, of the stability and transformation mechanisms of these alloys as a function of temperature;
- identification of the microstructure formed: morphology, size distribution, structure and orientation relationships of the different precipitates formed in the Ti-Cr systems produced by ion implantation and laser alloying; on the nucleation, growth and interaction between these precipitates.

The metastable Ti-Cr and Ti-alloy-Cr surface regions will be produced by high fluence ion implantation, laser processing or a combination of both techniques, in order to develop layers containing fine dispersions of intermetallic compound particles. The distribution of Cr concentration will be followed mainly by Rutherford backscattering, while X-ray diffraction and transmission electron microscopy will be used to study the crystalline structure and lattice parameter of the embedded intermetallic compounds.

Wear and corrosion tests will be carried out to evaluate the functional properties of the materials.

Results

Polycrystalline titanium discs of 99.999% nominal purity were implanted with 140 keV Cr^+ ions to nominal fluences in the range of $1\text{-}20\times10^{17}/cm^2$, at RT and at temperatures up to ~ 800 and 1000 ºC. The samples implanted at RT were also heat treated in vacuum, up to 1000 ºC, in order to compare against high temperature implantations.

Implantations at temperatures from RT up to 800 ºC lead to peak shaped concentration profiles, with maximum Cr concentrations of up to 55 at.%, whereas implantations of the same fluence but at 900-1000 ºC result in very broad Cr profiles with maximum concentration of only 13 at.%. The location of the concentration maxima clearly shifts towards larger depths with implantation temperature.

Upon vacuum annealing at 1000 ºC the Cr concentration profiles do not show any longer in the RBS spectra, whereas PIXE measurements yield a concentration value of 0.3 at.%. At the same time a clear growth of crystalline grains of macroscopic size is detected.

References

Further work

TEM in the Cr implanted Ti samples. Laser alloying Cr with Ti, followed by characterisation work as described for ion implanted samples. Same work as described with α-Ti-based commercial alloys.

* PRAXIS/P/CTM/10037/98, 7.5 × 10^6 PTE for two years, **ITN**, IST, CFNUL.

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Growth and structural properties of thin $Si_{1-x}Ge_x$ layers

A. Rodríguez[1], T. Rodríguez[1], J. Olivares[1], J. Sangrador[1], A. Kling, M.F. da Silva, J.C. Soares, C. Ballesteros[2]

Objectives

Crystalline and poly-crystalline $Si_{1-x}Ge_x$ thin films grown by different methods on Si, SiO_2 and glass substrates have been used in the fabrication of semiconductor devices and are intensively investigated with respect to applications in flat displays. In this study special emphasis has been given to the defect structure and strain evolution in crystalline layers grown by SPE on Si substrates as well as to the crystallization of amorphous films grown by LPCVD on SiO_2 and glass.

Results

The influence of composition and growth temperature on the strain and defect structure of $Si_{1-x}Ge_x$ layers with $0.21 \leq x \leq 0.34$ on Si by SPE has been investigated by Raman spectroscopy and RBS/channeling. The defect structure was studied by TEM. While the Raman data yielded only an averaged strain in the layers strain depth profiles with a high resolution have been extracted from the channeling data showing the dependence of the relaxation on SPE temperature, composition and the influence of boron doping (Rodríguez, A., Rodríguez, T., Kling, A., Soares, J.C., da Silva, M.F., Ballesteros, C., *J. Appl. Phys.* **82** (1997) 2887-2895; Rodríguez, A., Rodríguez, T., Sanz-Hervás, A., Kling, A., Soares, J.C., da Silva, M.F., Ballesteros, C., Gwilliam, R.M., *J. Mat. Res*. **12** (1997) 1698-1705;Rodríguez, A., Rodríguez, T., Kling, A., Soares, J.C., da Silva, M.F., Ballesteros, C., *Nucl. Instr. Meth.* ***B*** **136-138** (1998) 395-399). Three different relaxation mechanism have been identified and characterized [1].

The crystallization kinetics and microstructure of $Si_{1-x}Ge_x$ layers obtained by SPE from LPCVD grown films on SiO_2 and glass have been studied in dependence of Ge content, substrate material and annealing time. It was found that the transient time and the characteristic crystallisation time decrease with increasing Ge content. The films with low Ge content have a strong (111) preferred orientation. No difference in the growth kinetics and grain structure was observed between films grown on SiO_2 and glass [2].

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Further work

A promising alternative to the thermally induced crystallisation of $Si_{1-x}Ge_x$ layers is the ion beam induced recrystallisation which has already been successfully demonstrated for silicon. Investigations on the feasibility of this process for $Si_{1-x}Ge_x$ and its dependence on parameters like the ion species, implantation dose, current density and temperature are in progress.

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Study of a thin Al_2O_3 layer in a magnetic multilayer by resonant scattering

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Objectives

Magnetic multilayer systems showing tunneling magnetoresistance (TMR) are highly interesting for devices like read heads for computer hard disks. Their properties are strongly influenced by the quality of the insulating buffer layer [1-3]. In order to study the properties of an oxide layer with a thickness of less than 20 Å the elastic scattering resonance for helium ions at 3.042 MeV.

Results

The magnetic multilayer system had the following sequence (starting from the surface): MnRh / CoFe / Al_2O_3 / CoFe / NiFe / Ta / Cu / Ta / SiO_2. The investigated sample was annealed at 210ºC. Varying the energy of the He^+ beam from 3.01 to 3.35 MeV enabled the observation of separate O signals for the surface oxide, the thin Al_2O_3 layer and the thick SiO_2 layer on the Si wafer. The energy dependence of the resonant contribution of the elastic scattering at the O was fitted with the RUMP program. The results for the thin Al_2O_3 layer show clearly that the oxygen is confined in the oxide layer [4].

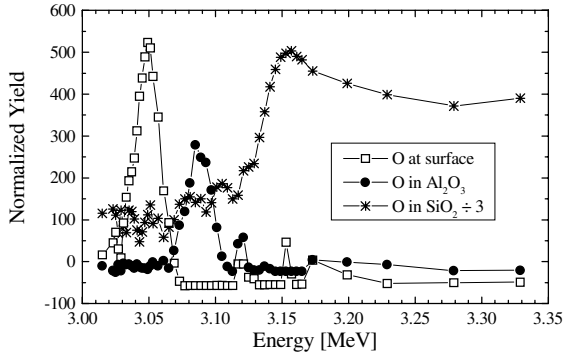

Fig.1: Energy dependence of the resonant scattering yield for the three oxidized regions

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Further work

For the future thinner Al_2O_3 layers (in the region of 9 –12 Å) are required in order to further improve the properties of TMR devices and will be a challenge for the method of resonant profiling.

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Study of the interface diffusion effect in spin dependent tunnel junctions by RBS

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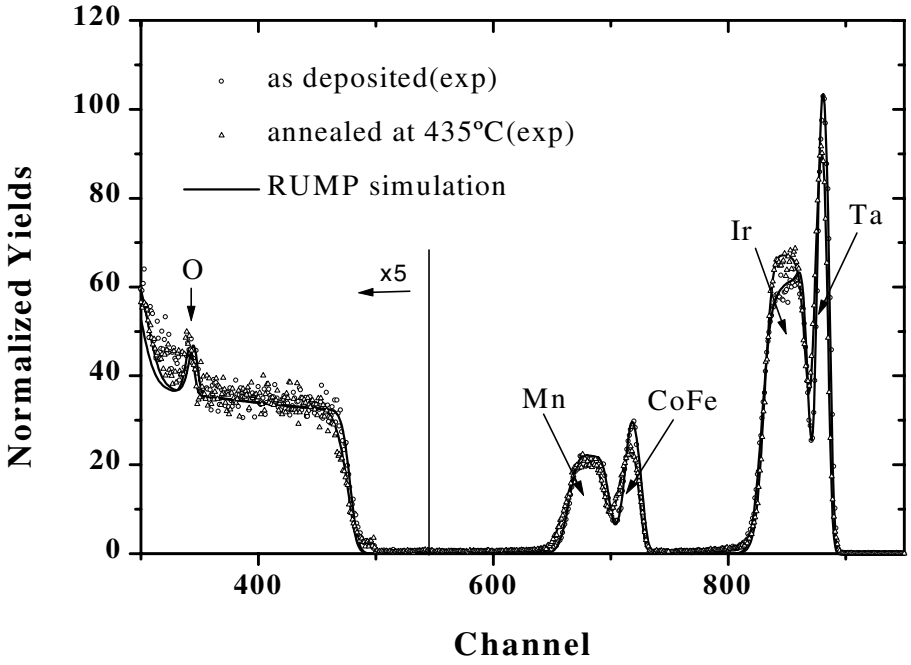
Objectives

Structural changes after annealing of tunnel junctions are directly responsable for the change of magnetic moment observed in those systems. Due to their multilayer structure and to the comparable atomic weight of their constituents, it is difficult to analyse this structural change before and after annealing. In this work, the interface diffusion in specially prepared samples was studied with RBS.

Results

The samples, Si(substrate)/Al_2O_3(1500Å)/MnIr(100Å)/CoFe(40Å)/Ta(30 Å), were studied as-deposited and after anneal at 435°C. RBS studies were performed with a 2.3 MeV He^+ beam with a tilt angle of 82°. The signals of CoFe and Mn, Ta and Ir were clearly separated. The interface diffusion between the CoFe and MnIr layers was observed.



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Further work

Investigations using heavy ion backscattering are in progress in order to get better mass resolution.

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Measurement of (p,p) non-Rutherford elastic scattering cross sections for C, O, N, Si and *Al*

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Objectives

Light impurities contamination (namely C and O) plays an important role in phase formation during high dose ion implantation for silicide formation [1]. In order to improve the sensitivity of RBS to these elements, proton beam measurements at resonant energies have to be made. The exact cross sections have to be experimentally determined, as they have a strong angular dependence and are difficult to model analytically (Amirikas, R., Jamieson, D.N., Dooley, S.P., Measurement of (p,p) elastic cross sections for C, O and Si in the energy range 1.0-3.5 MeV, *Nucl. Instr. and Meth* ***B*** **77** (1993) 110-116). Measurements for our particular detection geometry had never previously been made.

Results

The cross sections for Al, N, Si and O were measured on thin AlN and SiO_2 films, which were sputter deposited on carbon substrates. The cross section for C was measured on a self-supporting C thin film. All cross sections were measured in the 0,5 to 2,5 MeV range, for 140 and 180 degrees scattering angles. The results obtained for C, O and Si agree well with theoretical predictions (see Gurbich, A.F., Evaluation of non-Rutherford proton elastic scattering cross section for carbon, *Nucl. Instr. and Meth* ***B*** **136-138** (1998) 60-65, and references therein). All cross sections measured were used to simulate spectra taken from known standard samples with excellent results.

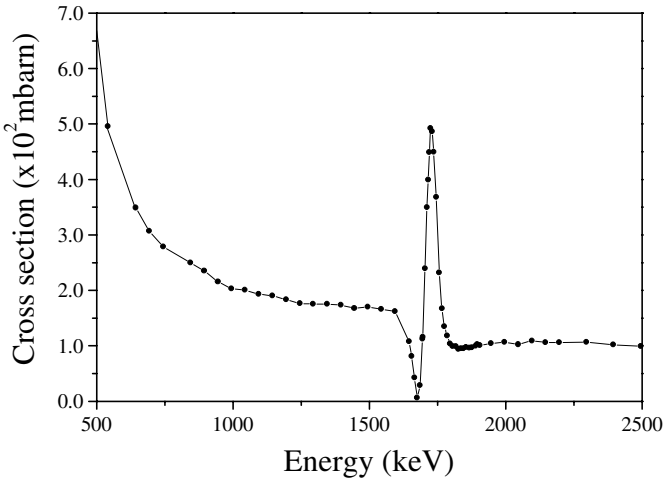

Fig. 1 Measured crossection for the reaction ^{12}C(p,p)12C for the 140 scattering angle.

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The Ion Beam Analysis DataFurnace

N.P. Barradas, C. Jeynes[1], R.P. Webb[1]

Objectives

Development of a computer program for the fully automated analysis of Ion Beam Analysis data, including Rutherford backscattering (RBS), elastic recoil detection analysis (ERDA), nuclear reaction analysis (NRA), neutron depth profiling (NDP), Medium Energy Ion Scattering (MEIS) and particle induced x-ray analysis (PIXE), using the Simulated Annealing (SA) algorithm.

Results

Rutherford backscattering spectrometry and related techniques have long been used to determine the elemental depth profiles in films a few nms to a few microns thick. However, although obtaining spectra is very easy, solving the inverse problem of extracting the depth profiles from the spectra is not possible analytically except for special cases. It is because these special cases include important classes of samples, and because skilled analysts are adept at extracting useful qualitative information from the data that ion beam analysis is still an important technique.

We have recently solved this inverse problem using the simulated annealing algorithm. We have implemented the solution in the "IBA DataFurnace" code [1-4], which has been developed into a very versatile and general new tool that analysts can now use to rapidly extract quantitative accurate depth profiles from real samples on an industrial scale. The code developed so far can be applied to RBS, ERDA, MEIS and non-resonant NRA, for any incident ion and energy. We have applied the code to many different systems [5-10].

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Further work

We shall extend the capabilities of the IBA DataFurnace to NDP and PIXE.

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Bayesian Inference analysis of Ion Beam Analysis data

N.P. Barradas, C. Jeynes[1], R.P. Webb[1], M. Jenkin[1], P. K. Marriott[1]

Objectives

Ion Beam Analysis techniques are geared towards the non-destructive determination of elemental depth profiles. However, no general method has so far been presented that allows one to calculate error bars on the depth profiles obtained. We have developed a computer code based on Bayesian inference with the Markov Chain Monte Carlo algorithm that performs that task.

Results

Rutherford backscattering spectrometry (RBS) and other Ion Beam analysis techniques are a well established and powerful technique for determining the elemental composition of thin films using an energetic light ion beam (say, 1.5MeV $^4He^+$). Because the data analysis is very simple in principle, RBS can be used to obtain quantitative and traceable information about homogeneous films.

However, most interesting real films are not homogeneous. In such a case it is usually very hard to devise a computational method transparent enough to obtain depth profiles from the spectrum whose accuracy is traceable. Moreover, most spectra obtained from real samples are sufficiently complex to preclude manual extraction of any accurate depth profiles at all in a reasonable time.

We have developed a computational method that is able to generate information about the errors involved in the depth profiles obtained [1-4]. These methods are based on Bayesian statistics, and give, for the first time, analysts a tool which they can use routinely to determine the reliability of the information they extract from the data.

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Further work

The calculations involved in the method developed are computationally expensive. We shall optimise and improve the existing algorithm, and apply it to more real-life cases.

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Nuclear microprobe set-up*

L.C. Alves[1,2], M.B.H. Breese[3], M.R. Silva[2], A. Paúl[4], M.F. Silva[1,2], J.C. Soares[1,2]

Objective

To have fully operational the nuclear microprobe with well known parameters.

Results

Although a good beam spatial resolution was attained in the first experiments, a deeper understanding of the working conditions was needed. For optimising the set-up, parameters as beam line intensity and focusing, object and collimator slits apertures, have been varied and final results recorded. Measured lens demagnification was compared with theoretical values (PRAM, obtained from D. N. Jamieson, University of Melbourne, Australia) and main limitations identified as due to the restricted beam line length. Brightness was also measured and the value of ~1 pA/(μm^2 $mrad^2$ MeV) was found to be similar to other microprobe set-ups (Breese, M.B.H.,. Jamieson, D.N., King, P.J.C., *Materials Analysis using a Nuclear Microprobe*, Wiley, New York, 1996). The whole beamline was checked with a search coil for stray fields that would degrade the resolution (Watt, F.,. Choo, T.F , Lee, K.K., Osipowicz, T., Orlic, I., Tang, S.M., *Nucl. Instr. Meth.* ***B*** **104** (1995) 101.). Several nearby components were found which gave large high frequency fields, particularly vacuum gauges measuring devices, a turbo control unit and the computer monitor. These were either changed or moved further away from the beam line.

The present operational characteristics and main features of the set-up are described in the following text. The analysis chamber is vacuum pumped and a pressure of 10^{-6} mbar attainable. A front viewing stereo microscope at a backward angle of 45º is used for focusing the beam on a glass and for identification of the sample area to be analysed. The focused beam is raster scanned over the sample surface using magnetic dipoles and the maximum area that can be scanned, using 2.3 MeV protons, is 2.5×2.5 mm^2. Up to eight detectors can be used to build up 256×256 pixels images of the sample. The system allows point analysis of any single spot within the scanned area; in routine analysis, samples with surface dimensions up to 1.5×4 cm can be mounted on a sample holder that is coupled to a *xyz* manipulator. It is possible to use a fixed stage sample holder with normal beam incidence or a one-axis goniometer. The system is ready to perform PIXE (Particle Induced X-ray Emission), RBS (Rutherford Backscattering Spectrometry), STIM (Scanning Transmission Ion Microscopy) and CSTIM (Channelled STIM) type of analysis. For PIXE analysis a Si (Li) detector with an active area of 80 mm2 is located at a backward angle of 45º. For RBS analysis the former 25 mm^2 surface barrier detector was substituted by a 200 mm^2 one positioned at a backward angle of 40º in the Cornell geometry. For the transmission analysis there is a rotating flange that allows a particle detector to be wound on to the beam axis. As resolution is not the major issue in transmission analysis, the initial surface barrier detector used was substituted by a cheap silicon photodiode. Apart from the immediate qualitative analysis that can be extracted from the generated elemental or energy band maps, quantitative analysis is now only possible from the RBS analysis. Quantitative PIXE results are still dependent on a proper detector calibration.

Further work

Although several implementations are planned in a short period the major two are the development of an image system based on the emission of secondary electrons and the construction of a two-axis goniometer capable of channelling analysis in small crystals. Some initials test for the image system have already been undertaken but further work is needed.

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[4] PRAXIS Post-Doctoral.