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Preface

The Ion Implantation Laboratory (IIL) is an ion beam center at the Institute of Physics (IF) at the Federal University of Rio Grande do Sul (UFRGS), Brazil. The IF-UFRGS is located in the city of Porto Alegre (state of Rio Grande do Sul) and it is ranked as the most important research center of Physics in southern Brazil.

The IIL has three accelerators that provide a wide variety of positive ions in a broad energy range and are used by tens of researchers from Brazil and other countries from Latin America for ion-beam analysis, ion implantation and ion irradiation. Several beam lines with different analytical techniques are available to scientists from different fields. The techniques are:

- **PIXE** (Particle-Induced X-ray Emission): provides elemental concentrations of the order of part per million;
- **RBS** (Rutherford Backscattering Spectrometry): used for characterization of different structures, including multi-layered targets;
- **NRA** (Nuclear Reaction Analysis) and **NRP** (Nuclear Reaction Profiling): ideal to detect and profile specific isotopes respectively;
- **Microprobe**: allow the use of techniques like PIXE, RBS and STIM with micrometer beam size;
- **MEIS** (Medium Energy Ion Scattering): it is a high-resolution RBS technique with isotope-separation capability;
- **Ion Implantation**: used for modification of materials under controlled parameters.
The infrastructure of the laboratory includes a large variety of ovens, a cleaning room, a MEV microscope and photoluminescence laboratory. A fully dedicated workshop allows the maintenance of the laboratory in a regular basis. A general view of the laboratory is shown below, featuring the beam lines of the Tandetron accelerator.

This is the forth issue of our activities and covers two years (2013-2014) of scientific production of all staff members, post-docs and students of the IIL. Comparing this issue with the previous one where we have celebrated our 30th foundation anniversary, we observe a change in our scientific production. This reflects the retirements and new entries of researchers in our lab. In fact many young and talent researchers have joined the IIL recently and have already improved our scientific production in spite of strong difficulties arising from the increase of academic duties in our university and restriction of budget. After all we are committed to go further.

Pedro L. Grande
Head of Ion Implantation Laboratory
Staff - 2014

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Facilities

Besides the accelerators the laboratory has the following facilities

a) Photoluminesce laboratory

b) Cleanroom

c) Furnaces, ovens and reactor chambers

d) Scanning electron microscope
Oxygen reduction in low-energy ion implantation of silver ions in titanium


Introduction

Biocompatible materials have been widely studied especially to confer antibacterial properties to these materials, e.g. modifying them by implantation of silver ions at near surface region. The biological results are encouraging, given its antimicrobial activity which is effective against the bacteria E. coli and S. aureus. Titanium medical class stands out for its high degree of biocompatibility. However, this process can have its efficiency harmed by the presence of oxygen, which reacts at room temperature with titanium forming a thin oxide layer. This layer can act as a barrier hindering both the process, for implantation of ions and for releasing of Ag\(^+\) from inside the metal.

To remove the oxide layer three kinds of cleaning prior implantation process were evaluated: chemical etching by immersion in common industrial acid solution, plasma etching and chemical etching followed by plasma.

Results

The performance of Monte Carlo simulations shows is crucial in determining the energies to be used in deployments, as predicted approximately profiles dose/depth of Ag\(^+\) implanted in titanium.

The characterization physicochemical technique was used showed that RBS quantitative doses of silver, where concentrations were in the order of magnitude of $10^{15}$ atoms/cm\(^2\) results.

The GD-OES indicates that silver is in fact located on the steel surface, indicating a depth of penetration of less than 10 nm silver. The oxygen profile in depth qualitatively shows a reduction for treated samples.
Research outlook

Plasma etching has the advantage to occur in the same implantation chamber and the process may be considered environmentally friendly because it doesn’t use chemicals therefore does not produce effluent.

Accordingly, defends the hypothesis to make this process of surface modification on an industrial scale, although data are lacking to prove your income and the total cost of the treated product. However, there is a perspective of a total gain hundred times in yield compared to the high energy ion implantation.

Publications

**GeO$_2$/Ge Structure Submitted to Annealing in Deuterium**

*C. Radtke*

**Introduction**

The downscaling of the silicon (Si) based metal oxide semiconductor field-effect transistors (MOSFET’s) lead these devices to their physical and technological limits. New semiconductors are demanded to continue the scaling process. Germanium (Ge) is considered to be an interesting material for replacing Si due to its high charge carrier mobility, narrow bandgap, and low dopant activation temperatures. Particularly, it presents the highest hole mobility among all the known semiconductor materials, being the most promising contender for PMOS devices. However, the passivation of the Ge surface is still an issue. In this work, we investigated the incorporation of H in GeO$_2$/Ge upon thermal treatments. GeO$_2$ can be formed, intentionally or not, on the top of the Ge substrate, acting as the major passivation agent. Thus, understanding the effects of H incorporation in this layer is crucial to tailor efficient passivation routes for Ge based devices.

**Results**

Figure 1(a) shows D areal densities (determined by nuclear reaction analysis) as a function of the annealing temperature of thermally oxidized Si and Ge substrates. Following annealing at 250 °C, D was not detected within the sensitivity of the technique. For 350 °C and above, GeO$_2$/Ge samples incorporate higher amounts of D than their SiO$_2$/Si counterparts. This observation is probably related to the production of oxygen vacancies in GeO$_2$/Ge samples which constitute incorporation sites for D. In both cases, a maximum of D incorporation is observed for samples annealed at 450 °C. Quantification of the remaining $^{18}$O amount of each sample following these treatments was performed to probe the stability of such structures (Fig. 1 (b)). It’s clear the lower thermal stability of GeO$_2$/Ge structures with respect to SiO$_2$/Si counterparts.
The present results constitute important benchmarks to the choice of forming gas annealing (FGA) parameters of Ge based devices. They also provide a deeper insight into the physico-chemical modifications and related electrical characteristics of Ge MOS structures submitted to FGA.

D incorporation in GeO$_2$/Ge structures following D$_2$ annealings was investigated. Higher D concentrations were obtained for GeO$_2$/Ge samples in comparison to their SiO$_2$/Si counterparts annealed in the same conditions. Oxygen vacancies produced during the annealing step in D$_2$ constitute defect sites for D incorporation analogous to defects at the SiO$_2$/Si interfacial region. These vacancies are created both at the GeO$_2$/Ge interface and at the GeO$_2$ surface. The latter mechanism results from the interaction of D$_2$ with the oxide. Besides D incorporation, volatilization of the oxide layer is also observed following D$_2$ annealing. In parallel to this volatilization, oxide stoichiometry is also modified: reduction of GeO$_2$ to metallic Ge takes place.

**Research outlook**

The present results constitute important benchmarks to the choice of forming gas annealing (FGA) parameters of Ge based devices. They also provide a deeper insight into the physico-chemical modifications and related electrical characteristics of Ge MOS structures submitted to FGA.

**Publication**

Passivation of Native Point Defects in ZnO Nanowires by SiO\textsubscript{2} Sputtering Deposition


Introduction

Zinc Oxide (ZnO) is a transparent semiconductor with a direct band gap of about 3.37 eV. Its large exciton binding energy (60 meV) makes it suitable for short-wavelength optoelectronics devices operating at room temperature. Nevertheless, photoluminescence (PL) experiments performed on nanostructured ZnO usually present strong deep-level-emission (DLE) band related to native point defects such as oxygen vacancies (V\textsubscript{O}), zinc vacancies (V\textsubscript{Zn}), interstitial oxygen (O\textsubscript{i}) and interstitial zinc (Zn\textsubscript{i}). In the case of ZnO nanowires (NWs), the higher surface to volume ratio increases the number of surface-related trap states, directly affecting the near-band-emission (NBE). In this work, atomic defect passivation mechanisms are applied aiming to improve the NBE emission of ZnO nanowires.

Results

Vertically aligned ZnO nanowires were grown by vapor-liquid-solid (VLS) mechanism on sapphire substrates (Fig. 1). The nanowires were then coated with SiO\textsubscript{2} by RF sputtering. Transmission electron microscopy (TEM) and electron energy loss spectroscopy (EELS) confirm the presence of an approximately 10 nm SiO\textsubscript{2} layer surrounding each nanowire (not shown). The PL measurements showed a strong decrease in the visible band emission consistent with a V\textsubscript{O} passivation mechanism during SiO\textsubscript{2} deposition (Fig. 2 (a)). However, annealing treatments at increasingly higher temperatures favored the oxygen desorption and the increase of deep-level states again (Fig. 2 (b)).
Laboratório de Implantação Iônica

Research outlook

SiO$_2$-ZnO core-shell nanowires were obtained through SiO$_2$ sputtering deposition on vertically aligned ZnO nanowires forest. A strong oxygen vacancies passivation were observed resulting in a drastic suppression on visible light emissions in favor of UV ones.

Publications

Tunnel Barries for Graphene
Spintronics

B. Canto, J. E. Schmidt, D. L. Baptista

Introduction

Graphene is a potential material for spintronic applications because of the combination of its expected long spin lifetime and high electron mobility. However, previous studies showed that electrical spin injection from such ferromagnetic electrodes in direct contact with graphene is not effective because of the conductance mismatch. Instead, the use of a thin insulating layer (few nanometers) acting as a tunnel barrier between the graphene layer and the metal electrodes has proven to be an effective solution. Nevertheless, complete control of tunneling barrier fabrication on graphene sheets is still distant. Barrier structural and chemical non-uniformities seem to play a crucial role in the experimental spin relaxation time values.

Results

A detailed investigation of the structural and chemical characteristics of thin evaporated $\text{Al}_2\text{O}_3$ tunnel barriers of variable thickness grown onto single-layer graphene sheets was performed. Advanced electron microscopy (HRTEM, STEM) and spectrum-imaging techniques were used to investigate the Co/$\text{Al}_2\text{O}_3$/graphene/$\text{SiO}_2$ interfaces. Direct observation of pinhole contacts was achieved using FIB cross-sectional lamellas. Spatially resolved EDX spectrum profiles confirmed the presence of direct point contacts between the Co layer and the graphene (Fig. 1).
The high surface diffusion properties of graphene led to cluster-like $\text{Al}_2\text{O}_3$ film growth, limiting the minimal possible thickness for complete barrier coverage onto graphene surfaces using standard Al evaporation methods. The results indicate a minimum thickness of nominally 3 nm $\text{Al}_2\text{O}_3$, resulting in a 0.6 nm rms rough film with a maximum thickness reaching 5 nm.

**Research outlook**

**Publications**


Energy deposition of H and He ion beams in hydroxyapatite films

R. C. Fadanelli and M. Behar

Introduction

Ion-beam cancer therapy is a promising technique to treat deep-seated tumors; however, for an accurate treatment planning, the energy deposition by the ions must be well known both in soft and hard human tissues. Although the energy loss of ions in water and other organic and biological materials is fairly well known, scarce information is available for the hard tissues (i.e., bone), for which the current stopping power information relies on the application of simple additivity rules to atomic data. More knowledge is needed for the main constituent of human bone, calcium hydroxyapatite (HAp), which constitutes 58% of its mass composition.

In our work, the energy loss of H and He ion beams in HAp films has been obtained experimentally. The experiments have been performed using the Rutherford backscattering technique. These measurements are used as a benchmark for theoretical calculations (stopping power and mean excitation energy) based on the dielectric formalism together with the MELF-GOS (Mermin energy loss function-generalized oscillator strength) method to describe the electronic excitation spectrum of HAp. Finally, the mean excitation energy is used to determine the depth-dose distributions needed for ion-beam cancer therapy.

Results

At high proton energies, the experimental stopping power values are compatible with those provided by the different models. Besides, the agreement for the He ion beam between the experimental stopping data and the calculated results is rather good, in particular around the maximum stopping power.
Research outlook

An important outcome of this work for the stopping of H and He beams in HAp is the successful application to the calculation of the energy loss of charged particles in human cortical bone, whose presence intercepting the ion-beam path in cancer treatment planning must be properly taken into account.

Publications

D$_2^{18}$O annealing effects in SiO$_2$/SiC structures

F. C. Stedile

Introduction

The properties of silicon carbide (SiC) make it a suitable semiconductor to replace Si in devices used in high power, high frequency, and/or high temperature applications. Although recent advances in reducing the interface state density ($D_{it}$) in the SiC/SiO$_2$ interface allowed metal-oxide-semiconductor (MOS) devices based on SiC to be commercially available, several aspects must still be investigated to allow to explore their full potentialities. For example, it is known that interaction with water vapor has a major influence in electrical properties of both Si and SiC devices. To better understand these effects, annealings in isotopically enriched water vapor ($D_2^{18}$O) of SiO$_2$/SiC structures formed following different routes (thermal oxidation and/or sputtering deposition) were investigated.

Results

Deuterium areal densities incorporated from water vapor annealings in different temperatures were obtained by D($^3$He,p)$^4$He nuclear reaction analysis (NRA) which results are presented in Figure 1 (left). The highest amount of D was obtained in the sample with the SiO$_2$ film deposited by sputtering, route that presented the highest negative effective charge concentration and leakage current, suggesting that the D incorporation occurs in defects in the structure that exist prior to the annealing.
Figure 1 – (left) Deuterium areal densities in SiO$_2$/SiC samples after annealing in D$_2^{18}$O performed at different temperatures. SiO$_2$/SiC samples prior the water annealing were synthesized according to three different routes: oxidized in O$_2$ at 1100 °C for 10 h (thermally grown SiO$_2$ films); only with a SiO$_2$ film deposited by sputtering (deposited SiO$_2$ film); or oxidized for 5 min followed by SiO$_2$ film deposition by sputtering (5 min in $^{18}$O$_2$+SiO$_2$ deposition). Bars correspond to experimental accuracy of 10%. (right) Experimental (symbols) excitation curves of the $^{18}$O(p,α)$^{15}$N nuclear reaction around the resonance at 151 keV and the corresponding simulations (lines) for a SiO$_2$ film 20 nm thick thermally grown on SiC submitted to D$_2^{18}$O annealing at 600 °C and for SiO$_2$ films 23 nm thick deposited by sputtering and submitted to D$_2^{18}$O annealing at 600 or at 800 °C. Inset: $^{18}$O profiles obtained in the simulations using the same line types and colors. $4\times10^{22}$ $^{18}$O/cm$^3$ corresponds to the oxygen concentration in stoichiometric SiO$_2$.

$^{18}$O profiles were determined by nuclear reaction profiling (NRP) using the narrow resonance in the $^{18}$O(p,α)$^{15}$N cross-section curve and are presented in Figure 1 (right). Isotopic exchange between oxygen from the water vapor and oxygen from SiO$_2$ films deposited on 4H-SiC was observed in the whole depth of the films, differently from the behavior of SiO$_2$ films thermally grown on 4H-SiC. Such result evidences that the route used in the SiO$_2$/SiC formation also influences the oxygen incorporation.

Research outlook

The present results indicate that the route to obtain the SiO$_2$ film on SiC has a major role in the water vapor interaction. Further investigations intend to better understand the consequences of such interactions.

Publications


Thermally-driven H interaction with HfO$_2$ films deposited on Ge(100)

G.V. Soares

Introduction

There has been significant research on high-mobility semiconductors due to their potential to replace silicon (Si) in future MOSFETs. Germanium (Ge) has the highest hole mobility of all semiconductor materials, however, it has been reported that high-k films on Ge present a high interface state density ($D_{it}$). In this way, different approaches have been investigated in order to improve the electrical characteristics of the dielectric/Ge interface, such as the use of hydrogen post-deposition annealing (PDA).

Results

D uptake in HfO$_2$ films crystallized at 500°C in the cubic phase are show in Fig. 1, which shows D areal densities as a function of the D$_2$ annealing temperature for 5 (squares) and 100 nm (circles) thick HfO$_2$ films on Ge submitted (full symbols) or not (open symbols) to a previous N$_2$ annealing. At 300°C, D incorporation is the same for both thicknesses ($5 \times 10^{13}$ D.cm$^{-2}$), indicating that the crystalline structure and/or the thickness of the film play a minor role in D concentration at this temperature. This result corroborates that most of the D is incorporated in the interface region of the HfO$_2$/Ge. At higher annealing temperatures, a higher D incorporation is observed in films annealed in N$_2$, as well as in the thicker films. This suggests that (i) D is incorporated in the bulk of the HfO$_2$ films for temperatures above 400°C and (ii) HfO$_2$ cubic phase presents more sites for D incorporation. This second point is probably related to the formation of a higher density of grain boundaries when compared to the amorphous HfO$_2$, since the films crystallized in the cubic phase also present some portions of the monoclinic phase.
Research outlook

These results evidence that hydrogen annealing induces physico-chemical modifications in HfO$_2$/Ge structures beyond passivation of interfacial defects like in the SiO$_2$/Si system. The control of such modifications is a key issue for obtaining dielectric/Ge structures with the desired characteristics. Future works will focus in the use of gate stacking aiming at further reduction in the electric active defects at the dielectric/Ge interface.

Publications

SiN_xO_y Films Deposited by Reactive Sputtering

G. Sombrio, P. Franzen, R. Maltez and H. Boudinov

Introduction

Silicon is by far the most used material in the microelectronics industry. The reasons for this preference are its excellent electrical and mechanical properties. However, even with its favorable characteristics, silicon has limitations. Silicon being an indirect band gap semiconductor is a poor light emitter. In the current state of device integration, it is highly desirable to integrate the light source inside the same chip. Many attempts were made to eliminate this obstacle and the problem is a subject of continuing research. Some of the most promising results were obtained by creating silicon nanostructures embedded in dielectric materials, such as porous silicon, silicon quantum dots and erbium doped silicon quantum dots. Those low-dimensional structures disturb the band gap configuration, increasing the radiative recombination rate of the excitons and therefore can result in improved light emission.

Results

In this work, we used samples prepared by reactive sputtering to create layers of silicon oxy-nitride. By carefully controlling the amount of nitrogen, argon and oxygen mixture in the sputtering chamber we controlled the composition and the subsequent photoluminescence emission.

**TABLE I**: Compositions of the samples, extracted from RBS spectra

<table>
<thead>
<tr>
<th>Sample</th>
<th>Si (%)</th>
<th>N (%)</th>
<th>O (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL1</td>
<td>31</td>
<td>56</td>
<td>13</td>
</tr>
<tr>
<td>PL2</td>
<td>43</td>
<td>57</td>
<td>-</td>
</tr>
</tbody>
</table>
Research outlook

RF reactive sputtering technique from Si target was employed for the fabrication of $\text{SiN}_x\text{O}_y$ samples. The presence of UV band emission with an energy of about 3.8 eV (320 nm) was demonstrated, when the oxygen concentration increases, so this emission is possible to be explored for UV electroluminescence applications.

Publications


Insulating characteristics of polyvinyl alcohol for organic electronics

E.A. van Etten, E.S. Ximenes, L.T. Tarasconi, I.T.S. Garcia, M.M.C. Forte and H. Boudinov

Introduction

Great progress has been reported on the development of organic field effect transistors (OFET), which became a very promising technology to gain space on low cost, high volume and flexible applications. OFET can be constructed on different substrates imparting many unconventional properties to the device, such as biocompatibility. There have been a large amount of research groups reporting advances on the semiconductor side of the development, but much less attention has been paid on the development of a suitable organic dielectric layer. The utilization of a polymeric gate insulator is likely to be an important step in developing an all-organic transistor and flexible electronics. The chemical structure of a polymer gate dielectric can impart various surface and bulk properties that strongly influence a device’s performance, making its composition an important factor affecting the fabrication and performance of OFETs.

Results

The goal of this work is to evaluate the effect of different molecular weight, hydrolysis degree and reticulation on the performance of the polyvinyl alcohol (PVA) when used as a dielectric material in organic field effect transistors. Three types of PVA were obtained from Sigma-Aldrich were evaluated. The physical characteristics of these polymers are described in the Table I. Ammonium Dichromate (ADC) was used as a cross linking agent.

TABLE I. PVA physical characteristics

<table>
<thead>
<tr>
<th></th>
<th>Molecular Weight (MW)</th>
<th>Degree of Hydrolysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>PVA 1</td>
<td>31 – 50k</td>
<td>87 – 89</td>
</tr>
<tr>
<td>PVA 2</td>
<td>31 – 50k</td>
<td>99</td>
</tr>
<tr>
<td>PVA 3</td>
<td>146 – 186k</td>
<td>87 – 89</td>
</tr>
</tbody>
</table>
Fig. 1 presents the insulation response of the 6 studied samples. Considering that the insulation effect on any layer is strongly dependent on its thickness, we normalized the responses to the electrical field, by dividing the applied voltage by the dielectric thickness.

**Research outlook**

It was demonstrated that PVA is a promising candidate to be used as a gate dielectric in OFET structures due to its suitable properties such as low leakage current, high dielectric constant, simple application and good film formation properties. It also permits the engineering of its structure, through control of cross linkage, hydrolysis degree and molecular weight, which can be used to optimize the characteristics of the film depending of the desired properties. The major impact on the polymer properties is given by reticulation. The choice of PVA for an organic insulator must be on cross-linked polymer with high molecular weight and incomplete hydrolyzation.

**Publications**


Elemental Quantification of Large Gunshot Residues

J. F. Dias

Introduction

In the present work we embarked on the evaluation of the Sb/Pb, Ba/Pb and Sb/Ba elemental ratios found in relatively large particles (of the order of 50 - 150 µm across) ejected in the forward direction when a gun is fired. These particles are commonly referred to as gunshot residues (GSR). The aim of this work is to compare the elemental ratios of the GSR with those found in the primer of pristine cartridges in order to check for possible correlations. To that end, the elemental concentration of gunshot residues and the respective ammunition were investigated through PIXE (Particle-Induced X-ray Emission) and micro-PIXE techniques. The ammunition consisted of a .38 SPL caliber (ogival lead type) charged in a Taurus revolver. Pristine cartriges were taken apart for the PIXE measurements. The shooting sessions were carried out in a restricted area at the Forensic Institute at Porto Alegre. Residues ejected at forward directions were collected on a microporous tape. The PIXE experiments were carried out employing 2.0 MeV proton beams with a beam spot size of 1 mm². For the micro-PIXE experiments, the samples were irradiated with 2.2 MeV proton beams of 2 x 2 µm².

Results

The analysis of the primer revealed that barium is found in relatively large concentrations, followed by lead and antimony. However, it must be stressed that the composition of each ammunition may vary according to the manufacturer and its purpose. Different applications may require particular compositions. Therefore, the results shown here relate specifically to the ammunition under study.
The present study will be extended to other types of ammunition in order to provide a broader perspective on the subject.

The present results do not indicate any degree of correlation between the elemental concentrations of the primer and the GSR. The degree of dependence of these results in the ammunition and the firearm employed in the present study can be assessed by new measurements were these parameters are changed.

The relative concentrations of barium, antimony and lead found in the residues shows that there is no correspondence with those results obtained for the primer. In this case, lead becomes the element with the highest concentration of all. Clearly, a complex mechanism of nucleation during the burnout takes place. On one hand, lead stemming from other parts of the cartridge take part in this process. But the increase of lead alone cannot explain the differences obtained for the primer and the GSR. Barium and antimony also have substantial changes during the burnout, leading to the results shown here.

Research outlook

The presente study will be extended to other types of ammunition in order to provide a broader perspective on the subject.

Publications

Magnetic interactions in exchange-coupled yet unbiased IrMn/NiCu bilayers

R. Cichelero, A. Harres, K. D. Sossmeier, J. E. Schmidt, J. Geshev

Introduction

Very recently, the remanence plots method has been extended to systems with shifted hysteresis loops resulting in a number of distinct remanence plots four times greater than that obtained classically. Also, another two types of remanence plots related to the asymmetry of the magnetization reversal have been introduced and the method has been applied to polycrystalline Co/IrMn exchange-bias films.

The present work employs the remanence plots technique in order to estimate the interactions present in IrMn/NiCu bilayers and check if there exists a correlation between the interactions’ type and the $H_C$ variations caused by ion irradiation. Model simulations were also performed and used for the interpretation of the experiments.

Results

AF/FM bilayer with $T_N > T_C$ and composition Si(100)/Ni$_{60}$Cr$_{40}$(6 nm)/Ir$_{20}$Mn$_{80}$(7 nm)/Ni$_{75}$Cu$_{25}$(30 nm)/Cr(4 nm) has been grown on Si substrate. Pieces of the as-made films have been subjected to 40 keV irradiation with He$^+$ or Ge$^+$ ions at different fluences. $M(H)$, DCD and IRM curves were measured via an alternating gradient-force magnetometer with $H$ applied in the plane of the films. Henkel plots together with the corresponding $\delta M$ ones have been constructed. Aiming at a better understanding and interpretation of the experimental magnetization data, computational simulations based on the Landau–Lifshitz–Gilbert equation (LLG) were implemented.
Figure 1 – $H_C$, $\delta M_{\text{max}}$, $|\delta M_{\text{min}}|$ and $H(\delta M_{\text{min}})$ vs. the fluence of the He$^+$ irradiation or Ge$^+$ implantation (low-fluence region); the inset gives the full-range variations for the He$^+$ irradiated samples.

Both experimental and model results showed that the remanence plots technique allows outcomes of interactions inside the FM layer to be distinguished from those coming from changes of the exchange coupling at the FM/AF interface, and also that demagnetizing interaction effects could be achieved without the presence of dipolar interactions. The excellent agreement between experiment and model implies that the magnetic characteristics of our samples might be determined by exchange interactions only, and that the $H_C$ enhancement has been formally attributed to the rotatable anisotropy that accounts for interfacial spins which switch together with the FM magnetization.

Research outlook

Our findings show that systematic remnant magnetization experiments can be used to selectively obtain information on modifications caused, e.g., by ion irradiation in each layer of the film.

Publications

Self-Diffusion in HfO$_2$ Studied by Electron Spectroscopy


Introduction

Self-diffusion is an important but difficult to study phenomenon. Even for silicon, probably the best studied and understood material, the topic of self-diffusion remains an active field of research. There are few options for measuring self-diffusion, with most studies relying on the use of radioactive or low natural abundance isotopes. Analysis is then typically performed with secondary ion mass spectroscopy or other ion-beam based techniques. Oxygen diffusion in HfO$_2$ and HfSiO$_x$ alloys is of particular interest, as they are being used to replace SiO$_2$ as the gate dielectric in integrated circuits and are of interest for the fabrication of resistive random access memories based on resistive switching.

Results

Oxygen self-diffusion was investigated by monitoring the redistribution of $^{16}$O$_2$ and $^{18}$O$_2$ in a special sandwich structure [1]. The oxygen elastic peak for 40 keV electrons was resolved for different annealing temperatures. There was a systematic movement of the maximum of the peak to lower energy losses with increasing annealing temperature. This is due to $^{18}$O moving from the underlying Hf$^{18}$O$_2$ film towards the surface. The diffusion lengths are shown in Fig. 1. The dashed line is proportional to the expected diffusion length (for a constant anneal time). At first sight this appears to fit the experimental data reasonably well. However, at the lower temperatures, where conventional annealing experiments were also done, the diffusion length is found to increase much more slowly with annealing time than expected.
The measured RTA diffusion data were consistent with oxygen having an activation energy for self-diffusion near 1 eV at temperatures above 700°C. At lower temperatures the diffusivity decreases significantly with time, indicating that more than one mechanism is operative.

In the present context, it is interesting that the activation energy of 1 eV that seems to fit the higher temperature data, is consistent with values employed in models of diffusion in resistive random access memory structures and determined by theoretical means. While this is encouraging, it is clear that the current experiments go only part way to addressing diffusion in HfO$_2$ and a detailed understanding is still a difficult task.

**Research outlook**

The measured RTA diffusion data were consistent with oxygen having an activation energy for self-diffusion near 1 eV at temperatures above 700°C. At lower temperatures the diffusivity decreases significantly with time, indicating that more than one mechanism is operative.

**Publications**

Structural characterization of planar sets of NPs in a solid matrix


Introduction

The physical properties of metallic nanoparticle (NP) systems embedded in dielectric substrates depend not only on the NP parameters (location, size distribution and number concentration) but also on the quality of the dielectric matrix around the NPs. This applies for photonic devices and most particularly for the development of nonvolatile, high areal number density and low power memory devices. In the present contribution [1] we demonstrated that Medium Energy Ion Scattering (MEIS) measurements and Rutherford Backscattering Spectrometry (RBS), in combination with pertinent data from Transmission Electron Microscopy (TEM) and/or Grazing Incidence Small Angle X-ray Scattering (GISAXS) can indeed provide a rather complete and accurate characterization of buried NP systems. Our studies are performed considering a model case system consisting of planar arrangements of Au NPs containing three distinct Au concentrations embedded in a SiO$_2$ film.

Results

Au atoms were deposited onto 200 nm thick SiO$_2$ films, thermally grown from (001) Si wafers. The deposition was performed by RF magnetron sputtering, in a pure Ar plasma and with an RF power of 20 W, using an AJA magnetron sputtering device (ATC Orion-8 UHV from NANOLAB, Institute of Physics - UFRGS). A set of three samples containing different amounts of Au were obtained considering deposition times of 25, 50 and 100 s. Without breaking the vacuum, immediately after the Au deposition the samples were covered with a 35 nm thick layer of SiO$_2$, deposited using similar conditions but with an RF power of 90 W for 67. In this work we investigated three planar sets of buried Au NPs embedded into SiO2 matrix synthesized with different sputtering deposition times.
We observed that, for the samples prepared with 25 and 50 s, a significant fraction of the total amount of Au atoms becomes dissolved within the dielectric matrix around the NPs. Combining MEIS, RBS, TEM and GISAXS measurements, we were able to quantify the fraction of Au atoms effectively contained in the NPs, as well as the concentration and depth distribution of the complementary Au amount dispersed in the matrix around the NPs. These results open new perspectives for a more complete characterization of embedded metallic NPs systems into a solid matrix and the improvement NP bases devices.

Research outlook

In this work we demonstrate that Medium Energy Ion Scattering (MEIS) measurements in combination with Transmission Electron Microscopy (TEM) or Grazing Incidence Small Angle X-Ray Scattering (GISAXS) can provide a complete characterization of nanoparticle (NP) systems embedded into dielectric films.

Publications

Ne-He bubble formation in co-implanted Si(111) substrates

R.L. Maltez

Introduction

It was reported in a previous work [1,2] that formation of over-pressurized bubbles in a Si(111) substrate is able to improve the crystalline quality of GaN/AlN (AlN is a buffer layer) structures heteroepitaxially grown on it. The bubbles band in the substrate attracts the misfit dislocations generated at AlN/Si and GaN/Si interfaces and redirects them toward the substrate, thus reducing the dislocation density in the overgrown GaN layer. We are now investigating Si(111) samples containing Ne and Ne-He bubbles obtained from different implantation parameters and annealing temperatures (Ne bubbles are stable at high temperatures). Our final purpose is to find bubble systems able to keep comparable pressure to the He case even at higher temperature than the 650°C threshold of He. This requires understanding of which parameters play fundamental role on defining the morphology and pressure in such co-implanted system. Ne implantation was performed with the sample kept at 350°C to reduce implantation damage. These samples were submitted to RTA annealing for 2 minutes in the 400 to 1000°C temperature range. Characterization by Rutherford Backscattering Spectrometry/Channeling (RBS/C), Elastic Recoil Detection (ERD) and Transmission Electron Microscopy (TEM) are employed.

Results

We have found [3] that bubbles are always present in Ne-He hybrid system, even after annealing at 1000°C. However, while bubbles have increased in size as increasing the annealing temperature, the channeling in the samples has rather improved. It is an opposite behavior to the one expected if the dechanneling was a consequence of over-pressurized bubbles: the channeling degree in the samples should be worst during bubbles coarsening. In this sense, by RBS/C we do not observe clear correlation of dechanneling as a result of bubble formation for all investigated temperature range.
An special set (1x10^{15} \text{ cm}^{-2} \text{ Ne and 1x10^{16} cm}^{-2} \text{ He}), however, has showed slightly different thermal behavior, mainly for the annealing temperatures of 400 and 600°C: they show channeling spectra whose dechanneling in the implantation depth overcomes the as-implanted one. In addition, this specific co-implantation set and temperatures also demonstrated a peak shape for the dechanneling which resembles the one observed for the He pure case. However, we have observed a consistent correlation between channeling improvement and implantation damage reduction, suggesting that defects are the main cause of the observed dechanneling for the systems containing Ne. From TEM analysis it was also clear that bubbles in the hybrid system are all spherical ones and does not recall any similarity to the He system, even with Ne:He ratio of 1:10. Thus, from a morphologic point of view, the He and Ne hybrid system is very alike to Ne pure bubbles for the chosen implantation parameters.

Research outlook

Since residual implantation damage plays an important role on defining the pressure and morphology of such co-implanted system, the next step is aiming to an almost total damage annihilation during the implantation. Increasing of the sample implantation temperature and also interchange the implantation order of the Ne and He ions are the present status of the investigation.

Publications


Effect of ion irradiation on the thermal stability of thin polymer films

C.R.B. Esteves, R.S. Thomaz, L.I. Gutierres, R.M. Papaléo

Introduction

Polymer thin films are commonly used as protective or active layers in a large number of technological applications, from lithography to medical implants. One of the most common methods to prepare the films is spin-casting from diluted solutions. Thin polymeric films produced by spin-coating are, however, usually metastable systems due to the rapid decrease in mobility of the chains and freezing of molecular conformation during the fast evaporation of the solvent. Because of that, polymer molecules in thin layers are often in an unfavorable coil conformation, as compared to bulk conditions and may present structure evolution upon mild thermal treatments, leading to surface relaxation, roughening, or full dewetting of the layers. Detailed knowledge of the factors controlling stability of thin polymer coatings is thus of utmost importance for successful processing and reliable functioning of nanostructured devices based on polymers.

Results

In this work, we have investigated the stability of poly(methyl methacrylate) films, exposed to a low fluence irradiation, against annealing under different atmospheres (air, N₂ and vacuum). 300 keV H⁺, 2 MeV H⁺ and 18 MeV Au⁷⁺ ions were used to bombard the samples, using various fluences, depending on the ion species and energy. The films subjected to thermal treatments, without any irradiation showed an increased roughness; the effect being more pronounced for samples annealed under vacuum. The effect of the irradiation only on samples bombarded at room temperature was to slightly increase the surface roughness with increasing fluence, thus favoring the dewetting process. This was observed for both H⁺ and Au⁷⁺ irradiations.
The surface morphology of the PMMA films responded in a relatively complex way to the different conditions of ion bombardment. The effect of irradiation to a fixed moderate fluence on the stability of the film morphology upon subsequent annealing can be seen in the SFM images in Fig 1. Those images illustrate the evolution of the surface topography with time for the control and irradiated films (40nm thick). The surface of unirradiated films subjected only to the thermal treatment under vacuum (a-c), evolve slightly with time, tending asymptotically to a roughness value close to 0.45 nm. For samples pre-treated with H⁺ radiation (d-f), a smoothening effect was observed upon annealing, stabilizing the film at roughness levels below those of control samples (films not exposed to the beam but annealed under the same conditions). Irradiation with 18 MeV Au ions (g-i), on the contrary, destabilized the films, causing strong changes in surface morphology, and a roughness increase to levels close to ~2nm after 15h of annealing.

![Figure 1](image_url) - Stability of irradiated and control PMMA films of thickness h=40 nm upon thermal annealing at 100°C. (a-c) SFM images of control films not subjected to any bombardment, but which underwent the same thermal treatment as the bombarded samples. (d-f) SFM images of samples irradiated by $10^{14}$ ions/cm$^2$ of 2 MeV H⁺. (g-i) SFM images of samples irradiated by $10^{11}$ ions/cm$^2$ of 18 MeV Au$^{7+}$.

**Publication**

Publications in peer reviewed journals


Conference Proceedings


2) D. Puglia; G. Sombrio; R.M.S dos Reis; H.Boudinov, “Photoluminescence from Doped Silicon Nanocrystals in SiO2 matrix” In: SBMicro2013, 2013, Curitiba.


6) Radiation Effects on Insulators, Helsinki 2013 M. Behar et al.


8) OLIVEIRA, JOAO W. L.; CAUDURO, ANDRE L. F.; Baptista, Daniel L. ‘Growth of ZnO nanowires and microbeam lithography for field emission applications. 28th Symposium on Microelectronics Technology and Devices (SBMicro 2013).


18) S.A. Corrêa, C. Radtke, G. V. Soares, J. Han, S. Dimitrijev, F.C. Stedile, “Influence of the Pt electrode in the incorporation of D in dielectric films on SiC obtained by annealing in O2 and/or NO”, European Conference on Silicon Carbide and Related Materials - ECSCRM-2012, São Petersburgo, Rússia, setembro de 2012, We6-5.pdf.

Books and book chapters

1) Johnny Ferraz Dias, Lívio Amaral, Maria Lúcia Yoneama, *The 13th International Conference on Particle Induced X-ray Emission (PIXE 2013)*, Editor: Elsevier
ISSN: 0168-583X (2014).


Oral contributions and invited talks


12) Radiation Effects on Insulators, Helsinki 2013 M. Behar et al.


19) Oral presentation at REI, Helsinki 2013

20) Raul Carlos Fadanelli Filho: XXXVI Encontro Nacional de Física da Matéria Condensada, Águas de Lindóia, Brasil (maio 2013). Invited talk.


39) R. Giulian: Structural, electrical and optical properties of InSb and AlSb modified by ion irradiation. 19th International Conference on Ion Beam Modification of Materials (19th IBMM), Leuven, Belgium (2014). Oral presentation

Supervision of thesis and dissertations (completed)

1) Dario Ferreira Sanchez, "Caracterização do arranjo estrutural de sistemas enterrados de nanopartículas pela técnica de MEIS". 21/12/2012, Thesis, Supervisors Paulo Fernando Papaléo Fitchner and Pedro Luis Grande

2) Maurício de Albuquerque Sortica, "Caracterização estrutural de nanocristais compostos via espelhamento de íons de alta resolução", 20/05/2013 - Thesis, Supervisors : Pedro Luis Grande and Claudio Radtke


**Patent**

Organization of conferences

1) The 13th International Conference on Particle Induced X-ray Emission (PIXE 2013), Gramado, Brazil (2013). Chairs Johnny Ferraz Dias, Lívio Amaral and Maria Lúcia Yoneama.

2) VII Encontro Sul Americano de Colisões Inelásticas na Matéria (VII ESCIM), Gramado, Brazil (2014). Chairmen Pedro Luis Grande and Moni Behar

Members in international committees and in editorial boards of scientific journals

• Fernanda C. Stedile - Member of the international committee of the International Conference on Ion Beam Analysis (IBA)

• Pedro L. Grande - Member of the international committee of the International Conference on Ion Beam Analysis (IBA)

• Pedro L. Grande - Member of the international committee of the International Conference of Atomic Collision in Solids (ICACS)

• Pedro L. Grande - Member of the international committee of the International Workshop on High-Resolution Depth Profiling (HRDP)

• Pedro L. Grande - Member of the International Committee of Colisiones Inelasticas na Materia

• Pedro L. Grande - Editorial Board Nucl. Instr. and Methods B
• Fernanda C. Stedile - Editorial Board Nucl. Instrum. and Methods B

• Johnny Ferraz Dias - Member of the International Committee of the International Conference on Particle-Induced X-ray Emission

• Johnny Ferraz Dias - Member of the International Committee of the International Symposium on BioPIXE

• Moni Behar - Member of the International Committee of the Radiation Effects in Insulators.

• Moni Behar - Member of the International Committee of Ion Beam Modification of the Materials

• Moni Behar - Member of International Committee of Radiation Effects on the Materials.

• Moni Behar - Member of the International Committee of Colisiones Inelasticas na Materia.

- Moni Behar, Member of the International Committee of Radiation Effects on the matter

• Ricardo Papaléo - Member of the International Committee International Symposium on Swift Heavy Ions in Matter

• Gustavo Azevedo - Member of the Executive Committee of the IXAS (International XAFS Society)

• Paulo F. P. Fichtner - Member of the International Committee of the Ion Beam Modification of Materials Conference.
Partners (Universities, Research Institutes and Companies)

- CAPES/MES, Cuba 2010/2012
- CAPES/MES, Cuba 2012/2014
- CAPES/COFECUB, França 2012/2014
- Capes/Myncyt, Argentina, 2010-2012
- CAPES/FCT, Portugal, 2014/2015

École Polytechnique de Nancy, CAPES – Brafitec. Dupla diplomação em nível de graduação e pós-graduação em engenharia e ciência dos materiais.

Pontifícia Universidade Católica - RJ, INCT e PROCAD.

UFSC and UFPR. Área de corrosão eletroquímica de aços revestidos e/ou tratados termicamente.
Projects

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