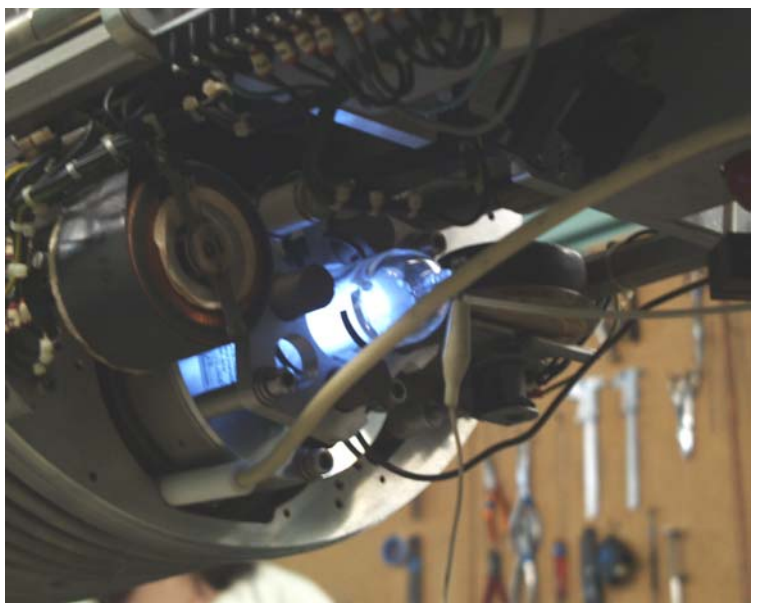


Physics Sector



Physics*

Eduardo Alves

The Physics Department focused its activities on Fundamental and Applied Research as well as Education and Training. The groups settled in the sector have a long tradition of combining High level Education (MSc, PhD, etc) with their own research projects making the area a living space. Despite the difficulties there is also some applied research using the nuclear technologies to solve industrial problems. Most of this work relies on the existence of a few specialized facilities unique in Portugal:

1-The Ion-beam Laboratory has a 2.5 MV Van de Graaff Accelerator (able to run at 3.1 MV pushed to its limits) associated with an ion microprobe and a 210 keV high fluence ion implantor. This infrastructure is open to external users mainly from the University but also from other research Institutions and Industry. The engagement on training and education of students and young researchers, originated a strong collaboration between ITN and the Nuclear Physics Centre of the University of Lisbon (CFNUL).

The infrastructure is used in the fields of material sciences, environmental health, atomic and nuclear physics (cross section measurements). This last subject is carried out by a research group, **Nuclear Reactions Group**, formed with staff from the Universities of Lisbon. The activity developed in the Ion Beam Laboratory is presented in the following pages under the headings *Nuclear Solid State Physics using ion beams, Nuclear Reactions and Health and Environmental studies using ion beams*.

2- The High Temperature Materials Laboratory, Ma³t completed its third year of activity. The research activities were carried out in two fronts: the characterisation of materials and the continuation of the development of the Hotbird capabilities through the implementation of new hardware components and software applications. Presently three main geometries are implemented: the *high-intensity mode*, the *double-crystal diffractometer* and the *triple-axis* geometries.

The current research projects are centred on the study of advanced materials used in the electronic industry as well as high-temperature alloys used in the aeronautical industry. Many of these studies are performed in collaboration with foreign research groups that request beam time at the Hotbird owing to its high specificity and enhanced capability to solve difficult problems. Two new projects have been approved this year to study (1) nanometer-

thick buried layers in SiGe devices and to characterise (2) magnetic tunnel junctions used for hard-disk reading heads. This will allow to reinforce the team with two new students for the next two years.

3-The Neutron Spectrometers are installed at the ITN nuclear research reactor RPI. Commissioning of the basis configuration of the 2-axis Diffractometer DIDE equipped with a “banana” multidetector took place in 2002. A Small Angle Neutron Scattering Instrument, EPA, is currently under installation. ETV, a TOF Diffractometer for educational purposes is operational. Current research and development work focused on the structural characterization of new materials and instrument optimisation, is carried out in collaboration with national partners and foreign groups notably in Aveiro, Saclay, Budapest and Sofia.

Activities pertaining to sections 2. and 3. are presented under **Condensed Matter Physics**.

4-The Gamma Radiation facility (UTR) is a Cobalt-60 facility with a semi-industrial dimension constructed in collaboration with the International Atomic Energy Agency (IAEA), as a demonstration unit for the national industry.

For logistic reasons the unit is under the Physics Department coordination. Its operation started in Nov.1988 with an activity of approximately 300kCi. Today the irradiations are carried out with an activity about 56kCi.

The main activity developed around this infrastructure is concerned with the radio sterilisation of medical devices and pharmaceuticals and the decontamination of other products. A new field is presently being developed to apply ionising radiation techniques to waste water treatment. The research carried out is presented under the title **Radiation Technologies Processes and Products**.

5- Other activities. The activity of the **Nuclear Instruments and Method Group** is focussed in *Computational Physics* (calculation of radiation fields and modelling of cold plasmas for material processing applications) and *Instrumentation and Technical Assistance* (development of equipment for internal groups, fabrication of equipment for specific applications and assistance to industrial companies and scientific institutions as well as technical consulting).

* Dr^a M.F. da Silva was the responsible for the Physics Department until March 2002 when she retired.

Structure of the Sector and Technical staff

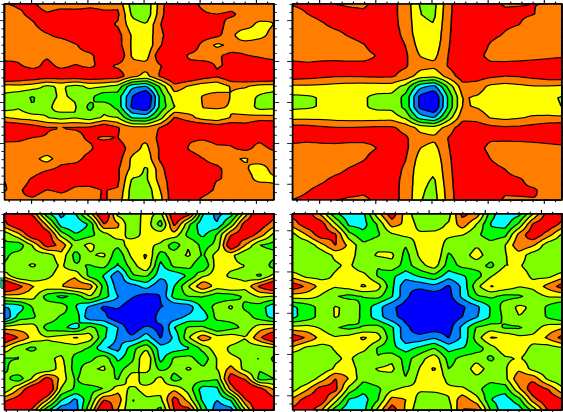
Research groups in the Physics Sector

- Nuclear Solid State Physics using Ion Beams
- Condensed Matter Physics
- Nuclear Reactions
- Health and Environmental Studies Using Ion Beams
- Nuclear Instruments and Methods
- Radiation Technologies: Processes and Products

Administrative and Technical staff

- ANA FARIA
- DIAMANTINA VENÂNCIO
- JOSÉ VIEIRA HENRIQUES
- MANUEL CABAÇA
- MARIA LUISA OLIVEIRA
- MARIA TERESA PIRES

Nuclear Solid State Physics using Ion Beams



Nuclear Solid State Using Ion Beams*

Eduardo Alves

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

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

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

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

The work in insulators is a continuation of ongoing projects or bilateral collaborations to modify the optical and electrical properties of α -**Al₂O₃** and **LiNbO₃**. Recently a bilateral project was started with

the University Carlos III in Spain to study the doping of MgO with transition metals.

Besides this, research has also been continued on the **synthesis of new compounds, namely of metastable intermetallic compounds in Al, Ti and Mg based alloys** using ion implantation. It must be also pointed out that due to the potential of ion beam techniques to study thin films and multilayers important work has been continued in the characterisation of **magnetic thin films for magnetic spin valves and tunnel junctions**.

The activity in the technology programme of the **European Fusion Development Agreement** in association with the Centro de Fusão Nuclear of the Instituto Superior Técnico was pursued as a strategic effort to include ITN in the European Fusion activities. Deliverables in several tasks were obtained, namely the study of compatibility of SiC/SiC_f with other structural materials, surface studies of enriched 6Li ceramic breeders (Li₄SiO₄ and Li₂TiO₃) and Be pebble bed resistivity under ionising radiation.

Finally, the group has also a participation in the **ISOLDE/CERN** using the Emission Channeling technique, applied to studies of lattice site location of transition metals and rare earths in semiconductors. There Perturbed Angular Correlations are used to characterize the charge carrier blocks and charge ordering in High-Tc superconductors and on colossal magnetoresistive oxides. Actually our projects further afford the development of new fast 2D- Si electron detectors to extend the use of EC to probe elements only available by using short-lived isotopes (like ²⁷Mg). These new detectors are the basis of a new concept on high efficient PET technologies to be submitted on 2003 to the EU 6th framework program of research, in collaboration with CERN. Recently, we have initiated a program of cooperation with high-tech industry in materials characterization for new optoelectronic applications.

Besides these research activities the group as also strongly engaged in training graduate and undergraduate students, through the supervision and work of M.Sc. and Ph.D. students, Final year degree thesis and Socrates Students as well. In 2002, 47 papers were published in international Journals and 29 accepted for publication. The main achievements of the research developed in 2002 are summarised in the following pages.

* During 2002 Dr^a Maria Fernanda da Silva the leader of ITN group retired and a new direction was appointed for ITN. As a consequence a reorganization of the Ion Beam Laboratory is underway but the Members of the group decided to maintain the designations and structure on the 2002 report.

Nuclear Solid State Using Ion Beams

Research Team

Researchers^(*)

- E. ALVES, Auxiliary researcher
- R.C. da SILVA, Auxiliary researcher
- J.G. CORREIA, ITN Contract
- U. WAHL, FCT Post-Doctoral
- K. LORENTZ, European Post-Doctoral fellowship
- M.F. da Silva² Principal researcher
- L.C. ALVES, Research assistant (75%)¹
- N. BARRADAS, Auxiliary researcher, Reactor (10%)
- A.R. RAMOS Auxiliary researcher, Reactor (10%)
- A. KLING, Auxiliary researcher, Reactor (5%)

- JOANA V. PINTO, Ph.D. student, FCT grant
- C.P. MARQUES, M.Sc. student, ITN grant
- E. RITA, Ph.D. student, Univ. Lisboa, FCT grant
- V. MATIAS, Last Year B.Sc. Student,
- A.C. MARQUES, Last Year B.Sc. Student, PRAXIS grant

Technical Personnel

- J. ROCHA
- F. BAPTISTA
- P. MARQUES

Collaborators

- J.C. SOARES, Full Professor (CFNUL)
- M.R. DA SILVA Auxiliary Professor (CFNUL)
- A.A. MELO (Scientific Adviser)

Students

- L. PRUDÊNCIO, Ph.D. student, FCT grant

^(*) Also members of CFNUL.

¹ Doing Ph.D.

² Retired in March 2002

Funding (€)

Research Projects:	253515
Outros:	4995
Total:	258510

Publications

Books:	1
Journals:	49 and 21 in press
Proceedings:	4 and 1 in press
Conf. Communications:	31
Other publications:	1
Theses: Ph.D.	1
M.Sc.	2
B.Sc.	1

Thin Films and Quantum Wells

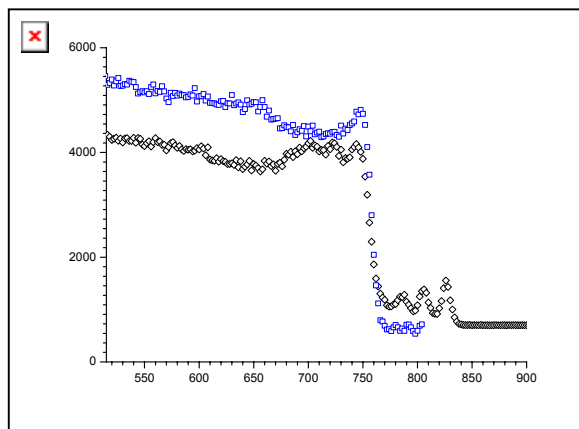
E. Alves, N. Barradas¹, A.D. Sequeira², A. Gonçalves³, S. Pereira⁴, R. Correia⁴, L. Rebouta⁵

Objectives

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

Results

The work carried out in this field was concentrated mainly in two systems: Semiconducting films [1-5] and metallic coatings [6 - 9]. The research work in Semiconductors was focused in the GaN/InGaN system. The performance of InGaN-based devices is responsible for the intensive research developed during the last decade with these materials. Our purpose was to study and correlate the composition gradient and strain with the optical properties of the InGaN films. The capabilities of ion beam techniques to provide information on these issues is illustrate in the figure.



The figure shows two random spectra of an InGaN/GaN multilayer obtained with large tilt angles, allowing the determination of composition and interface roughness.

The work carried out on metallic films aimed at study the composition, thickness and roughness of the interfaces.

Published, accepted or in press work

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2. R. Correia, S. Pereira, A. Cavaco, E. Pereira, E. Alves, Preliminary investigations of infrared Er-related photoluminescence in ion-implanted $\text{In}_{0.07}\text{Ga}_{0.93}\text{N}$, *Appl. Phys. Lett.* **80** (2002) 4504.
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6. and photoluminescence peaks in InGaN/GaN layers, *Mat. Sci. and Eng.* **B 93** (2002) 163-167.
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¹ Nuclear reactor group, ITN

² Condensed Matter Physics group, ITN

³ Condensed Matter Physics group, ITN

⁴ Dep. de Física, Universidade de Aveiro 3810-193 Aveiro, Portugal.

⁵ Universidade do Minho, Azurém 4800-058, Guimarães Portugal

Doping of wide band-gap semiconductors with ion beams

E. Alves, K. Lorenz, E. Rita, U. Wahl, W. Jiang¹, W. J. Weber¹, E. Wendler², T. Monteiro³

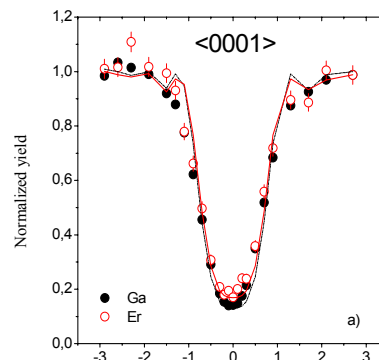
Objectives

The main goal of the work carried out at the ion beam laboratory of ITN in this field was the doping of GaN and ZnO with optical and electrical active ions. This research was done in collaboration with other institutions where the optical studies were performed. The implantations were done at ITN and the group studied the damage recovery and lattice site location of the dopants.

Results

The work performed during this year in this area was concentrated on the implantation of Rare Earth ions in GaN and ZnO [1-10]. We studied the influence of implantation parameters (temperature and dose) on the damage production and determined the best annealing conditions. Simultaneously we follow the lattice site location of the implanted ions. The capabilities of ion beam techniques to provide information on these issues are illustrate in the figure where the angular scan along the $\langle 0001 \rangle$ axis shows the complete

overlap of Er and Ga curves indicating that Er is



Angular scan along the $\langle 0001 \rangle$ axis revealing the complete overlap of Ga and Er curves.

substitutional along this direction when implanted in GaN. Similar results found along the $\langle 10\bar{1}1 \rangle$ axis clearly show that Er occupies mainly the Ga site in the lattice.

Published, accepted or in press work

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2. E. Alves, N.P. Barradas, T. Monteiro, R.Correia, U. Kreissig, Ion Beam Studies of MBE grown GaN films on (111)silicon substrates, *Nucl. Instr. and Meth. B*188 (2002) 73-77.
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5. C. Boemare, T. Monteiro, M.J. Soares, J.G. Guilherme, E. Alves, Photoluminescence studies in ZnO samples, *Phis. Stat. Sol. 308-310* (2002) 985-988.
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7. W. Jiang, W.J. Weber, C. Wang, E. Alves, Defect properties of ion implanted Gallium Nitride, *Nucl.Instr.and Meth. B* .
8. E. Wendler, A. Kamarou, E. Alves, K. Gärtner, W. Wesch, Three-step amorphisation process in ion-implanted GaN at 15 K, *Nucl.Instr. Meth. B*.
9. E. Alves, R. Correia, S. Pereira, U. Wahl, B. De Vries, A. Vantomme, Annealing behavior and lattice site location of Er implanted InGaN, *Nucl. Instr. and Meth. B*.
10. E. Alves, E. Rita, J.G. Correia, U. Wahl, T. Monteiro, J. Soares, C. Boemare, Lattice site location and optical activity of Er implanted ZnO, *Nucl. Instr. Meth B*.

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² Pacific Northwest National Laboratory, United States

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Buried Nanoprecipitates in Ceramic Oxides

C. Marques, J.V. Pinto, T. Monteiro¹, B. Fernandes², M.M Cruz³, A.D. Sequeira⁴,
R.C. da Silva, E. Alves

Objectives

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

Our work concentrates on the ion beam synthesis of metallic and metal like nanoprecipitates buried in the crystalline oxides Al_2O_3 and MgO , and on the study and characterization of its structural and electromagnetic properties and stability.

Results

1. Ion beam synthesis of metallic nanoprecipitates in transparent ceramic oxides:

1.1. In sapphire (Al_2O_3)

High dose implantations of Fe, Ti, and Co ions were used to form buried layers of metallic (Fe and Co) or intermetallic nanoprecipitates (Al_5Ti_2 in the case of Ti). Combining XTEM with ion beam methods it was possible to show that, depending on the implanted dose, the nanoprecipitates may form continuous films which display electrical conductivity of the metallic type. XRD measurements showed that in all instances the precipitates are metallic and crystalline, and tend to align parallel to the sapphire surface. The Fe and Co nanoprecipitates display ferromagnetic behaviour.

RBS-C showed that the implanted layer is left in a highly disordered state which recovery depends strongly on the annealing atmosphere and much less strongly on the type of crystalline surface.

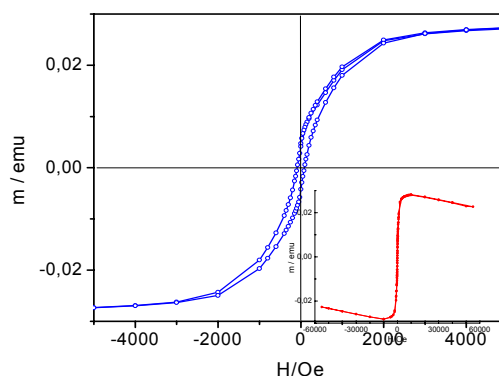
In general annealing in vacuum produces a thin buried metal rich layer while annealing at the same temperatures in air leads to the formation of mixed

oxides. This is associated with a change of colour of the sapphire that turns blue-green (with Co), yellow (with Fe) or becomes transparent, in the case of Ti implantations.

1.2. In crystalline MgO

High dose implantations of Li, Co, Ni and Cu ions were carried out in MgO single crystals to promote the formation of metallic precipitates, after a suitable thermal treatment is performed. Ni doped MgO that has undergone TCR treatment was also studied for comparison.

RBS-C and optical absorption measurements in Li implanted MgO show that the implantation damage is heavy with a large fraction of defects identified as anion vacancies. Analysis of the evolution of the extinction bands observed after thermal annealings indicate the formation and growth of Li colloids which dissolve at 1250 K. The MgO crystals become transparent.



Metallic nanoprecipitates formed in Ni doped MgO single crystals following thermochemical reduction at 1550 K exhibit ferromagnetic behaviour (a small diamagnetic component due to the MgO host is also observed).

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3. C. Marques, M.M. Cruz, R.C. Da Silva, E. Alves, Optical changes induced by high fluence implantation of Co ions on sapphire, *Surf. and Coatings Technology* 158-159C (2002) 54-58.
4. E. Alves, C. Marques, R.C. Da Silva, T. Monteiro, J. Soares, C. Mchargue, L.C. Onone, L.F. Allard, Structural and optical studies of Co and Ti implanted sapphire, *Nucl. Instr. and Meth. B*.
5. E. Alves, R.C. da Silva, B. Savoini, D. Cáceres, I. Vergara, R. González, Y. Chen, Radiation-damage recovery in undoped MgO crystals implanted with lithium ions, *accepted Nucl. Instr. and Meth. B*.

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Synthesis of New Intermetallic Compounds by Ion Implantation

L.M. Prudêncio^{1,2}, I. Nogueira⁴, J.C. Waerenborgh³, A.D. Sequeira², R.C. da Silva²

Objectives

Ion beams are used to modify the surface region of technologically important light metals, without altering its bulk properties. In particular intermetallic phases can form embedded in the light metals matrix. These surface layers can act as a protection against corrosion and/or improve wear resistance.

Our work concentrates on the direct ion beam synthesis of intermetallic precipitates embedded in the surface region of Al and Ti, and on the study and characterization of its structural properties and stability.

Results

Ion beam synthesis of intermetallic precipitates in light metals

1. In Aluminium

Cr, Fe, Ti and Cu implantations

High purity polycrystalline Al samples were implanted with Cr, Fe, Ti and Cu, with fluences in the range $1\text{-}5 \times 10^{17}$ /cm², at different substrate temperatures.

1.1. Al-Cr system: three different types of surface layers were achieved directly by Cr ion implantation in Al at different temperatures. Cr(Al) solid solution at RT, Al₈₆Cr₁₄ at 270 °C and Al₁₃Cr₂ at 500 °C (*Surface and Coatings Technology 128-129 (2000) 166-9*). Samples prepared with these three different systems are now being examined by TEM.

1.2. Al-Fe system: high fluence Fe implantations have been performed at different temperatures. RBS and CEMS results show that implantations at RT produce Al₈₆Fe₁₄, while at 200 °C and 400 °C lead to the formation of Al₅Fe₂. Thermal annealings at 630 °C transform Al₅Fe₂ into Al₁₃Fe₄ (*Surface and Coatings Technology 158-159 (2002) 339-42*). Recent measurements indicate that Al₈₆Fe₁₄ transforms into Al₆Fe by annealing at 220 °C.

1.3. Al-Ti system: RBS, GIXRD and SEM analyses showed that AlTi₃ precipitates form directly by high fluence Ti ion implantation at RT, while when implantations are carried out at higher temperatures, 270 °C and 500 °C, no intermetallics are formed.

It was also found that Al₃Ti forms by annealing the implanted samples at 500 °C, irrespective of the implantation temperature.

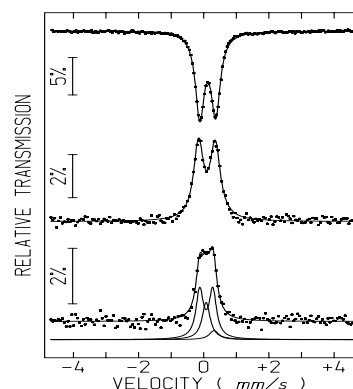
1.4. Al-Cu system: RBS and GIXRD analyses show that no Al-Cu intermetallics form by high fluence Cu ion implantation at RT. However, precipitates of Al₂Cu appear after annealing at 300 °C. Further annealings at 400 °C and 500 °C result in extensive diffusion of Cu into the Al matrix and the complete dissolution of the Cu-Al intermetallic precipitates.

2. In Titanium

Cr implantations

Cr implantations were performed in two different Ti systems, polycrystalline Ti discs and Ti single-crystal grown by the Czochralski method at ITN, with fluences in the range $5 \times 10^{16}\text{-}10^{18}$ /cm².

2.1. Polycrystalline Ti-Cr system: no evidence was



found for intermetallic compounds forming by ion implantation at any of the implantation temperatures -180 °C, -110 °C, RT, 220 °C, 300 °C, 800 °C and 900 °C, nor even after different isothermal and isochronal annealings. Very fast Cr transport was found to occur at temperatures above 800 °C.

2.2. Single-crystal Ti-Cr system: 5×10^{16} /cm² Cr ion implantation destroys the crystalline quality of the single-crystal surface. Recovery by thermal annealing treatments have been tried with only partial success.

Published, accepted or in press work

1. L.M. Prudêncio, I.D. Nogueira, J.C. Waerenborgh, A.P. Gonçalves, O. Conde, R.C. Da Silva,

¹ Faculdade de Ciências e Tecnologia da Universidade Nova de Lisboa.

² Departamento de Física, Instituto Tecnológico e Nuclear.

³ Departamento de Química, Instituto Tecnológico e Nuclear.

⁴ Instituto Superior Técnico, Universidade Técnica de Lisboa.

Formation of Al-Fe surface alloys by ion implantation of Fe in Al, *Surface and Coatings Technology 158-159 (2002) 339-342*.

Study of Advanced Materials for Fusion Reactors using the Nuclear Microprobe

L.C.Alves^{1,2}, E. Alves^{1,2}, A. Paúl³, M.R. da Silva², B. Riccardi⁴, A. La Barbera⁵, G. Piazza⁶, J.D. Lulewicz⁷, M.F. da Silva^{1,2}, J.C.Soares^{1,2}

Objectives

Under the framework of the Nuclear Fusion Programme supported by the EURATOM, the nuclear microprobe installed at the Ion Beam Laboratory of ITN has been applied in the characterisation of structural and breeder blanket fusion reactor materials for determining impurity contents, their spatial distribution and chemical compatibility between several materials under relevant fusion reactor working conditions.

Results

Elemental characterisation was performed on Be pebbles, Li ceramic pebbles, SiC composites and several batches of a martensitic steel known as Eurofer. Chemical compatibility studies between Be pebbles and SiC composites and between Li ceramic pebbles and Eurofer were also performed [1,2].

The elemental analysis of the Be pebbles revealed, from the point of view of the production of long lived isotopes, the undesired presence of U in concentrations around 100 µg/g.

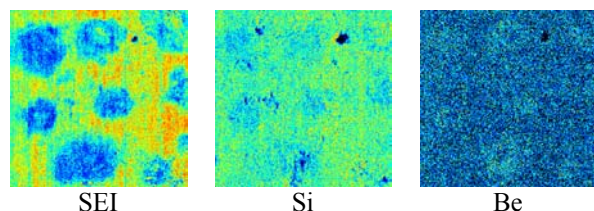


Fig. 1 – Nuclear microprobe images from an uncoated SiC sample after the experiment at high temperatures. The images were obtained from a 2640×2640 µm² scan, and the secondary electron image (SEI) clearly reveals the spots where the Be pebbles were in contact with the SiC material. Also shown are the Si and Be elemental maps.

Published, accepted or in press work

1. A. Paúl, L.C. Alves, E. Alves, B. Riccardi, Structural characterization of SiC/SiC_f composites exposed to chemical interaction with Be at high temperature, *J. Nucl. Materials*.

Two different kinds of SiC/SiC_f composite were manufactured at ENEA, Italy, one of them presenting an extra SiC coating obtained by CVD technique. Several samples of both materials were placed inside a Be pebble bed and the whole set-up annealed at 800°C for 550 h in a reducing atmosphere. For the uncoated samples, surface oxidation is accompanied by a strong C depletion and a Be diffusion. The coated samples presented, as general behaviour, an increase in the number and extension of the cracks already observed at the surface of the coated virgin samples, what highly compromises their use in fusion reactors.

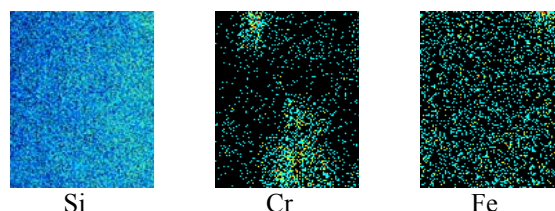


Fig. 2 – X-ray elemental maps from a 53*53 µm² scan from an orthosilicate Li pebble after exposure to Eurofer sample.

Li ceramic breeders, both orthosilicates and titanates, were characterised after chemical compatibility tests with Eurofer. The whole set-up was annealed in fusion reactor conditions for exposure periods that ranged from 20h to 2000h. The analysis shows that the Fe and Cr contents of the pebbles changes in time, indicating diffusion from the Eurofer sample. The presence of independent clusters of Cr and Fe also indicates the formation of different chemical compounds. The reaction is more noteworthy for the orthosilicate Li pebbles.

2. L.C. Alves, A. Paúl, M.R. Da Silva, E. Alves, B. Riccardi, J.C. Soares, High Temperature Chemical Compatibility between SiC Composites and Be Pebbles, *accepted Nucl. Instr. and Meth. B*.

¹ ITN, Physics, E.N. 10, 2686-953 Sacavém, Portugal.

² CFNUL, Av. Prof. Gama Pinto 2, 1699 Lisboa, Portugal.

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⁵ Association EURATOM-ENEA, CR-Casaccia, Rome, Italy

⁶ FZK, Forschungszentrum Karlsruhe, Germany.

⁷ CEA, Saclay, France.

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

J.G. Correia¹, E. Alves¹, V. Amaral², J.P. Araújo³, A.C. Marques¹, E. Rita¹, U. Wahl¹, A.M. Lopes², J.C. Soares¹ and the ISOLDE collaboration⁴

Objectives

The radioactive ion beam laboratory infrastructure of ITN/CFNUL at ISOLDE/CERN provides complementary analysis techniques to the ion beam techniques at ITN-Sacavém with respect to characterizing the microscopic behavior of elements into materials. The scientific work is actually centered in three research subjects approved by the ISOLDE Scientific Committee (INTC) from 11 / 2000 to 02 / 2001: a) IS368 "Lattice Location of Transition Metals in Semiconductors, b) IS360 "Studies of High-Tc Superconductors doped with radioactive isotopes" and c) IS390 "Studies of colossal magnetoresistance oxides with radioactive isotopes". At ISOLDE we have introduced and developed the e^- - γ Perturbed Angular Correlations (e^- - γ PAC) and, more recently, the Emission Channeling Techniques (EC), working with new 2-Dim. electron detectors. Our projects further afford the development of new fast 2D- Si electron detectors, in collaboration with CERN, to extend the use of EC to short lived elements. These detectors are the basis of a new concept on high efficient PET technologies, to be submitted in 2003 to the EU 6th framework program of research. Recently our group at CERN ISOLDE initiated a program of cooperation with high-tech industry in material characterization for new optoelectronic applications.

Results

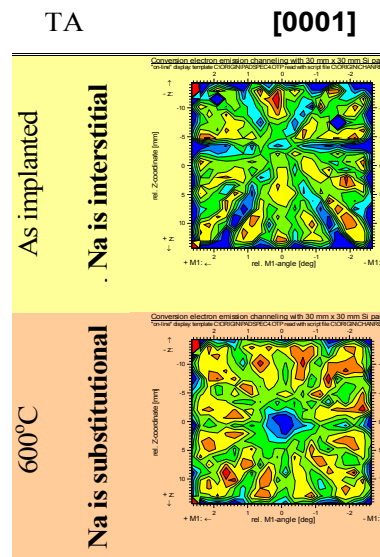
Studies of High-Tc Superconductors Doped with Radioactive Isotopes (IS360).

Experiments undergoing on $\text{HgBa}_2\text{CuO}_{4+\delta}$ show that, high concentrations of dopant fluorine atoms order along [100] or [010] rows or in the centre of the Hg channel. This behaviour consequence, regarding the efficiency of charge carrier injection to the Cu planes, is under study with the help of first-principle calculations of the charge density [to be published].

Lattice Location of Transition Metals in Semiconductors (IS368).

Published, accepted or in press work

1. J.P. Araujo, U. Wahl, E. Alves, J.G. Correia, T. Monteiro, J. Soares and the ISOLDE Collaboration, Erbium implantation in Strontium Titanate, *Nuclear Instr. and Methods B* 191 (2002) 317-322.
2. U. Wahl, J.G. Correia, A. Vantomme, and the ISOLDE Collaboration, Lattice location of



The figure shows 2D- electron anisotropy yields (red < intensity < blue), by detecting β^- emitted from the decay of ^{24}Na implanted ZnO semiconductor single crystal. The two measurements were taken along the [0001] axis, showing also the principal planes which are collinear with this direction. The electron emission patterns show that sodium changes from interstitial (as implanted) to substitutional lattice sites by annealing at 600°C.

A large program of research has progressed in AlN, GaN and ZnO, where the lattice sites of several elements, Fe, Cu, Na, Ga, Hg and Au have been measured with the EC technique. Two of the most interesting cases are As and Na which show the occupation of multiple lattice sites as a function of temperature. Above we show an illustrative example of experiments done with ^{24}Na -implanted ZnO, which have been measured using the new cooled EC detector our group has implemented in 2002.

We further report on first experiments looking forward to determine the lattice site location of Cu in KLiTaNbO (KTLN), within the concept of the contract with industry Trellis Photonics.

implanted Ag in Si, *Nuclear Instr. and Meth. B* 190 (2002) 543-546.

3. U. Wahl, E. Rita, E. Alves, J.G. Correia, J.P. Araujo, and the ISOLDE collaboration, Implantation site of rare earths in single-crystalline ZnO, *accepted by Applied Physics Letters*.
4. K. Bharuth-Ram, U. Wahl, and J.G. Correia, Lattice location of Fe in diamond, *accepted by Nuclear Instr. and Meth. B*.

¹ Condensed Matter Physics group, ITN;

² Dep. de Física, Universidade de Aveiro 3810-193 Aveiro, Portugal ;

³ Dep. de Física, Universidade do Porto, 4150 Porto, Portugal;

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Magnetic Tunnel Junctions

N.P. Barradas, S. Cardoso¹, P.P. Freitas¹, U. Kreissig², A.R. Ramos, M.F. da Silva, J.C. Soares, J. Wang¹, P. Wei, Z.G. Zhang¹

Objectives

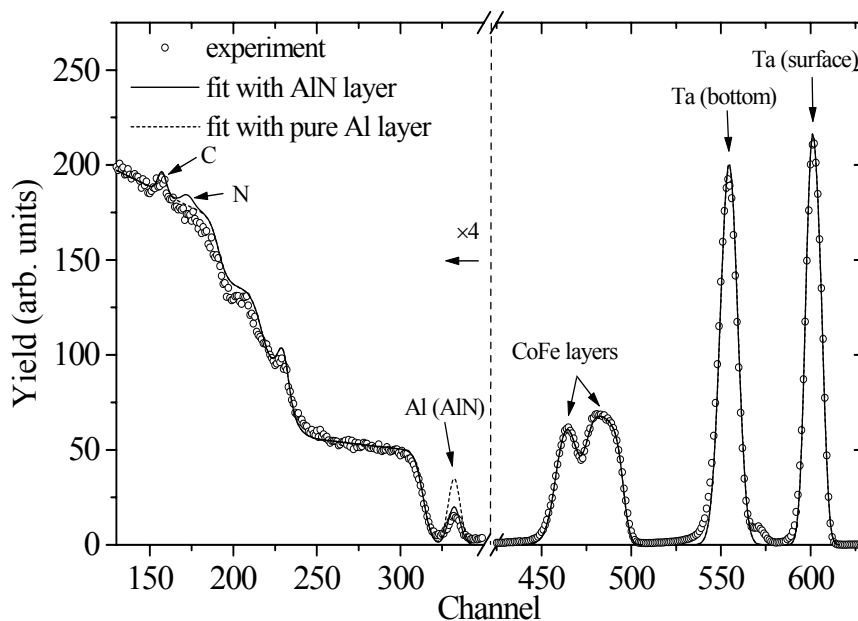
ITN has a long-standing collaboration with the INESC magnetic systems group led by Prof. Paulo de Freitas. The role of ITN is to provide structural characterisation of the highly complex advanced magnetic systems produced at INESC. This is a highly interactive collaboration that has proved to be very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

Results

This year the work was concentrated in the compositional characterisation of spin tunnel junctions [1-2]. These are made by two thin magnetic layers

separated by a thin insulating one. The tunnelling current across this barrier layer depends on the relative alignment of the magnetisations of the two magnetic layers. Hence such a device can be used either as a reading head (in which the external field - e.g. from a hard disk - leads to a change in the resistance of the system), or as a non-volatile memory (where the external field forces the direction of the magnetisation).

The highlights were to determine the composition of the AlN_xO_y barrier in $\text{Si}/\text{Al}_2\text{O}_3$ 750Å/Ta 40Å/CoFe 30Å/ AlN_xO_y 10Å/CoFe 40Å/Ta30Å tunnel junctions.



RBS spectrum of a $[\text{Si}/\text{Al}_2\text{O}_3]_{\text{substrate}}/\text{Ta}/\text{CoFe}/\text{AlN}_x/\text{CoFe}/\text{Ta}$ tunnel junction.

Published, accepted or in press work

1. Z.G. Zhang, P.P. Freitas, A.R. Ramos, N.P. Barradas, J.C. Soares, Effect of natural oxidation conditions on low resistance spin tunnel junctions *J. Appl. Phys.* 91 (2002) 8786-8788.
2. P. Wei, N.P. Barradas, J.C. Soares, M.F. Da Silva, U. Kreissig, S. Cardoso, P.P. Freitas, Composition analysis of the insulating barrier in magnetic tunnel junctions by high resolution RBS; *Nucl. Inst. and Meth. B* 190 (2002) 684-688.

¹ INESC, R.Alves Redol 9-1,1000 Lisboa, Portugal

² FZR, Postfach 510119, 01314 Dresden, Alemanha

Artificial Neural Networks Analysis of Ion Beam Analysis Data

N.P. Barradas, A. Vieira¹, R.N. Patrício, E. Alves

Objectives

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

Results

We have previously developed a code, the IBA DataFurnace, that uses the simulated annealing

algorithm to automatically analyse IBA data. We have continued a new line of work that we recently opened, namely using artificial neural networks (ANNs). We have developed ANNs both to analyse data as such, extracting the relevant desired parameters in an instantaneous way [1,2], and in a code suitable for automated control of an experimental setup, given an interface to the relevant hardware [3,4].

Published, accepted or in press work

1. A. Vieira, N.P. Barradas, E. Alves, Analysis of sapphire implanted with different elements using artificial neural networks; *Nucl. Inst. and Methods B190* (2002) 241-246.
2. A. Vieira, N.P. Barradas, A training algorithm for classification of high dimensional data; accepted by *Neurocomputing*.
3. N.P. Barradas, R. Patrício, A. Vieira, RBS without humans; *Nucl. Inst. and Methods B190* (2002) 231-236.
4. N.P. Barradas, A. Vieira, R. Patrício, Artificial neural network algorithm for automation of Rutherford backscattering spectroscopy experiments and data analysis; *Phys. Rev. E* 65 (2002) Article 066703.

Light Impurities Influence on the Ion Beam Synthesis of Silicides

A.R. Ramos, F. Pászti¹, N.P. Barradas, M.F. da Silva, J.C. Soares

Objectives

Silicides have wide use and applications in microelectronics. The formation of several interesting silicides – Er and Hf silicide, for instance- may be impaired by the presence of low concentrations of light impurities in silicon, such as oxygen and carbon. Ion implantation has been shown to promote silicide formation in oxygen and carbon contaminated silicon substrates, such as porous silicon [1]. In the present case, we have studied the behaviour of oxygen atoms during the Ion Beam Synthesis of HfSi₂.

Results

We have studied the oxygen distribution behaviour during the IBS of HfSi₂ by HIERD (Heavy Ion Elastic Recoil Detection) and oxygen Resonant Scattering. Czochralski and Float Zone silicon substrates were analysed. The low oxygen concentration results in low statistics, shadowing results. Therefore, HfSi₂ layers were prepared on oxygen pre-implanted samples and the oxygen redistribution resulting from Hf implantation studied. Results show no oxygen incorporation in the Hf implanted layer: Ion Beam Synthesis favours silicide formation even in the presence of O [2].

Published, accepted or in press work

1. A.R. Ramos, F. Pászti, E. Kotai, É. Vázsonyi, O. Conde, M.R. da Silva, M.F. da Silva, J.C. Soares, Synthesis of Cobalt Silicide on Porous Silicon by High Dose Ion Implantation, *Nucl. Instr. and Meth.*, B178 (2001) 283.
2. A.R. Ramos, F. Pászti, N. P. Barradas, U. Kreissig, M.F. da Silva, J.C. Soares, Oxygen Influence on

the Ion Beam Synthesis of Hafnium Silicides: HIERD and Resonant Scattering Analysis, 12th General Conference of the European Physical Society, EPS-12 Trends in Physics, Europhysics Conference Abstracts, Ed. G. Battistig, Cs. Hadjdu, D.L. Nagy, e. Szilágyi, vol. 26G, 2002 (ISBN: 2-914771-07-X).

¹KFKI-Res. Inst. for Particle and Nuclear Physics, PO BOX 49 H-1525, Budapest, Hungary.

Laboratory Operation and Development

M. Ribeiro da Silva

3 MeV Van de Graaff Accelerator Electronics

Some instabilities were detected in the data acquisition and control line connections between the control room and the Van de Graaff experiment lines. A complete overhaul has decided to improve the connections. The signal, power and control lines between the Van de Graaff control room and the experiment lines undergone a complete and integral substitution with upgrading of connections, panels and cabling.

New Laboratory

During the year a complete new Vacuum Test Laboratory was mounted and made operational (Fig.1) in the Physics Hall.



Fig. 1 - General view of the Vacuum Test Laboratory.

This laboratory includes large volume and low temperature (LN₂) thermal choc facilities and a very

Published, accepted or in press work

L.C. Alves, A. Paúl, M.R. Da Silva, E. Alves, B. Riccardi, J.C. Soares, High Temperature Chemical

low level (10^{-9} torr.litre.s⁻¹) He-leak test stand. Parts up to Ø20xL100 cm can be tested.

In the scope of a joint collaboration between the ITN and the industry (Velan) in this laboratory are now routinely tested the bodies of LHe valves for the CERN-LHC.

Also for this laboratory the electronics shop designed and tested new devices for manual and automatic LN₂ level control (liquid depth down to 1,3 m and 1 cm resolution)

General purpose tube furnace

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

This unit presents the following parameters:

max. temperature 900°C, control - K thermocouple;

max. sample Ø 28 mm, solid, powder or pebble;

the sample volume can hold HVac or be pressurised up to 3 bar of inert or slight aggressive gas[1].

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

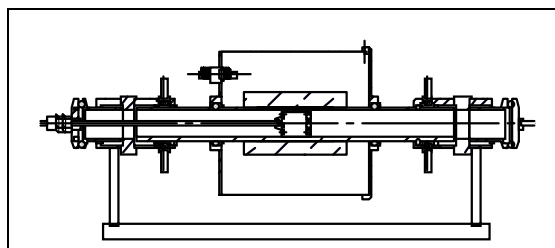
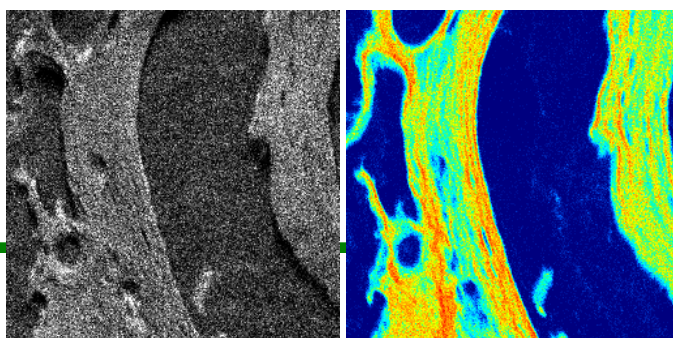


Fig. 2 - General purpose tube furnace.

Compatibility between SiC Composites and Be Pebbles, *accepted Nucl. Instr. and Meth. B.*

Health and Environmental Studies Using Ion Beams



Health and Environmental Studies Using Ion Beams

Teresa Pinheiro

The activities carried out at the Ion Beam Laboratory (IBL) related with Health and Environmental Studies derive from the instrumental and expertise capabilities existing at ITN for the determination of chemical elements. The application of these aptitudes to biological matrices orientated the research activities to the study of human pathological conditions and to the response of biological systems to environmental stimulus.

Presently the instrumental capabilities are becoming fully explored and rapidly overwhelmed by qualified instruments developed in the past decade. Having these premises in consideration, two projects aiming at the modernisation of ITN laboratory facilities were prepared and submitted to FCT by the IBL and the Environmental Chemistry group, in which the HESUIB team was directly involved, for the acquisition of a new Tandem accelerator and an HR-ICPMS instrument, respectively.

The collaborations established with teams from the Biochemistry Centre, the Environmental Biology Centre and the Faculty of Medicine, from the University of Lisbon, enabled to get financial support mainly from national foundations in the scientific areas of Biomedicine and Eco-toxicology. Within current projects, the additional collaborations with the Azores University and with the INSA (Instituto Nacional de Saúde Doutor Ricardo Jorge), enable to study insular populations as a sub-group in the

Portuguese population and to assess specific allelic profiles. Thus, the range of parameters that will be appraised within current projects are considerably enlarged by report to previous studies, which is an outstanding value to characterise complex diseases such as Chronic Pulmonary Diseases and Atherosclerosis or to understand regulatory mechanisms.

International collaborations with the University of Seville and the X-Ray Fluorescence group of the European Synchrotron Radiation Facility at Grenoble have been reinforced, through research projects. Also, international collaboration will soon be enlarged by the consortium joint on NANODERM project supported by the EC.

The multidisciplinary characteristic of the joint teams also permit to attract young scientists to carry out their BSc and PhD thesis, which programmes of study are often within the activities of financed projects.

Apart from research activities, technical services are provided to private institutions, mainly on the characterisation of raw materials for the pharmaceutical industry.

The main achievements of the research developed during 2002 are summarised in the following pages.

Health and Environmental Studies Using Ion Beams

Research Team

Researchers

- T. PINHEIRO* , Auxiliary researcher
- L.C. ALVES* , Research assistant (25%)¹

- P. NAPOLEÃO, Last Year BSc. Student
- A. L. SIMÕES, Last Year BSc. Student, FCG grant

Collaborators

- M.C. SANTOS, Auxiliary Professor, FCUL (100%)
- A.M.V. CRESPO, Principal researcher, FCUL
- A.B. DE ALMEIDA, Full Professor, FMUL
- P. FILIPE, Auxiliary Professor, FMUL
- J.N. SILVA, MD, St. Maria Hospital, FM-UL
- P. MONTEIRO, MD, St. Maria Hospital, FM-UL

Technical Personnel

- R. PINHEIRO
- J. ROCHA
- F. BAPTISTA

Students

- M.D. YNSA, PhD student, Univ. Seville, Spain
- C. MONTEIRO, PhD Student, ISCS, Fac. Sciences, Lisbon University

(*) Also members of CFNUL.
¹ Doing PhD.

Funding (€)

Research Projects:	7 519
Total:	7 519

Publications

Journals:	2 and 5 in press
Proceedings:	1
Conf. Communications:	12
Other publications:	
Theses: BSc	1

Biomedical and Eco-toxicological Studies

MC. Santos¹, C. Monteiro^{1,2}, A.M. Viegas-Crespo³, M.L. Mathias³, C. Sousa Reis⁴, P. Napoleão⁴, A. Bugalho de Almeida⁵, P. Monteiro⁵, J.N. Silva⁶, P. Filipe⁶, F. Marujo⁷, M.D. Ynsa⁸, M.F. Araújo⁷, A. Barreiros⁹, L.C. Alves¹⁰ and T. Pinheiro¹⁰

Objectives

The ion beam laboratory of ITN runs several collaborations with other institutions in the field of biology and medicine. The main research activities focus the relationship of morphological and biochemical markers that may express the response of organisms in health and disease conditions. The role of ITN is to provide information on the morphological characterisation of biological tissues, in particular the distribution of trace elements that are toxic or related with the redox equilibrium.

Results

1. Biomedical Studies - Human Diseases

1.1. Chronic pulmonary diseases

The study of physiological alterations in patients with Chronic Obstructive Pulmonary Disease (COPD) and their relationship with biochemical markers in biological fluids are being carried out under the contract FCG/SDH.IC.I.01.22 since December 2001. Sample collection procedures were established and validated. Several techniques and methodologies were developed for the determination of specific enzyme activities, specific proteins and cell factors. Sample collection started in October and so far, 20 patients and 6 healthy donors were studied.

Also, the study of morphological characteristics of lung tissue and the diffusion of respired toxic elements in respiratory mucosae has been assessed combining nuclear microprobe and synchrotron radiation [1].

1.2. Atherosclerosis

The study of atherosclerotic pathology in what concerns the relationship of apoE allelic profiles with the redox equilibrium is being carried out under the Project POCTI/ESP/41008/2001 since last September.

Published, accepted or in press work

1. T. Pinheiro, L.C. Alves, F. Araújo, A. Barreiros, S. Bohic, A. Simionovici, Imaging and quantification of trace metals in biomedical samples using Synchrotron Induced X-ray Fluorescence and Nuclear Microprobe techniques, *J. Phys. IV* (in press).
2. T. Pinheiro, J.N. Silva, L.C. Alves, P. Filipe. Iron deposition in skin of patients with haemochromatosis, Submitted to *Nucl. Instr. and Meth. B*.

So far, selection of students (graduated and PhD) to join the project and validation of techniques for DNA and protein isolation were the activities carried out.

1.3. Skin Studies

Skin alterations in haemochromatosis, which include the use of Fe content of skin as an indicator for the disease evolution and skin morphology are being studied [2]. This last aspect is connected with the launching of NANODERM project early next year (EC/QLK4-CT-2002-02678) that has been selected by the Commission for financial support.

2. Eco-Toxicological studies

2.1. Biological monitoring in terrestrial ecosystems.

The study of bioavailability of lead in small mammals at mine contaminated areas in Alentejo was initiated during 2002 under the project POCTI/BFE/3991/2001. The environmental evaluation of the study areas, has been carried out and sample collection started in September. The project includes a PhD programme of study in Biology.

2.2. Biological monitoring in marine ecosystems

The Octopus as a bio-indicator of the marine ecosystem and the study of its life cycle are investigated. A Biology Graduation Thesis [3] was done within the work programme of the project PRAXIS XXI/2/2.1/MAR/170/95-1999-2002.

3. Se Biochemistry

The effect of early Se supplementation in rats at moderate levels is studied in rat liver, testes and blood to assess the toxicity of selenite [4]. The work includes a PhD thesis in Biochemistry.

4. Osteoporosis - Portugal-Spain Cooperation

Study of the effect of steroid-based therapies in osteoporosis is being carried out using an animal model to assess bone and uterus alterations. The study includes a PhD programme in Applied Physics [5].

3. P. Napoleão, *Caracterização morfológica e elemental em Octopus vulgaris, Cuvier, 1797, na costa Portuguesa*, Biology Graduation Thesis, Fac. Ciências, Univ. Lisboa, 2002.
4. C. Monteiro, N. Raimundo, M.C. Santos, T. Pinheiro, R.E. Pinto, Rubidium levels are affected by Selenium Supplementation, Submitted to *J. Nutrition*.
5. M. D. Ynsa, F.J. Ager, M.A. Zubeldia, J.C. Millán, M.A. Respaldiza, T. Pinheiro, Long bone remodelling in osteoporosis, *Nucl. Instr. and Meth. B* (in press).

¹ Centro de Bioquímica, FC-UL, Portugal ² Instituto Superior de Ciências da Saúde, Portugal

³ Centro de Biologia Ambiental, FC-UL, Portugal ⁴ Dept. de Biologia Animal, FC-UL, Portugal

⁵ Clínica de Pneumologia, FM-UL, Portugal ⁶ Clínica de Dermatologia, FM-UL, Portugal

⁷ Chemistry Sector, ITN ⁸ CNA, Univ. Seville, Spain ⁹ INETI, Lisboa, Portugal ¹⁰ Physics Sector, ITN

University Group of Nuclear Reactions

Adelaide Pedro de Jesus

This group has been involved in the study of proton-induced nuclear reactions with the objectives to obtain cross sections of nuclear reactions relevant to nuclear astrophysics and to extend analytical capabilities to light elements.

So far, the experimental work has relied upon ITN – Ion Beam Laboratory, based on a 2.5MV Van de Graaff accelerator. This facility has allowed the development of an accurate method to measure in an absolute way cross sections of relevant nuclear reactions. Also for the applied point of view an effort has been done to complement the already installed PIXE facility by developing a set-up of PIGE analysis for light elements.

In order to proceed with experimental work related to astrophysically relevant nuclear reactions it is imperative to be able to measure the respective cross sections at very low energies, where the cross sections are lower than the picobarn. At these energies several challenges must be overcome: the effects of cosmic rays in the detectors must be reduced to almost zero with an adequate detector shielding, the Coulomb barrier is partially screened by the atomic electron cloud surrounding the nuclei, which leads to an enhanced cross section compared with bare nuclei cross section; stopping powers are

not well known. So, we have joined LUNA (Laboratory for Underground Nuclear Astrophysics) collaboration.

Work under LUNA collaboration has been centred on the study of electron shielding effects on nuclear reaction cross sections at very low energies and on the study of the reactions $d(p,\gamma)^3\text{He}$ and $^{14}\text{N}(p,\gamma)^{15}\text{O}$. Target preparation and stoichiometry analysis by ERD, RBS, PIGE and NRA, has been done at ITN.

In the short term the work to develop a calibrated PIGE set-up will be concluded, opening new perspectives in applied work for Environment, Materials and Health Sciences and Geology. Also, work under LUNA collaboration will proceed with the study of relevant reactions of the p-p chain and CNO cycles. This collaboration will imply some experimental work at ITN, namely for target preparation and stoichiometry analysis by ion beam techniques and cross section measurements for normalisation of LUNA measurements.

In the medium term, the acquisition of a new accelerator would open new research areas within nuclear astrophysics, as the study of relevant reactions of the p-process nucleosynthesis.

University Group of Nuclear Reactions

Research Team

Researchers

- A.P. DE JESUS, Full Professor (FCT/UNL), Group Leader
- J.P. RIBEIRO, Associate Professor (FCUL)

Students

- B. BRAIZINHA, PhD Student, FCT grant
- J. CRUZ, PhD Student, (FCT/UNL)
- R. MATEUS, PhD Student, FCT grant
- J.V. PINTO, BIC PRAXIS XXI

Funding

Budget in 2002 €

Research Projects:	9 100
Total:	9 100

Publications

Journals:	1 and 1 in press
Proceedings:	1
Conf. Communications:	1

Experimental Study of Nuclear Reactions for Astrophysics

A.P. Jesus^{1,2}, J. Cruz^{1,2}, B. Braizinha², R. Parafita, J.P. Ribeiro^{2,3}

Objectives

Our understanding of the evolution of the very early universe depends crucially on modelling the primordial nucleosynthesis of the light elements. Calculations of primordial and stellar nucleosynthesis are strongly affected by uncertainties in the nuclear input data. In order to lower the uncertainties of the astrophysical factor of relevant reactions of the p-p chain and CNO cycles, a research program was established involving:

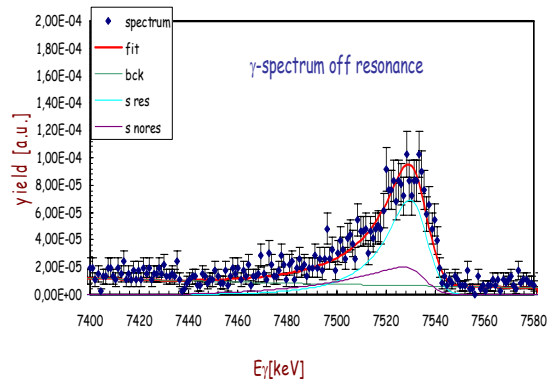
1. Development at ITN of an experimental method to determine in an absolute way the reaction cross sections.
2. Experimental work on reaction cross-sections at relevant energies (around the solar Gamow peak) under LUNA (Laboratory for Underground Nuclear Astrophysics) collaboration.

Results

At ITN, the work was focused on analysis of TiN and ZrN targets, produced both by sputtering deposition and implantation. X-Ray diffraction, AMF microscopy and RBS were used as analytical techniques. The aim of the work was the fabrication of pure and stable targets for the study of the reaction $^{14}\text{N}(p,\gamma)^{15}\text{O}$. During 2002, after tests and calibration procedures, the measurement of this reaction was made in Gran Sasso, within LUNA collaboration, below the 278 keV resonance down to 140 keV with solid targets [1-3].

Work with gas targets will start in 2003 aiming at the solar Gamow peak. The figure below pertains to a gamma-ray spectrum obtained at 170 keV, showing

the fitting procedure to the shape of the gamma-ray line to extract the reaction cross section.



Published, accepted or in press work

1. M. Junker (LUNA collaboration), "Advances in cross section measurements with underground accelerators", Nucl Phys. B Proceedings Supplement 110 (2002) 247.
2. S. Zavatarelli (LUNA collaboration), "The present status of the LUNA facility at the underground Gran Sasso Laboratory", AIP Conference Proceedings 610 (2002) 485.
3. A. Formicola (LUNA collaboration), "A new study of the $^{14}\text{N}(p,\gamma)^{15}\text{O}$ reaction at low energy", NPDC17 – Nuclear Physics in Astrophysics, Debrecen, Sep. 2002.

¹ Departamento de Física da Faculdade de Ciências e Tecnologia da Universidade Nova de Lisboa

² Centro de Física Nuclear da Universidade de Lisboa

³ Departamento de Física da Faculdade de Ciências da Universidade de Lisboa

Calibration of a PIGE Set-up

R. Mateus³, J. Cruz^{1,2}, J. P. Ribeiro^{2,3}, A.P. Jesus^{1,2}

Objectives

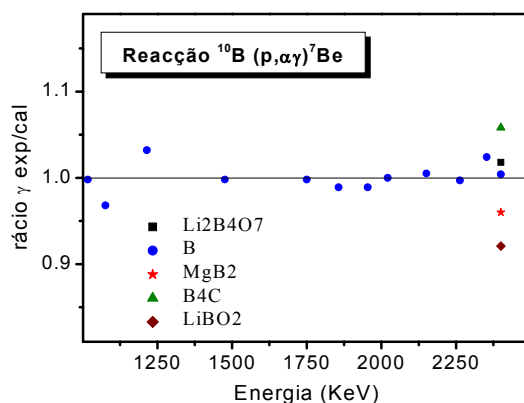
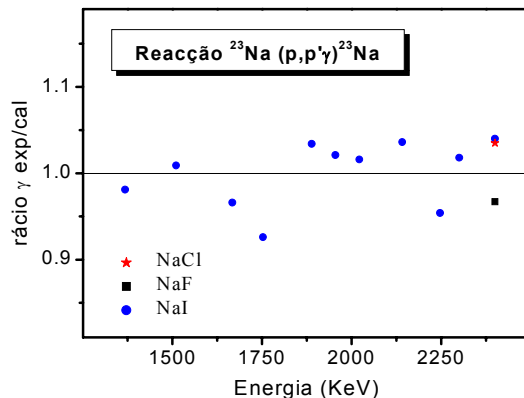
The aim of this work was the development of an analytical set-up for light element analysis, based on the detection of the gamma radiation induced by low energy protons, PIGE, in order to complement the already installed PIXE analytical facility.

This technique will open new perspectives of applied work in environment and health problems.

Results

A precise method based on a code that integrates the nuclear reaction excitation function along the depth of the sample was implemented for thick and intermediate samples. For that purpose some reaction excitation functions were measured in the same analytical conditions. The energy steps needed to define accurately the excitation function were used as energy intervals for the integration procedure.

After the work done in 2001 for F and Li [1], excitation functions for $^{23}\text{Na}(p,\alpha\gamma)^{23}\text{Na}$ were obtained and introduced as input. Thick target gamma yields for several samples containing Na were calculated and compared with yields obtained experimentally. The same was done for B. The agreement between experimental and calculated results is better than 5% for all proton energies used and for all the targets employed.



Published, accepted or in press work

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Lithium in thick samples", *Nucl. Instr. Meth. B* 190 (2002) 117-121.

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Condensed Matter Physics



Condensed Matter Physics

Frederico Gama Carvalho

The Group's field of research is the development and characterisation of materials, using radiation as a tool to investigate the structure and/or induce structural modifications in materials. The group is also active in the area of instrument development, including design, construction, and test of systems and components for low-energy neutron scattering and X-ray diffraction. Systems currently under investigation include semiconductors, high temperature alloys and organic-inorganic hybrid materials. Developing scientific contacts with national Universities has a high priority in tune with the policy of providing a specialised research infrastructure for external users built around the Group's local facilities. It is a concern of the Group to be instrumental in promoting the utilisation of neutron and X-ray techniques for structural studies among the country's scientific community and to do so in co-operation with European neutron scattering centres. Since 1998 participation of the Group's co-ordinator in the regular meetings of the European Neutron Scattering Association (ENSA) have served this purpose.

Neutron beam facilities. The Two-Axis Neutron Diffractometer, DIDE, equipped with a "banana" multidetector and the rotating chopper Time-of-Flight Diffractometer, ETV, are installed in two of the RPI research reactor beam tubes; installation work of a Small Angle Neutron Scattering Instrument (EPA) is currently in progress. Routine operation of DIDE and EPA will contribute to significantly increase the reactor utilisation and thus justify the continued operation of the reactor. The TOF diffractometer is dedicated to tests and student training. Presently, the thermal neutron flux available at the RPI with well designed beam-tube facilities will allow the measurement of only certain classes of samples and to carry out in-beam experiments of components and devices such as detectors, collimators and neutron optical components. Recourse to higher flux neutron sources abroad will in many cases be essential eventually after a convenient choice of sample and measurement parameters by carrying out preliminary measurements locally. In this context, the feasibility of a reactor power increase and eventually of installing a cold neutron source near the reactor core should be considered. Co-operation of the Reactor staff has been essential to smoothing away the numerous difficulties of installation work. Also in the case of DIDE funds were provided by the Reactor Department to purchase certain indispensable services and parts. Collaboration of the Nuclear Instrumentation Group of the Physics Department has also been essential in respect to the design and test of electronics and software.

Theoretical development of the concept and project of a prototype neutron Converging Multichannel Collimator (CMC) have experienced considerable progress in 2002. The work, funded by the National Science Foundation and the IAEA, has attracted the interest of the international neutron scattering community.

Co-operation with the Budapest Neutron Center in the area of instrument development, and collaboration with the Glass and Ceramics Dept. of Aveiro University, Laboratoire Léon Brillouin (Saclay), and Sophia University in the study of organic-inorganic hybrids, have continued in 2002.

The activity at the MA³T laboratory. During 2002, the third year of activity of the High Temperature Materials Laboratory (MA³T) the main aims was two fold: the characterisation of materials and the continuation of the development of the Hotbird capabilities through the implementation of new hardware components and software applications. Presently three main geometries are implemented: the *high-intensity mode*, the *double-crystal diffractometer* and the *triple-axis* geometries.

Concerning the research work, emphasis was put on the study of nano-structured materials. This has been a tendency over the years owing, in part, to the enhanced capabilities of the Hotbird diffractometer to study very thin layer structures when compared to commercial diffractometers.

The current research projects are centred on the study of advanced materials used in the electronic industry as well as high-temperature alloys used in the aeronautical industry. Many of these studies are performed in collaboration with foreign research groups.

Although most of this work was centred on single crystalline materials several polycrystalline materials have also been studied, namely magnetic multilayer sensors, nano-precipitates in implanted materials, ceramic powders, etc.

Two new projects have been approved by the end of the year to study (1) *nanometer-thick buried layers in SiGe devices* and to characterise (2) *magnetic tunnel junctions* used for hard-disk reading heads. This will also allow to reinforce the team with two new students for the next two years.

An application to the *National Program for the Large Scale Facilities* of the Portuguese Science Foundation has been prepared and submitted to the FCT. If approved this would allow to enhance of the MA³T laboratory capabilities with new techniques not yet available in Portugal.

Condensed Matter Physics

Research Team

Researchers

- F.G. CARVALHO, Senior Research Officer, Group Leader (90%)
- J.F.SALGADO, Senior Research Officer (30%),
- F.M.A. MARGAÇA, Research Officer
- A.N. FALCÃO, Research Officer
- A.D. SEQUEIRA, Auxiliary Researcher
- J.S. NEVES, Auxiliary Researcher (20%)

Students

- N.P. FRANCO, PhD Student, FCT grant
- P. FERREIRA, PhD Student, IST/ITN, FCT grant
- J.L. COSTA, PhD Student, IST/ITN, FCT grant
- M. CARRAPIÇO, BIC POCTI, MSc student
- E. SOCHA, undergraduate student
- D. SILVA, undergraduate student, ITN Fellowship

Funding (€)

Research Projects:	45 432
ITN:	27 893
Other Sources:	—
Total:	73 325

Publications

Journals:	12 and 4 in press
Proceedings:	3
Conf. Communications:	11
Theses:	2 BSc

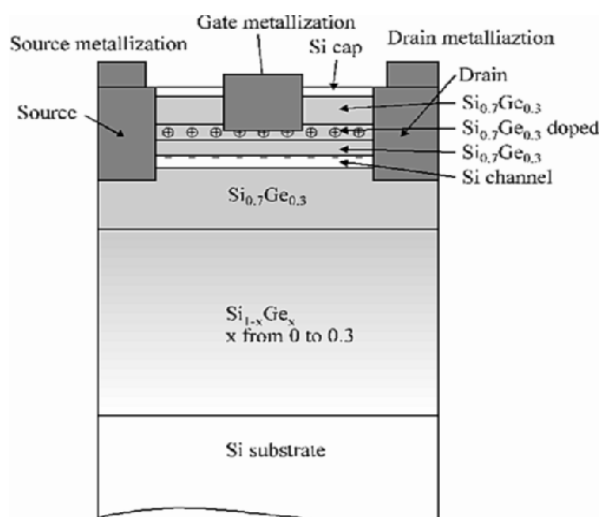
Strain Status and Diffusion in $\text{Si}_{1-x}\text{Ge}_x/\text{Si}_y\text{Ge}_{1-y}/\text{Si}$ (001) Heterostructures for MOSFET production

A.D. Sequeira, N. Franco, K.D. Chtcherbatchev¹, N.P. Barradas²,
M. Myronov³, O.A. Mironov³, E.H.C. Parker³

Objectives

The aim of the current study is the structural characterisation of p-type modulation doped $\text{Si}_{1-x}\text{Ge}_x/\text{Si}_y\text{Ge}_{1-y}/\text{Si}$ heterostructures using high resolution X-ray diffraction. In particular to study the strain status, thickness and thermal stability of H MOS transistors after thermal annealing at temperatures ranging from 600 to 900°C.

This work is being carried out in collaboration with Prof. Parker's group of the University of Warwick.



full relaxation of the intermediate part of the virtual substrate. Both the uppermost and lowermost parts of the virtual substrate regions were found to be not fully relaxed even after annealing at 750°C. The increase of the annealing temperature leads to a broadening of the

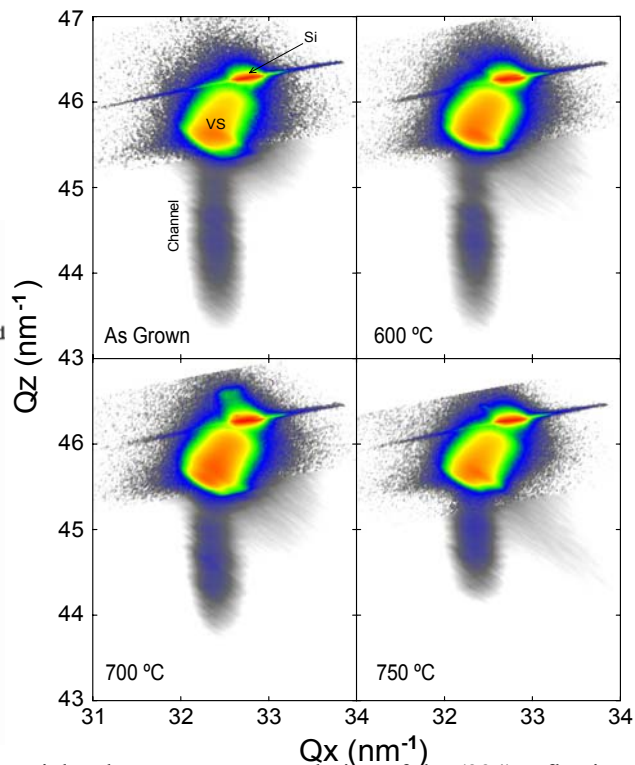


Fig. Left: The structure of a SiGe MOSFET transistor. Right: the temperature evolution of the (224) reflection (right) of the transistor structure shown on the left.

Results

To study the thermal stability and strain relaxation mechanisms in p-type modulation doped $\text{Si}_{1-x}\text{Ge}_x/\text{Si}_{1-y}\text{Ge}_y/\text{Si}$ (001) heterostructures on virtual substrates. Ex-situ post-growth furnace thermal treatments were done in a N₂ ambient at various temperatures. It was found that the high temperature annealings lead to the

channel layer with a decreasing of its Ge concentration though remaining fully strained.

The use of the Hotbird X-ray diffractometer with its optimised geometry allowed us to reveal and study the very weak intensity scattering from a 4nm thick $\text{Si}_{0.2}\text{Ge}_{0.8}$ channels.

Published, accepted or in press work

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2. A.D. Sequeira. Strain status of $\text{Si}_{1-x}\text{Ge}_x/\text{Si}_{1-y}\text{Ge}_y/\text{Si}$ (001) Nanostructures studied by High-Resolution X-ray Diffraction, 3^{as} Jornadas de Tratamentos Térmicos e Engenharia de Superfícies TTES-2002. IPN- Instituto Pedro Nunes, Coimbra, 6 Dez. 2002.

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³ Nuclear Solid State Physics using ion Beams group.

Determination of Structural Properties of InGaN

A.D. Sequeira, S. Pereira¹, E. Alves², N.P. Franco, K.P. O'Donnell³

Objectives

The main aim of the current project is to study the effect of strain on the optical properties of epitaxial single layers of $\text{In}_x\text{Ga}_{1-x}\text{N}$. The main characterisation technique is high-resolution x-ray diffraction.

Results

The material. Group III nitride epilayers are currently a major topic of research due to their widespread use in light emitting diodes (LED) and laser diodes (LD). Commercial indium gallium nitride (InGaN) LEDs, developed over the last decade, enjoy unrivalled performance in the UV, blue and green spectral regions

XRD analysis. In this study emphasis has been given to the characterization the structural properties of

InGaN structures of low dimensionality in particular multilayers and quantum dots (an example is give in the Fig.). The reciprocal space mapping is a technique particularly suited to determine structural parameters of these structures. In particular the strain status of the InGaN layers relative to the GaN substrate. Some of the typical problems that have been addressed and published are:

- A new method was developed for the determination of the composition of the InGaN films;
- Alternative model was developed to explain the appearance of two peaks that has been claimed to be phase separation;
- An anomalous dependence of the energy gap on the In content was found.

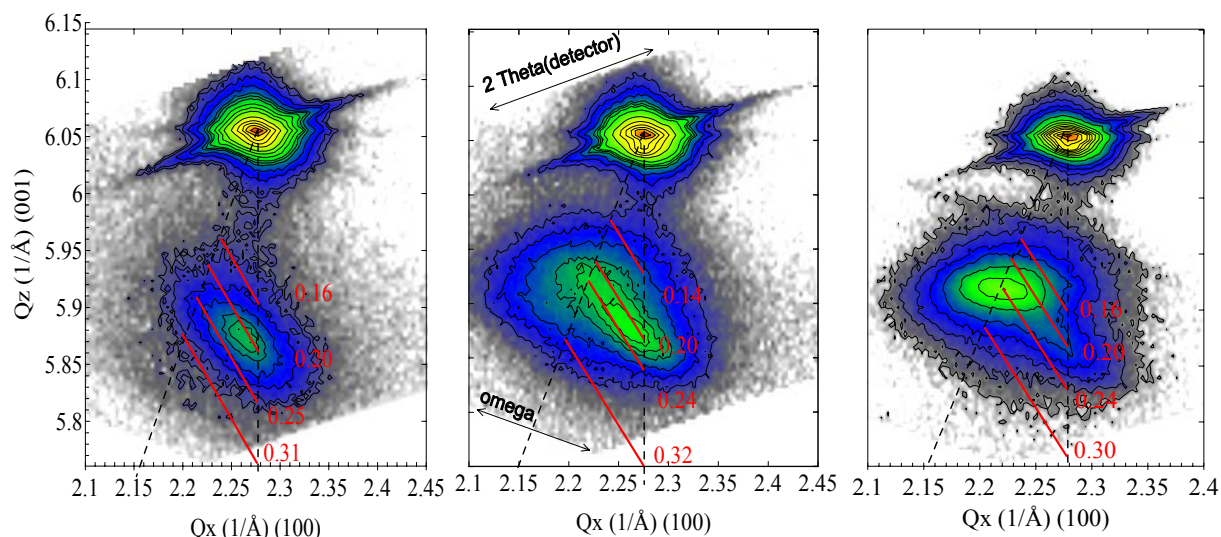


Fig. Reciprocal space maps for the (105) reflections of the three InGaN/GaN layers with increasing thickness. The lines connecting the fully strained to the fully relaxed dashed lines indicate the calculated relaxation directions in the reciprocal space for various InN mole fractions.

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Study of Thin Coatings for industrial Applications

A.D. Sequeira, C. Nunes¹, M.L. Prates¹, N.P. Barradas² and V. Teixeira³

Objectives

The aim of this project is to produce and characterise multilayered solar graded cermet selective coatings based on chromium and titanium deposited on Cu substrates to be used on solar energy collectors.

Results

The use of several characterisation techniques allowed to optimise the coating structure. For instance the RBS

analyses allowed the characterisation of the metal concentration gradients.

The best performance coating is composed of three cermet layers between the substrate and the reflecting layer. The total thickness should be between 100 e 300nm. The best results were obtained for Cr-Cr₂O₃ samples with 94% of light absorption and only 6% of emissivity.

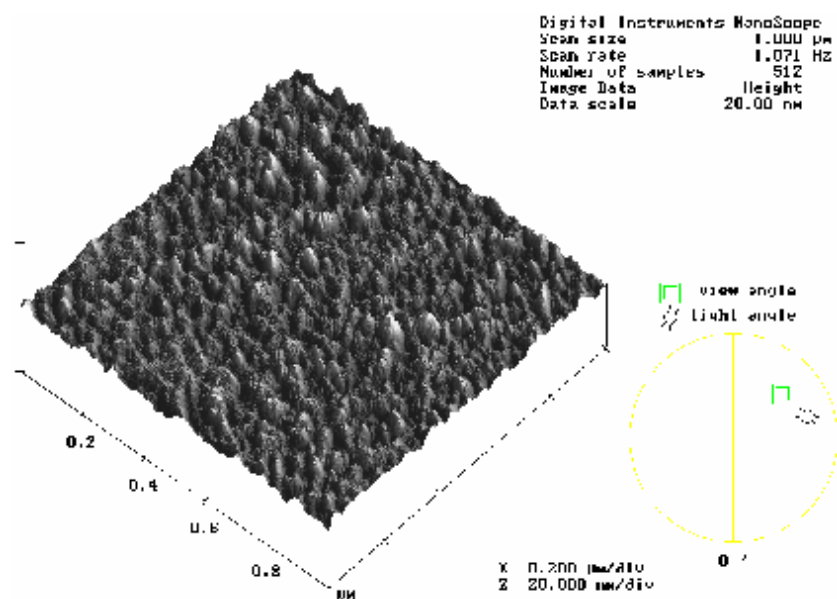


Fig. AFM image of the surface of a solar graded cermet of Ti-TiN_yO_x 200nm thick.

Published, accepted or in press work

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3. C. Nunes, V. Teixeira, M.L. Prates, N.P. Barradas, A.D. Sequeira, Revestimentos finos graduados de Cr-Cr₂O₃ e Ti-TiN_yO_x com selectividade espectral, Proceedings of TTES-2002, 3as Jornadas de Tratamentos Térmicos e Engenharia de Superfícies, Ed. Vasco Teixeira, Teresa Vieira, H. Santos, P. Loureiro, SPM-Sociedade Portuguesa de Materiais, Univ. Coimbra, 2002.
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³ CFUM-Centro de Física da Universidade do Minho, Universidade do Minho.

Determination of Defects Density on GaAs upon implantation by means of high-resolution XRD and RBS

A.D. Sequeira, K. Chtcherbatchev¹, E. Alves², N.P. Barradas²

Objectives

The aim of the current project is to study the influence of in-situ photo-excitation on the defect structure generation in GaAs crystals implanted by Ar⁺ ions with energy of 200 keV and various doses

The characterisation of the samples is performed using two main techniques: high-resolution X-ray diffraction (XRD) and Rutherford backscattering spectroscopy (RBS).

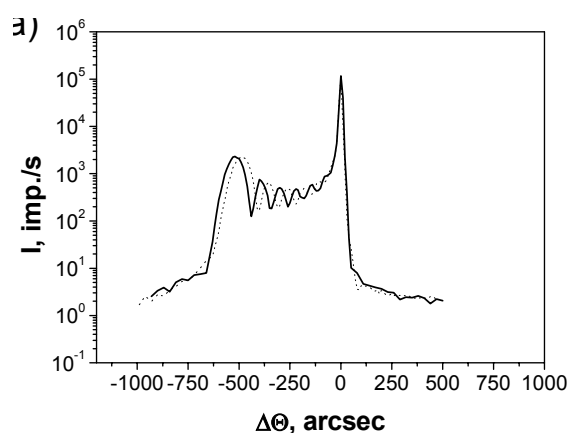


Fig. 1 X-ray diffraction patterns for GaAs wafers implanted by Ar⁺ ions in condition of in situ photo-excitation (dashed line) and without UV irradiation (solid line) with the dose of $1 \times 10^{13} \text{ cm}^{-2}$.

Results

The ion implantation induces a significant number of radiation point defects (RPD) into the semiconductor substrate. The damage depth profile in the implanted

Published, accepted or in press work

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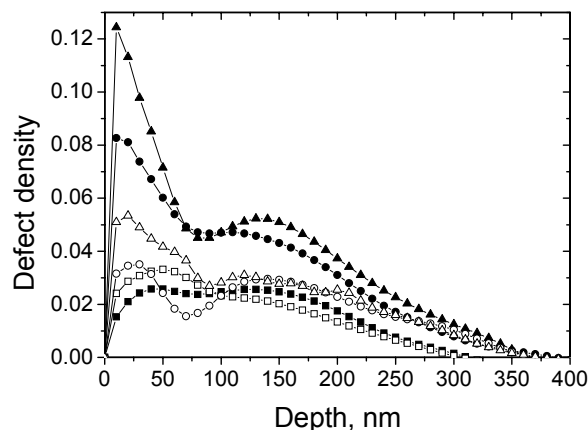


Fig. 2 Relative concentration of displaced lattice atoms as a function of depth for 200 keV Ar⁺ implantation into GaAs with various doses. Opened symbols corresponds to UV irradiated sample.

layers can be reconstructed from the diffraction curves obtained by the high-resolution X-ray diffraction in near non-dispersive triple-crystal arrangement ($n, -m, n$) with Bragg symmetrical reflections.

The distribution of intensity in the vicinity of the 400 reciprocal lattice point was analyzed using a triple-crystal arrangement. The in situ photo-excitation is found to provide for annihilation of Frenkel pairs that decrease a residual concentration of radiation-induced point defects. The amorphisation of the damaged layer proceeds by a generation and a growth of radiation-induced point defect clusters. Application of in situ photo-excitation can be used to react upon a residual concentration of point defects through the change of “cluster formation – annihilation” processes ratio.

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Study of Diamond Films, Implanted Metals, and Semiconductors by XRD

A.D. Sequeira, N. Franco, F.J. da Silva¹, V. Teixeira², A.P. Gonçalves³, R.C. da Silva⁴

Objectives

This line of activity includes multidisciplinary work on the characterisation of several materials in collaboration with various national research groups. The materials range from light metals modified by ion implantation, amorphous metallic coatings, ultra-hard diamond coatings, etc. Some of these materials have relevant industrial applications.

Results

In the figures one illustrates two examples of measurements performed on ultra-hard coatings of diamond used to enhance the properties of mechanical tools. Here the extremely high residual stresses (up to 10GPa) were measured in the outer film of diamond deposited on a steel with intermediate layers of Cu

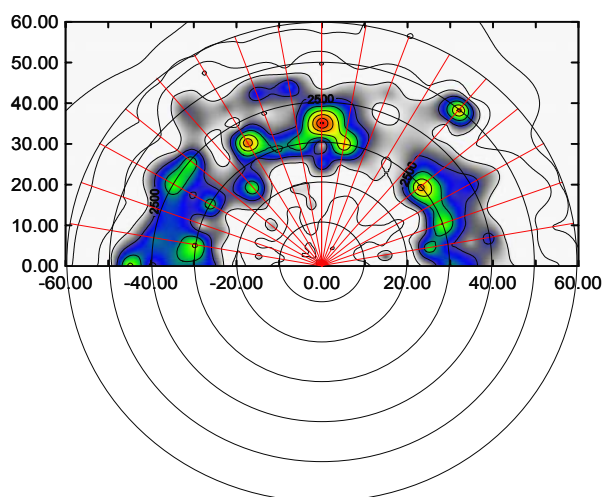


Fig.1 Pole Figure of a multilayered ultra-hard diamond coating.

and Co as buffer layers to accommodate partly the stresses generated during the deposition process. Another important feature of these films is the texture observed in the diamond layer. A (111) pole figure of one of these samples is illustrated in Fig. 1.

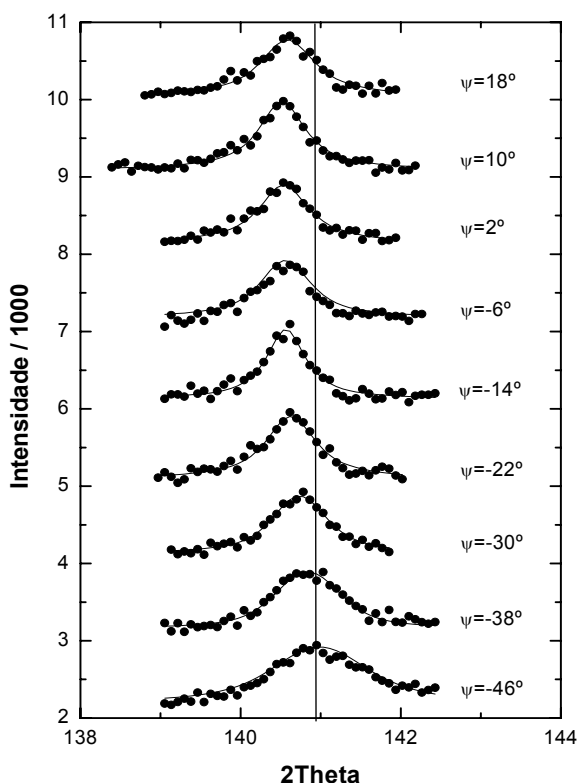


Fig.2 Sequence of spectra and fitting curves of the (331) Bragg peak for the determination of residual stresses on a ultra-hard diamond film.

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² CFUM-Centro de Física da Universidade do Minho, Universidade do Minho

³ Solid State Group.

⁴ Nuclear Solid State Physics using ion Beams group.

Software and Hardware Development at Hotbird

N.P. Franco and A.D. Sequeira

Objectives

The development work is being made in two fronts: specific hardware upgrades and the continuous updating of software used for data collection and data analysis.

Results

Software. During this year a few tools for data analysis have been improved and in particular two new applications has been developed. The first experimental procedure is called *Texture*. This application produces of intensity distribution maps

around specific crystallographic directions allowing for the determination of the level of texture on polycrystalline materials (see Figure).

A new method was developed to determine residual stresses on single crystalline materials was implemented in the program “Stress”.

Hardware. During the current year a few new sample holders has been developed to the Hotbird to allow using larger samples in the high-intensity geometry. The restructuring of the optical components of the diffractometer in the incident beam path have started and will be finished next year.

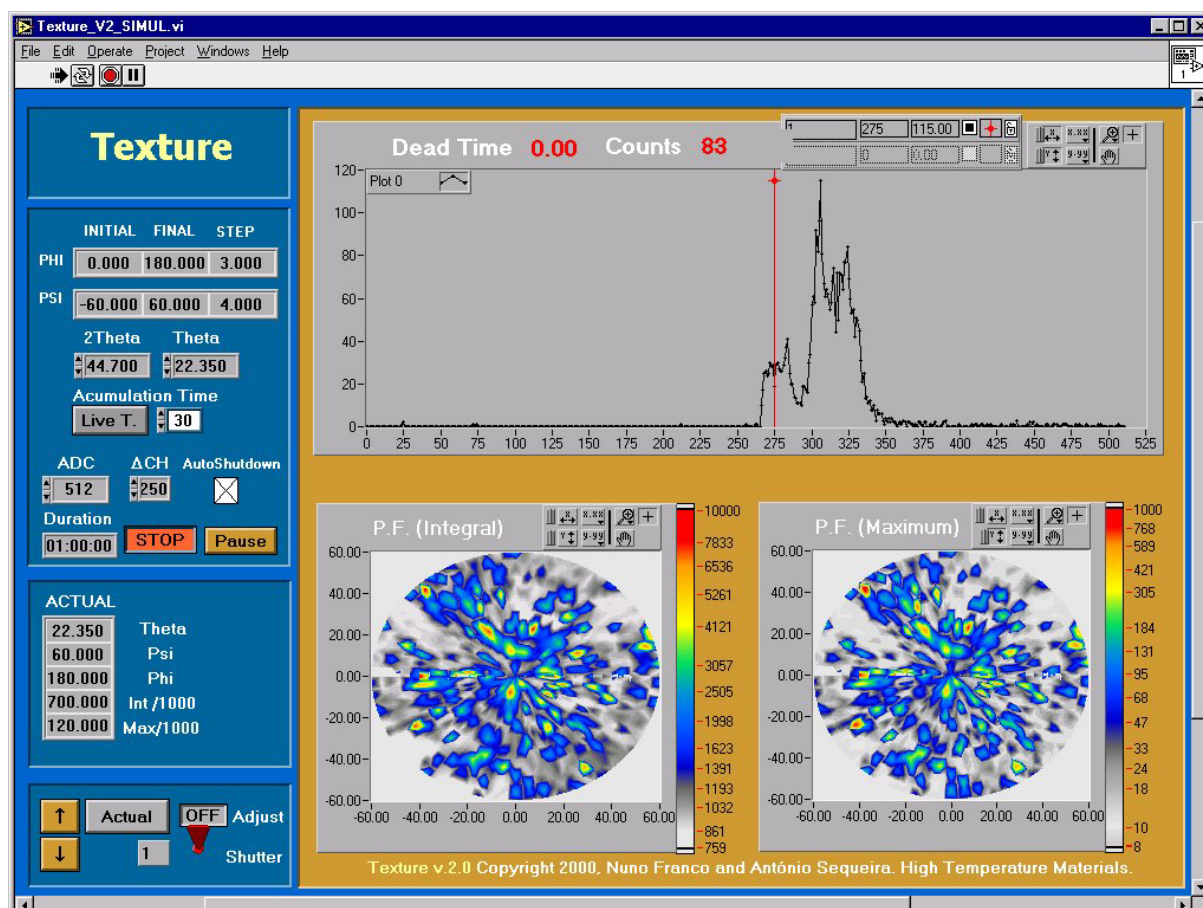


Fig. – User interface of the *Texture* application used to perform texture patterns.

Published, accepted or in press work

1. N. Franco, S. Pereira and A.D. Sequeira. Reciprocal Space Mapping on an Absolute Scale in X-ray Diffractometers Incorporating a Position Sensitive

Detector Applied to Group-III Nitride Alloys. *J. Phys. D: Appl. Phys.*

Preparation and characterization of materials by sol-gel and irradiation

A.N. Falcão, F.M.A. Margaça, I.M. Miranda Salvado¹ and F.G. Carvalho

Objectives

Preparation of hybrid materials with predictable properties by gamma irradiation and the alkoxide method of the sol-gel process. Sample characterisation, mainly by neutron scattering.

Results

Gamma ray irradiation together with classical alkoxide method procedures can lead to materials with a wide range of properties. This new combination of techniques is extremely promising as a new way to tailor material properties.

Samples have been produced with properties ranging from those characteristic of conventional glasses to those typical of flexible materials (elastomers), depending on the preparation protocol and on the organic-to-inorganic constituent ratio. Small angle neutron scattering, SANS, studies of some of these materials have been carried out [1,2].

Materials were obtained by sol-gel, mixing a silicon alkoxide with polydimethylsiloxane silanol terminated with an organic/inorganic volume ratio, VR, of 2/3. A small molar percentage of TiO₂ or ZrO₂ was incorporated into SiO₂

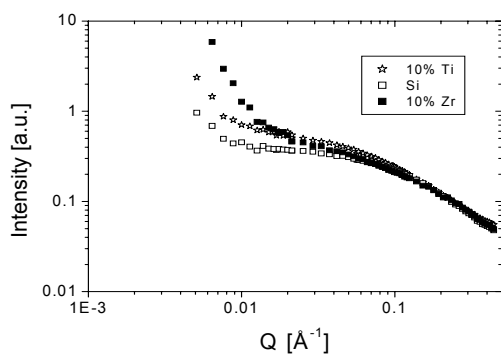


Fig.1 Influence of the incorporation of zirconia and titania in the SANS intensities from the samples.

Results showed that the polymer binds to the growing oxide regions during gelation, being well distributed inside the matrix. The local organization of low molecular weight polymer is more disturbed than that of the high molecular weight hybrids. If only the Si alkoxide is present, the low molecular weight polymer can arrange itself in some more elaborated structure, during the gelification process. If the highly reactive Zr alkoxide is present, gelification occurs so rapidly that the structure of the hybrid appears to be the quenched structure of the homogeneous sol mixture. SEM confirmed a good homogeneity in all samples.

Hybrid materials have also been produced by gamma irradiation of the precursors using ITN ⁶⁰Co source. The precursors were the same as used in the sol-gel prepared materials except for the molecular weight of the polymer that was higher for the irradiated samples. Samples with VR of 4/1, 3/2, 1/1, 2/3 and 1/4 were prepared. The irradiation dose for gel point depends on the volume ratio as expected and increases with decreasing volume ratio. For VR=2/3, samples were irradiated to several irradiation doses above that corresponding to the gel point, which becomes higher for higher fraction of added ZrO₂. The resulting samples are homogeneous, transparent and flexible and subject to swelling in a good solvent of the polymer. The microstructure of the samples was investigated by SANS.

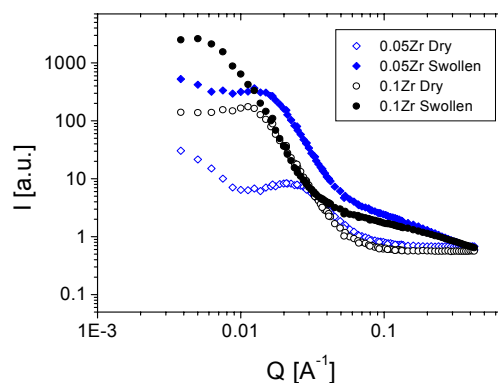


Fig.2 SANS results show the presence of dense inorganic clusters interconnected by polymer structures.

The addition of zirconia in the preparation phase changes the structure of the final product. Work is in progress to investigate if the observed structure results from microphase separation already present at the sol stage or if it is mainly an irradiation induced phenomenon.

Published, accepted or in press work

1. A.N. Falcão, M. Carrapiço, J. Santos Sousa, F.M.A. Margaça, L.M. Ferreira, F.G. Carvalho, I.M. Miranda Salvado and J. Teixeira, Investigation of organic-inorganic materials prepared by irradiation, *J. Sol-Gel Sci. and Tech.* **26** (2003) 349-352.
2. A.N. Falcão, J. Santos Sousa, F.M.A. Margaça, F.G. Carvalho, I.M. Miranda Salvado and J. Teixeira, SANS Study of zirconia-silica titania-silica hybrid materials, *J. Sol-Gel Sci. and Tech.* **26** (2003) 345-348.

¹ Glass and Ceramics Engineering Dpt., University of Aveiro 3810-193 Aveiro, Portugal.

Intensity and Resolution Effects in Converging Multichannel Collimation

A.N.Falcão, F.M.A. Margaça, F.G. Carvalho

Objectives

Computer simulation work performed to guide the construction of a first prototype of a converging multichannel collimator (CMC) with variable geometry, revealed that the performance of each individual channel is modulated by the coupling between the divergence of the incident neutron beam and the inclination of the individual CMC channel. This was thoroughly studied.

Results

In a CMC, all the elementary channels transmit the same angular divergence $\Delta\theta$ around their central axis. However, the angle θ_{channel} that the central axis of each channel makes with the beam axis, increases when moving in the direction of the outer channels of the CMC. These outer channels will turn useful neutrons that would be lost in a single collimator arrangement.

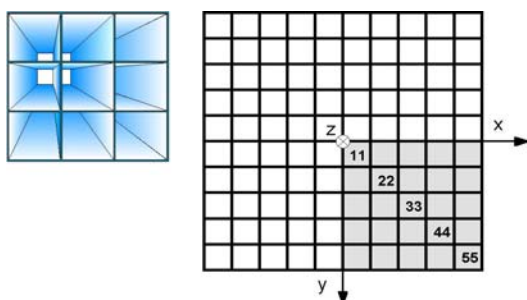


Fig. 1 - Schematic view of a CMC and entrance plane of a unit with 10x10 channels. Some reference channels are numbered.

To take full advantage of this feature, the divergence of the incident beam, α_{MAX} , has to be larger than the largest θ_{channel} . If β is the polar angle of a transmitted neutron flight direction, it is shown that when $\alpha_{\text{MAX}} > \beta_{\text{MAX}}$ all the elementary channels of the CMC perform in the same way, giving identical contributions to the overall resolution and intensity.

The situation is different when $\alpha_{\text{MAX}} < \beta_{\text{MAX}}$. Fig. 2 shows a contour plot of the intensity reaching the exit window of the CMC in that case. It can be seen that, particularly for the outer channels, those whose transmission is below the maximum possible, the spatial distribution of the transmitted neutrons is shifted towards regions more distant from the CMC centre.

Published, accepted or in press work

1. A.N. Falcão, F.M.A. Margaça and F.G. Carvalho, Intensity and Resolution Effects in converging Multichannel Collimators for SANS by Monte-Carlo Simulation, *Jour. Applied Crystallography* (2002). Accepted.

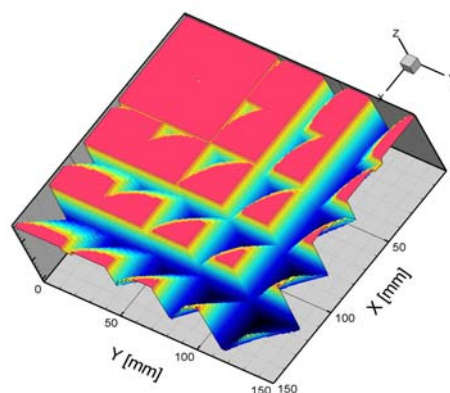


Fig. 2 - 3D plot of the intensity transmitted by the channels whose entrance is shaded in Fig. 1.

The shape of the resolution function was studied by simulating a zero-width Bragg peak. Along with the expected decrease in intensity, the peak shape and FWHM change as one moves to channels away from the beam axis. As the channel transmission begins to decrease, there is a slight reduction of the peak width. However, for the outer channels where transmission is at the poorest, the peak width increases and the shape of the peak is appreciably modified. The outermost channels generate "volcano-shaped" peaks as a result from the fact that the effective central direction of the channel (the mean direction of the transmitted neutrons) is different from the geometrical center direction of the channel and converges to a different point on the detector plane.

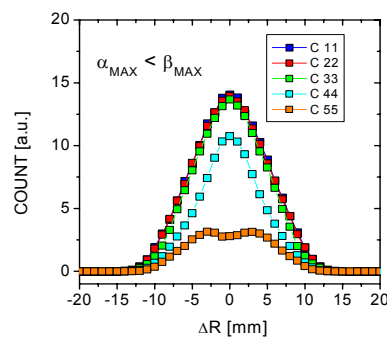


Fig. 3 – Zero width Bragg peaks collected at the detector produced by neutrons transmitted by the channels numbered in Fig. 1.

The work done is summarized in a paper [1], that concludes that the effects observed do not impair in any significant way the usefulness of the device. They should, however, be taken into consideration by the designer.

Development of a Neutron Converging Multichannel Collimator

A.N.Falcão, F.M.A. Margaça, F.G. Carvalho

Objectives

This activity started last year and aims at constructing the first prototype of a CMC with variable geometry to be used for small-angle neutron scattering. Particular attention was drawn to the neutron absorbing material to use. Materials with different compositions were developed and tested.

Results

The computer simulation that guided the initial design of the apparatus was upgraded to account for total reflection processes occurring at the ribbon surface. Results showed that, if the critical angle for total reflection was above a few mrad, there would be some contamination of the direct beam, capable of affecting in a negative way the resolution function. Following the guidelines of the program, materials produced from mixtures of an epoxy resin which Gd_2O_3 and Ti powders in different concentrations were prepared and studied. The experimental tests performed confirmed that the ribbons produced have, in all cases, adequate mechanical and neutron absorbing properties.

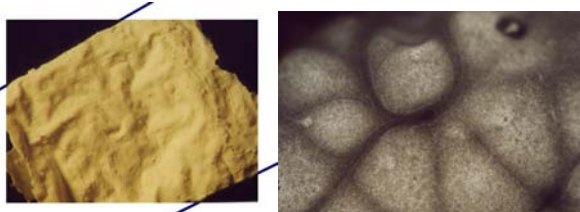


Fig. 1 - Sample of a curled strip of layers of aggregated gadolinium oxide grains obtained by incineration, and, magnification of a fragment of the strip.

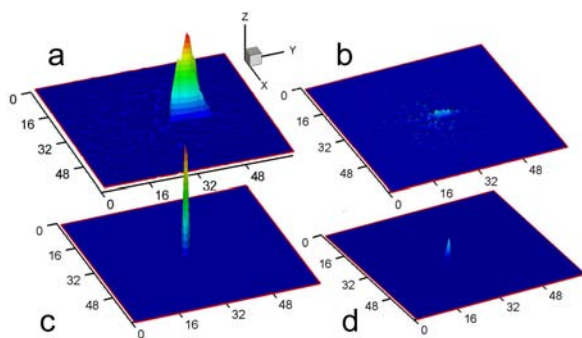


Fig. 2 - Scattered intensity recorded at a PSD placed perpendicular to the incoming beam. Samples prepared with Ti (b, d) and without Ti (a, c). Rough surfaces (a, b) and smooth surfaces (c, d).

Scattering experiments were performed on samples with different compositions. The results obtained show that the quality of the surface determines largely the angular spread of the reflection, and that the

addition of Ti reduces considerably the probability of total reflection.

Working life times of the ribbon were estimated to be larger than 50 years for a high neutron flux reactor and continuous work, following results of a computer simulation study of the dose deposited (~ 0.84 kGy year⁻¹), and an irradiation experiment in a ^{60}Co irradiation unit where severe working conditions were simulated.

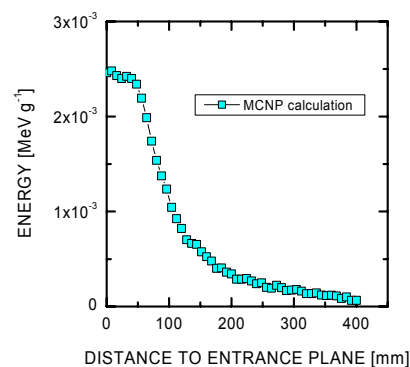


Fig. 3 - Energy per unit mass and per incident neutron deposited in the CMC strips as a function of the distance to the entrance plane of the device.

The design of a first prototype was reviewed and construction of the new version is under way.

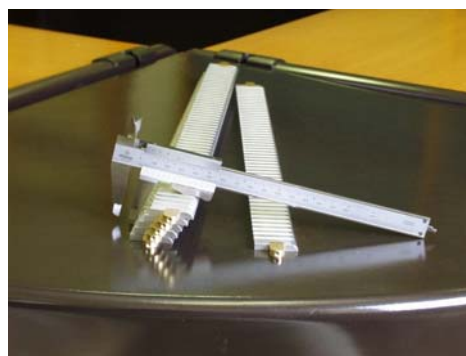


Fig. 4 - View of the aluminium rulers that support the thin ribbons. These are an essential part of the supporting frame of the CMC presently under construction.

Published, accepted or in press work

1. A.N. Falcão, F.M.A. Margaça and F.G. Carvalho, A contribution to the Practical Implementation of a Variable Geometry Converging Multichannel Collimator for SANS, *Jour. App. Crystallography* (2002). Accepted.

Contribution to the Development of the SANS Technique

F.G. Carvalho, A.N. Falcão, F.M.A. Margaça, J.F. Salgado and L. Csér¹

Objectives

To contribute to the study and design of a new generation of SANS instruments: smaller, modular, with enhanced capabilities and low servicing requirements.

Results

The work is sponsored by the IAEA and involves collaboration with the Budapest Neutron Centre. During 2002 progress has been made on a) the implementation of the converging multichannel collimation concept in actual SANS instruments and b) the assessment of the installation of a neutron guide tube to feed neutrons to the ITN SANS facility.

The ITN team had shown before that the principle of converging multichannel collimation, CMC, applied to SANS has significant advantages over the one-channel collimation as regards count rate at constant resolution and accessed Q-range. Now a detailed study of the performance of a SANS instrument with converging multichannel collimation [1] was carried out that enabled to lay down guidelines for the implementation of such collimator assemblies in actual instruments.

It was found that for a real collimator with a finite wall thickness there is an optimum number of channels that maximise the count rate to which also corresponds a certain collimator length. When the sample-to-detector distance is changed for a new instrument resolution, the collimator length should be changed to stay at the maximum of the count rate.

Now, it is unlikely for technical reasons to succeed in implementing a 3D CMC variable in length. Long 2D (X,Y) variable CMC units are not easy to build either. So the possibility of using an association of fixed length 2D CMC units to cover the required range of sample-to-detector distances was explored.

The overall instrument performance thus obtained is considerably improved over the conventional SANS arrangement with single-channel collimation.

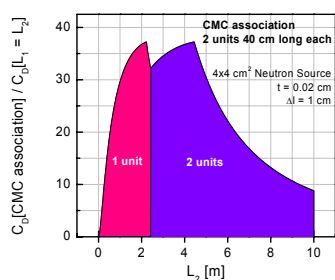


Fig. 1 - Relative count rate gain over the conventional SANS facility, for the same resolution and Q range.

The feasibility and usefulness of installing a neutron beam guide at the ITN SANS facility has been studied. The BNC team performed a simulation study involving a series of Monte Carlo calculations. The results discussed at ITN showed that although there is no expectable gain in flux at the sample the background consisting of higher energy neutrons and gamma rays will be greatly diminished.

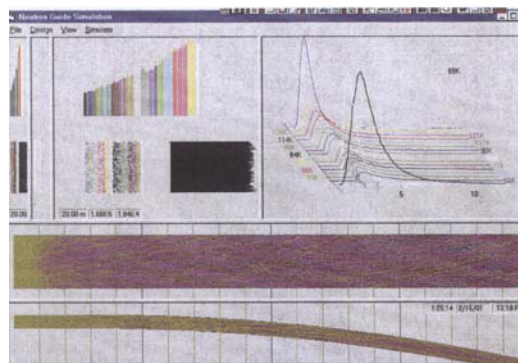


Fig. 2 - Simulation work on neutron guide for ITN.

For the single channel collimation case, a relatively short neutron bender using multilayer supermirrors separated from the neutron velocity selector by a length of straight guide should be an adequate arrangement. The single channel collimator would be placed after the neutron bender extending until the sample position.

In case a CMC is used, an angular divergence at the guide exit of $\sim 1^\circ$ will allow to take full advantage of the converging collimator. However even if the divergence is one half of that value it will be advantageous to use the CMC in terms of improved gain in flux. The results obtained so far by the partners relative to the performance of different neutron guide arrangements are a good basis for continuing the investigation into optimum beam tailoring under the space and source constraints existing at the RPI research reactor.

Published, accepted or in press work

1. F.M.A. Margaça, A.N. Falcão and F.G. Carvalho, Guidelines for the implementation of converging multichannel collimation in a specific SANS facility, *Jour. Applied Crystallography* (2002).
2. A.N. Falcão, F.M.A. Margaça, J.F. Salgado and F.G. Carvalho, A contribution to the development and practical utilization of the small angle neutron scattering technique (II), Paper presented at 2nd *Research Co-ordination Meeting of the IAEA CPR* "Development and practical utilization of the small angle neutron scattering applications", IAEA Headquarters, Vienna, Aug. 20-23, 2002.

¹ Research Institute for Solid State Physics and Optics and Budapest Neutron Center, Budapest, Hungary

Neutron Beam Facilities at the Portuguese Research Reactor

A.N. Falcão, F.M.A. Margaça, J. Neves, J.F. Salgado and F.G. Carvalho

Objectives

Work in progress aims at implementing locally a basis for the application of neutron scattering techniques for research and training available to external users, simultaneously improving the RPI reactor utilisation.

Results

Testing of the new two-axis neutron diffractometer (DIDE) was completed. The instrument is equipped with a “banana” multidetector donated by the French CEA, and a 24' mosaicity HOPG focusing monochromator. Flux at the sample is $\sim 8 \times 10^5 \text{ cm}^{-2} \text{ s}^{-1}$. In 2003 it will be necessary to complete the investment already done by purchasing the equipment necessary to condition the sample environment (furnace and cryostat).

Suppression of higher order components in the monochromated beam was satisfactorily achieved by using a 40mm-thick graphite filter (Fig.1). Also, spurious counting from electromagnetic interference observed in early tests has been essentially eliminated.

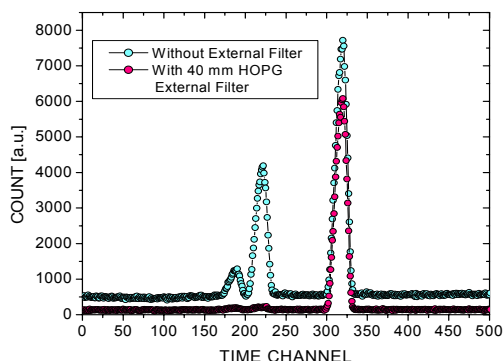


Fig.1 Neutron TOF distribution in the monochromated beam of the DIDE diffractometer. Suppression of 2d and 3d order reflections is patent

A light-weight chopper designed at ITN was constructed in the local workshops and successfully tested (Fig.2). The device is intended for the determination of the distribution of neutron energies in thermal neutron beams. Pulse width and frequency are remotely controlled by computer.

A small angle neutron scattering instrument (EPA) designed at ITN is currently being installed in the reactor tangential channel. The source is a thin disk-shaped light water scatterer placed near the reactor core (thermal flux at the scatterer about $10^{13} \text{ cm}^{-2} \text{ s}^{-1}$).

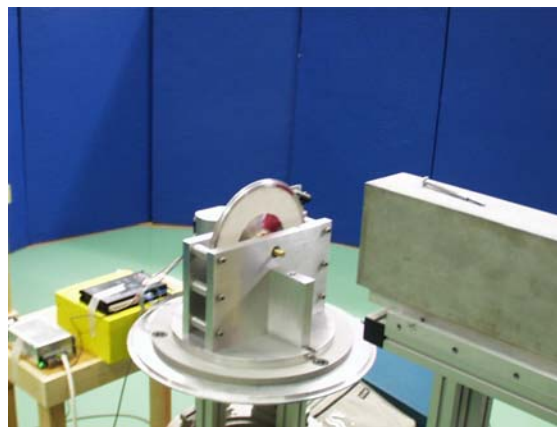


Fig.2 View of the light-weight neutron chopper for the determination of neutron energy distributions in thermal neutron beams. Pulse width and frequency are remotely controlled by computer

Installation of the out-of-pile components proceeded during the year and first measurements of beam profiles after the velocity selector were made. Unfortunately a malfunction of the in-pile shutter led to the interruption of the installation work that is to continue as soon as a new shutter that has been designed is operational.

Beam profile results confirm that it will be necessary in the near future to programme the implementation of a neutron guide section in the beam line of EPA in order to improve the signal to noise ratio at the higher usable wavelengths. A co-operation with the Budapest Neutron Center on this question already exists and is sponsored by the International Atomic Energy Agency, in Vienna. Upgrading of the ^3He detector banks of the neutron TOF diffractometer ETV is almost completed.



Fig.3 EPA - detector housing and PSD detector on its platform

Nuclear Instruments and Methods



Nuclear Instruments and Methods

José Salgado

The activity of the group is focussed in different lines: *Computational Physics, Experimental work and Instrumentation and Technical Assistance.*

Calculation of radiation fields

The calculation of radiation fields has a broad range of applications, such as medical physics, material processing, radiosterilisation, cross-section measurements, reactor irradiation and radiation transport in superconductors. The aims of this work are:

- Calculation of neutron resonance self-shielding factors in different materials and different geometries;
- Interpretation of its dependence on the physical and nuclear parameters of the studied materials;
- Calculation of multi-scattering effects for corrections in neutron cross-section measurements;
- Determination of the neutron and neutron-induced gamma ray backgrounds in a second experimental area for cross section measurements at the CERN nTOF experiment;
- Calculation of gamma doses in samples with complex shapes irradiated in the Gamma Irradiation Facility (UTR) using the voxel mode;
- Studies of the response of the superheated droplet detector - SIMPLE detector, under monochromatic neutron irradiation using filtered neutron beams of energies 25 keV (Fe+Al+S), 54 keV (Si+S) and 149 keV (Si+Ti).

Electrical Discharges on Environment and Material Processing Applications

The main objectives of this activity are:

- Study and modelling of the formation of carbon clusters obtained by laser ablation. These carbon clusters are the precursors for the formation of nanotubes;
- Preparation of reliable base data needed to numerical modelling of discharges. For this purpose

automatic fitting methods for the electron collision cross-sections have been study;

- Development and characterisation of a dielectrical barrier discharge chamber for the decomposition of volatile organic compounds, VOCs, resulting from the treatment of plastic wastes at atmospheric pressure.

Instrumentation and Technical Assistance

The main objectives of this activity are the development of equipment for internal groups, fabrication of equipment for specific applications and assistance to industrial companies and scientific institutions as well as technical consulting.

The technical assistance takes mainly the forms of specialized consultant engineering advice, installation of nuclear gauges, including calibration maintenance and repair and recharging of gauges with imported sources.

Co-operation with other institutions

The Group is involved in the n_TOF collaboration, a consortium of 40 laboratories in Europe and USA. There are also collaboration with the Centro de Física Nuclear, Universidade de Lisboa and Instituto de Pesquisas Energéticas e Nucleares, IPEN - S. Paulo, Brasil.

The work on decomposition of VOCs is included in the project "Treatment and valorisation of plastic waste", involving collaboration with the Department of Automation and Electric Engineering, Instituto Superior de Engenharia de Lisboa and Sociedade Ponto Verde.

The study of laser ablation carbon plasmas is included in a common project with the Research Laboratory for Materials and Environmental Chemistry, CRC, Hungarian Academy of Sciences.

Nuclear Instruments and Methods

Research Team

Researchers

- J. SALGADO, Coord. Researcher, Group Leader (85%)
- F. G. CARVALHO, Coord. Researcher (10%)
- J. MANTEIGAS, Auxiliary Researcher (70%)
- I. F. GONÇALVES, Auxiliary Researcher
- J. NEVES, Auxiliary Researcher (80%)
- C. CRUZ, Auxiliary Researcher
- N. PINHÃO, Auxiliary Researcher

Collaborators

- K. KUTASI, MSc, fellowship, Sociedade Ponto Verde
- A.G. BAPTISTA, ISEL, Secção de Electrónica Industrial

Funding (€)

Research Projects:	9 290
Services:	17 993
Total:	27 283

Publications

Journals:	4 and 1 in press
Proceedings:	2 in press
Conf. Communications:	6
Other publications:	1

Students

- M.S. SILVA, last year student, Mathematics degree, FCT/UNL.
- T.L.P. SANTOS, last year student, Mathematics degree, FCT/UNL.
- S.R.P.V. ILDEFONSO, last year student, Mathematics degree, FCT/UNL

Technicians

- T. Jesus, laboratory technician
- N. Inácio, laboratory technician (85%)

Neutron resonance self-shielding factors

I.F. Gonçalves, J. Salgado, E. Martinho

Objectives

The aims of this work are (a) to calculate the neutron resonance self-shielding factor in different materials and different geometries, (b) to interpret its dependence on the physical and nuclear parameters of the studied materials.

Results

The work carried out during this year consisted in the calculation of neutron self-shielding factors of materials submitted to different neutron fields: $1/E$ isotropic field, $1/E$ collimated beam and monoenergetic collimated beam. The calculations were performed with the MCNP code [1-5]. Figure 1 shows the energy dependent self-shielding factor, $G(E,t)$ of ^{232}Th irradiated by a monoenergetic collimated neutron beam. It is important to note that the values of $G(E,t)$ are greater than 1 for energies above the resonances.

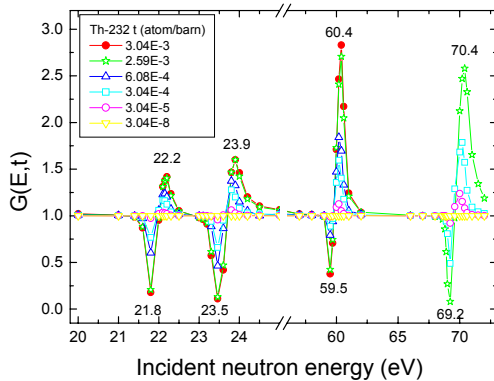


Fig. 1- Energy self-shielding factor of ^{232}Th for different neutron energies and thicknesses

As for foils immersed into a $1/E$ neutron field, a dimensionless variable can be introduced that converts the dependence of $G(E,t)$ on the nuclear and physical parameters into an unique curve valid for all nuclides. Figure 2 compares self-shielding factors of foils irradiated by monoenergetic and $1/E$ collimated neutron beams with the resonance self-shielding factor for a $1/E$ isotropic neutron field.

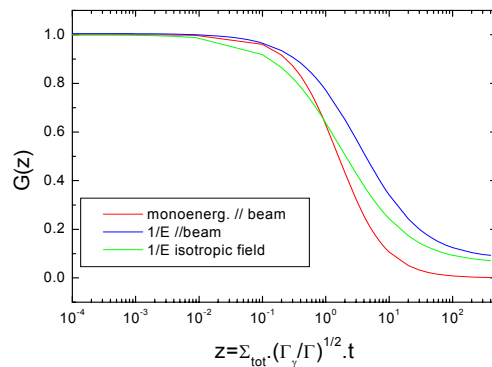


Fig. 2 – Comparison of universal curves calculated in the following conditions: *i*) collimated monoenergetic neutron beam (red); *ii*) $1/E$ collimated neutron beam (blue); *iii*) $1/E$ isotropic neutron field (green).

Published, accepted or in press work

1. I.F. Gonçalves, E. Martinho and J. Salgado, Monte Carlo calculation of epithermal neutron resonance self-shielding factors in foils of different materials, *Applied Radiation and Isotopes* 56/6, 945-951 (2002).
2. E. Martinho, I.F. Gonçalves, J. Salgado, Universal curve of epithermal resonance self-shielding factors in foils, wires and spheres, *Applied Radiation and Isotopes* (in press).
3. J. Salgado, I.F. Gonçalves, E. Martinho, Epithermal neutron self-shielding factors in foils for collimated beams, *Applied Radiation and Isotopes* (submitted).
4. J. Salgado, E. Martinho, I.F. Gonçalves, Neutron self-shielding factor of a group of isolated resonances, *5th International Topical Meeting on Industrial Radiation and Radioisotope Measurement Applications, Bologna, Italy*, (sent for publication in *NIM-B*) (2002).
5. I.F. Gonçalves, E. Martinho, J. Salgado, Extension to cylindrical samples of the universal curve of resonance neutron self-shielding factors, *5th International Topical Meeting on Industrial Radiation and Radioisotope Measurement Applications, Bologna, Italy*, (sent for publication in *NIM-B*) (2002).

Multi-scattering corrections for measurement of (n, γ) cross sections

J. Salgado, E. Martinho, I.F. Gonçalves

Objectives

Multi-scattering must be taken into account in the interpretation of (n,γ) cross-section measurements. The corrections depend on the sample thickness and cross-sections.

Results

The effect of multi-scattering in thick samples of ²³²Th, ¹⁹⁷Au and ⁵⁶Fe was studied using the MCNP code. A parallel monoenergetic neutron beam impinging perpendicularly to a foil with 0.75 cm radius and thickness varying from 0.00001 to 10 mm was simulated. The capture yield, $Y(E)$, i.e. the fraction of neutrons incident on a sample and undergoing the (n,γ) reaction is given by:

$$Y(E) = \left(1 - e^{-n\sigma_t(E)}\right) \frac{\sigma_\gamma(E)}{\sigma_t(E)} \times \chi(E)$$

The factor between brackets takes into account the auto-absorption in the foil. The effects of multi-scattering in the sample are taken into account by the factor $\chi(E)$. Neglecting multi-scattering events $\chi(E)$ must be equal to 1.

Figure 1 shows $\chi(E)$ for ²³²Th [1].

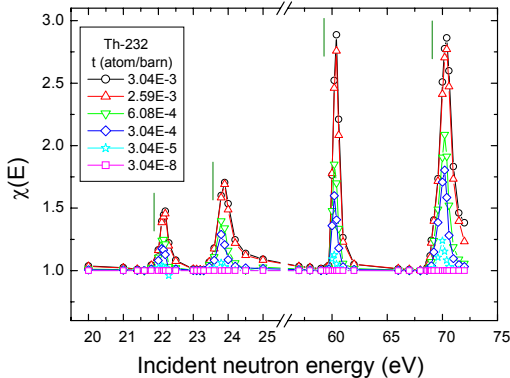


Fig. 1 - $\chi(E)$ for ²³²Th

$\chi(E)$ is greater than 1 particularly for energies above E_{res} .

Figure 2 shows the capture yield for ¹⁹⁷Au. In the case of ¹⁹⁷Au, for $t > 6 \times 10^{-5}$ atom/barn (0.01 mm) the yield

increases, attaining a value of 10% for $t > 6 \times 10^{-4}$ atom/barn (0.1 mm).

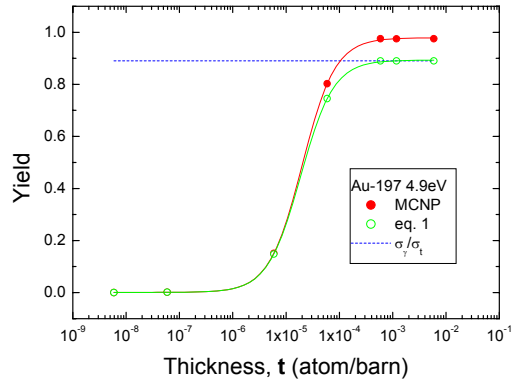


Fig. 2 - Capture yield for ¹⁹⁷Au.

Figure 3 shows the reaction rate of neutron impinging on the foil with energy $E_0 = 1.20$ keV depicted as a function of the absorbed neutron energy ($E \neq E_0$) and of the foil thickness. Most of the incident neutrons with $E = 1.20$ keV are captured with energy 1.15 keV (they have suffered one collision before capture).

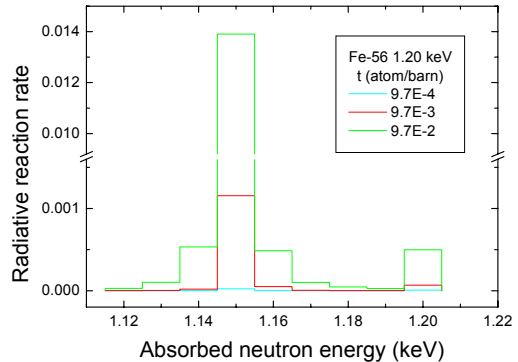


Fig. 3 - Spectra of the radiative reaction rate as a function of the absorbed neutron energy

Published, accepted or in press work

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cross-sections, 4th n_TOF Theory Group Meeting, ITN Lisbon, 14 December 2002.

Neutron and gamma background in a second experimental area for fission measurements at nTOF (CERN)

D. Cano-Ott¹, F. Calviño², G. Cortés², I.F. Gonçalves, E. Gonzáles¹, A. Poch², P. Vaz

Objectives

Determination of the neutron and neutron-induced gamma ray backgrounds in a second experimental area for fission measurements at the CERN nTOF experiment.

Results

A view of the geometry of the second experimental area of the nTOF experiment is shown in Figura 1.

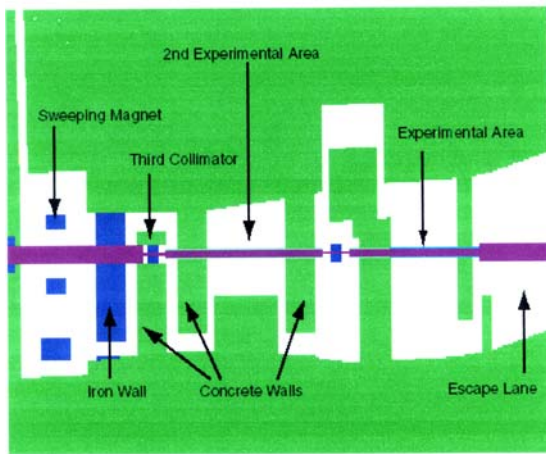


Fig. 1 – A view of the geometry of the second experimental area of the nTof experiment

The neutron and neutron-induced gamma ray backgrounds have been calculated at several regions of interest along the tunnel [1]. It had been verified that the contribution of the spallation process to the secondary area in terms of neutron and neutron-induced gamma ray backgrounds can be neglected with the improved version of the shielding. Figure 2 shows the neutron

Published, accepted or in press work

1. D. Cano-Ott, F. Calviño, G. Cortés, I.F. Gonçalves, E. Gonzáles, A. Poch, P. Vaz, Results of the Monte Carlo Simulation of the neutron and gamma background in a second experimental area for fission measurements at nTOT, Ciemat Report (2002).

background fluence observed by a fission detector in a region 20 cm above and 80 cm below the pipe and including the neutron beam. The neutron background outside the beam pipe was found to be 5 orders of magnitude below the neutron beam value.

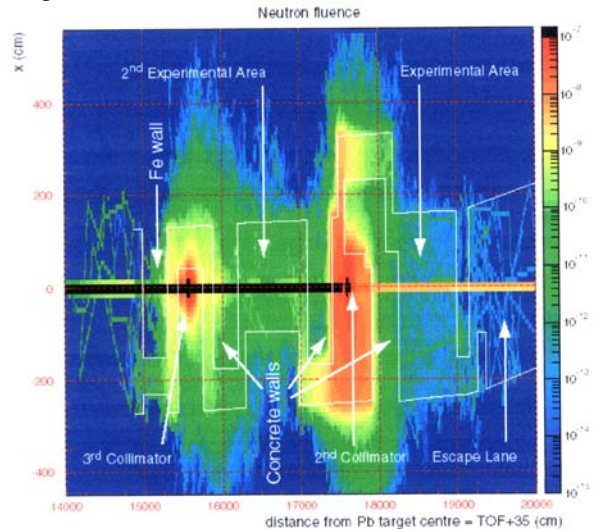


Fig. 2 – Neutron fluence distribution between 140 and 186 m from the centre of the PB target.

The neutron-induced gamma ray background level at the main experimental area is not affected by the 2nd experimental area.

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² Univerdidade Politécnicada Cataluña - Barcelona

Monte Carlo method – a useful tool to understand the transport and interaction of radiation with matter, the energy deposited phenomenon and the device responses.

C. Oliveira, J. Salgado, T. Girard¹, H. Yoriyaz², I. F. Gonçalves, L. M. Ferreira

Objectives

The objectives of this project are:

a) Calculation of gamma doses in samples with complex shapes using the voxel mode, irradiated in the Gamma Irradiation Facility (UTR).

b) A study of the response of the superheated droplet detector- SIMPLE detector, under monochromatic neutron irradiation using filtered neutron beams of energies 25 keV (Fe+Al+S), 54 keV (Si+S) and 149 keV (Si+Ti).

Results

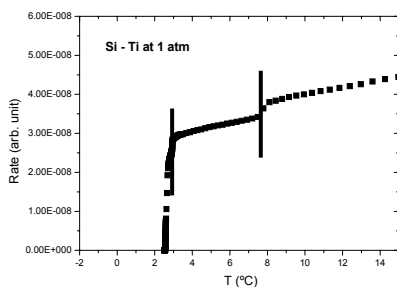
The simulations were carried out by using the MCNP code.

A new method for the design of the irradiated samples was introduced. It is based on CT image data. The data are read by a specific code transforming it into a Monte Carlo input file. At each element in the CT-based matrix, a Monte Carlo voxel is created. The voxel dimensions depend on the CT image resolution. It allows the calculation of doses at each voxel or at a zone (union of voxels). It also allows the knowledge of dose distributions anywhere without extra (more) definitions of surfaces or volumes [1].

2. The use of composite filters in calibration studies of superheated droplet detectors (SDDs) is seriously qualified by the detector's temperature-stabilizing water bath, and by the hydrogenated gel of the detector construction itself. In the three filter cases considered, the simulations indicate that the effect of the detector is to render an essentially identical beam on target comprised on only ~28 and ~50 keV neutrons [2-4].

The sharp cutoffs in the simulation spectra result from the primarily epithermal neutron spectrum adopted as input to the simulations.

Simulations results



Experimental results

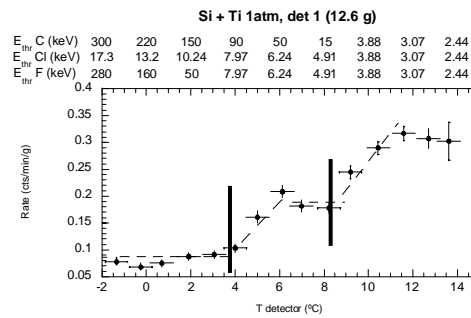


Fig.1- Simulations and experimental results, for Si+Ti filter, manifest kink at same temperature, ~2.5 and 8.0 °C.

Published, accepted or in press work

1. C. Oliveira, H. Yoriyaz, M.C. Oliveira, L.M. Ferreira, Monte Carlo simulation for dose distribution calculations in a CT based phantom at the Portuguese Gamma Irradiation Facility, *5th International Topical Meeting on Industrial Radiation and Radioisotope Measurement Applications, Bologna, Italy*, (sent for publication in *NIM-B*) (2002).
2. C. Oliveira, F. Giuliani, T.A. Girard, J.G. Marques, J. Salgado, J. Collar, T. Morlat, D. Limagne, G. Waysand, MCNP Optimization of filtered neutron beams for calibrations of the SIMPLE detector, *5th International Topical Meeting on Industrial Radiation and Radioisotope Measurement Applications, Bologna, Italy*, (sent for publication in *NIM-B*) (2002) (Oral presentation).
3. JI Collar, TA Girard, F. Giuliani, D. Limagne, J.G. Marques, HS Miley, T. Morlat, C. Oliveira, J. Puibasset and G. Waysand, SIMPLE: Present Status and Improvements. *Proc. XIIIeme Rencontres de Blois: Frontieres of the Universe*. (2002) (in press)
4. JI Collar, TA Girard, F. Giuliani, D. Limagne, J.G. Marques, HS Miley, T. Morlat, C. Oliveira, J. Puibasset and G. Waysand, The Status of SIMPLE in 2002, *Proc. DARK2002* (2002) (in press).

¹ Centro de Física Nuclear, Universidade de Lisboa

² IPEN - S. Paulo, Brasil

Electrical Discharges for Environment Applications and Evaluation of Electron Collision Cross Sections

N.R. Pinhão, K. Kutasi¹, M.S. Silva², T.P Santos², S.P.V. Ildefonso², A.G. Baptista³

Objectives

The main objective of this project is the use of cold plasmas on environment applications. Currently a dielectrical barrier type discharge is been optimized for the decomposition of volatile organic compounds resulting from the treatment of plastic wastes.

As research and development in this domain is also heavily based on numerical modelling one goal has been the preparation of reliable base data needed to numerical modelling of the discharges. For this purpose automatic fitting methods for the electron collision cross sections have been study.

Results

Experimental work

- A laboratory dielectric barrier discharge system for gas cleaning, a mass spectrometric gas analysis system for atmospheric pressure sampling and a gas mixture system for controlled admixture of volatile organic compounds have been used to study the decomposition of alcohol on nitrogen and on a H₂/CO/CO₂ gas mixture.

Published, accepted or in press work

1. L.M. Redondo, N. Pinhão, E. Margato, J. Fernando Silva, "Progress on high-voltage pulse generators, using low voltage semiconductors (<1kV), designed for Plasma Immersion Ion

This task is included in the project "Treatment and valorisation of plastic waste"

- A 5kHz, 10kV power supply has been build to supply the above DBD system.



Figure 1 - Detail of the DBD chamber with a discharge on N₂

Numerical modelling

- The application of a genetic algorithm to cross section fitting has continued, now with extended parametrization of the cross sections. The solutions obtained are been studied using a Markov Chain, Monte Carlo algorithm.
- The application of a simulated annealing algorithm to above problem is under development.

Implantation (PIII)", *Surface and Coatings Technology*, **156** (2002) 61-65.

2. N. Pinhão, M.R. Silva "Automatic fitting of electron collision cross sections with a genetic algorithm" ESCAMPIG 16, 2002.

¹ Fellowship, Sociedade Ponto Verde

² Final year student of Mathematics course, FCT/UNL

³ ISEL, Dept. of Electrotechnical and Automation Engineering

Study of Carbon Plasmas Obtained in Laser Ablation of Graphite Targets by Numerical Modelling and LIP Spectroscopy

N.R. Pinhão, L. Nemes¹, A. Keszler¹

Objectives

The main objective of this project is to study and model the formation of carbon clusters obtained by laser ablation. These carbon clusters are the precursors for the formation of nanotubes.

A time dependent, one-dimensional self-consistent model coupling the fluid dynamics, vibrational kinetics, chemical kinetics, radiation transport and Boltzmann equation for the electrons has been developed to study the plume expansion and the species kinetics. This model will be validated comparing the emission spectra from the C_n species (mainly C_2 and C_3) formed in the plume with spectroscopic measurements of the plume resulting from the interaction of a YAG laser with a graphite target in vacuum.

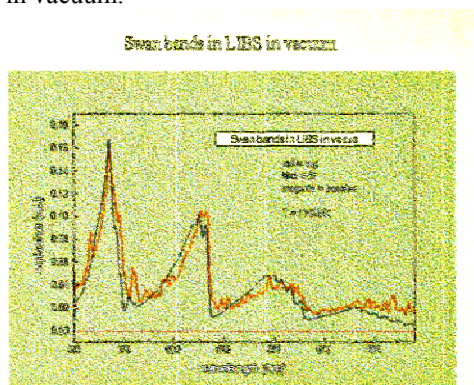


Figure 2 - Measured and simulated C_2 swan band spectra in vacuum

Published, accepted or in press work

1. A.M. Keszler, J.O. Hornkohl, N.R. Pinhão, L. Nemes "Optical diagnostics for the laser-ablation of graphite", *Diamond 2002* (submitted).

Results

Experimental work

- In Hungary graphite LIB spectra have been taken with all three wavelengths of a YAG laser allowing the estimation of the vibration-rotation temperature of the C_2 radical. The electron densities and kinetic temperatures have been calculated from carbon line Stark broadenings and as well as relative intensities of carbon lines.

Numerical modeling

- The Portuguese side of the cooperation has been extending the PLASMAKIN (chemical kinetics) and BOLTZKIN (electron kinetics) software libraries to handle photon-atom reactions and photon-electron and electron-electron collisions. Simultaneously electron collision cross section data for carbon have been collected, and kinetical and spectroscopic data have been gathered and evaluated for the C_n carbon molecular species.

¹ Research Laboratory for Materials and Environmental Chemistry, CRC, Hungarian Academy of Sciences.

Technical Assistance in the Field of Engineering Applications of Radiation and Radioisotopes

J.B. Manteigas, J. Neves, C. Cruz, N. Pinhão, F.G. Carvalho, J. Salgado

Objectives

The main objectives are the development of equipment for internal groups, fabrication of equipment for specific applications and assistance to industrial companies and scientific institutions as well as technical consulting.

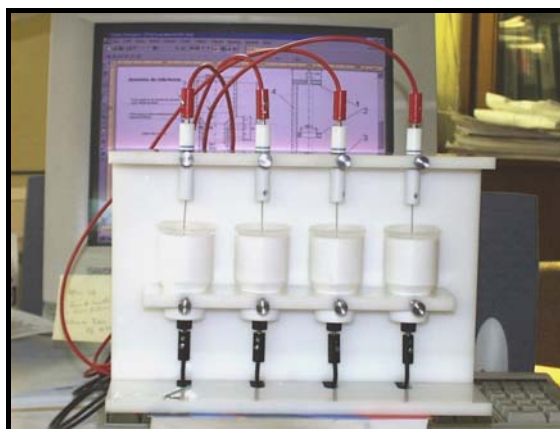
- (i) Technical and scientific participation in the n-TOF (PS213) experiment at CERN.
- (ii) Support in electronics for nuclear instrumentation (development and maintenance) to DPSR, Physics Sector, UTR and Reactor.
- (iii) Technical and scientific collaboration in testing equipment for the “CERN Large Hadron Collider” under contract agreement between ITN and VELAN.

Results

A summary of the more relevant work carried out is:

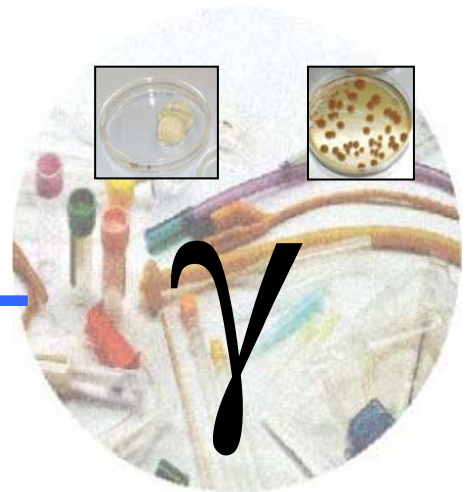
Design and construction of the RPI (Portuguese Research Reactor) prototype-simulator, controlled by a personal computer. The design involved three parts, mainly: electro mechanics (several step motors), controlling and driving electronics and software.

A summary of the more relevant services rendered in 2002 is presented below.



Activity	Quantity	Client	Price* (Euro)
Supply of plating electrode disks	500	ITN/DPRSN	548,67
Laboratory equipment for the determination of radioactive element traces by electrodeposition	1	Clemson Univ. (USA)	1212,25
	1	Neurotech, Inc. (USA)	1298,00
	1	Florida State Univ. (USA)	1126,33
	1	City Corp. for Medical Supply (Jordânia)	1143,00
	1	Centre Nat. Sci. Techn. Nucls. (Tunisia)	989,00
Supply of gamma level detector units	2	CIMPOR/Souselas	1496,38
Supply of gamma level controllers	2	CIMPOR/Souselas	1993,20
Measuring and control of source activities	27	SOPORCEL	2169,77
	2	S.N. – Produtos Longos, SA	573,52
<i>Technical assistance</i>			
- Troxler neutron gauge	1	ISA	148,39
- Detectors	2	PORTUCEL/V.V.Ródão	250,00
- RAD X 100	1	IMAGAIA	127,00
	1	Forças Armadas/Marinha	124,70
- Boltzkin Software Library	1	Chemical Res. Center (Hungary)	1000,00
- Source containers	4	PORTUCEL/Cacia	1446,52
- Gamma level indicators	2	CIMPOR/Cabo Mondego	1746,00
- DENSOLO + 2 control units	1	Esc. Sup. Agrária Beja	600,00
Total Amount (EURO)			17 992,73

Radiation Technology: Processes and Products



Radiation Technologies: Processes and Products

M. Luisa Botelho

The **Radiation Technologies: Processes and Products** activities focus on the development and demonstration of radiation processing applications. Nowadays these activities are closely related to the gamma radiation facility (UTR), whose main applications are the sterilization of medical devices and pharmaceuticals and the decontamination of other products.

The group, therefore, supervises the sterilization and decontamination procedures being carried out at the UTR. This work is carried out in collaboration with **Food, Medical Devices and Pharmaceutical Industries**. The group also develops work with National and International normalization, standardization and certification bodies (IPQ, CEN and ISO). UTR upgrade and maintenance is performed in compliance with ISO 9002 in the perspective of obtaining accreditation.

In order to develop the procedures at UTR, based on the Quality System, studies of dose distribution and determination of Dmin and Dmax are performed taking into account the Safety Assurance Level and the safety of the product.

materials in order to avoid damaging the final product. Copolymerisation, reticulation and other effects induced by radiation are tested (carried out The Group collaborates with several Universities ¹ and Politechnique and Research Institutes ².

New Projects are presently being developed to apply **ionising radiation techniques to Wastewater treatment**.

The Group develops work studying the impact of radiation on micro-organisms and materials such as synthetic and natural polymers.

The main purpose of the **microbiological** work is to develop and implement validation technologies for procedures of inactivation of micro-organisms, mainly by ionizing radiation (e.g.: γ and e-beam). These technologies are based on microbiological studies on the bioburden in/on the products, and aim to improve quality in this field.

Thus, hazard analysis and the control of critical points in the production lines of the studied products are part of the validation studies, carried out for the **Pharmaceutical and Food Industries**. Environmental control in surgical operation theatres at hospitals is also carried out.

In order to improve our understanding of the Radiation Procedures the influence of dose rate and the type of radiation are studied on materials and micro-organisms.

The **Synthetic and Natural Polymers** studies aim to use radiation both for developing new materials and for improving the quality of existing materials. These studies also aim to improve understanding about the interactions between radiation and these (and characterised) to improve the properties and biocompatibility of new materials to be used for biomedical and environmental applications.

This technology could contribute to the remediation and control of contamination by anthropogenically produced pollutants of water resources.

¹ Universidade de Lisboa, Universidade de Coimbra, Universidade de Uppsala –Suécia, Faculdade de Ciências Veterinárias.

² INIA, ESAS.

Radiation Technologies: Processes and Products

Research Team

Researchers

- M.L.BOTELHO, Auxiliary Researcher (Group Leader)
- L.M. FERREIRA, Research Assistant (Responsible for the UTR)

Collaborators

- M.H. GIL (M.H.Casimiro PhD Coordinator)
Department of Chemical Engineering, Faculty of Science and Technology, University of Coimbra.
- R. TENREIRO (S. Cabo Verde PhD Coordinator)
Department of Vegetal Biology, Faculty of Science, University of Lisbon.
- M.J. TRIGO, INIA.

Students

- S. CABO VERDE, FCT grant
- M.H. CASIMIRO, FCT grant
- R. CORDEIRO, Training student, ESAS
- L. ALVES, Fellowship, ERASMUS ¹
- A. COSTA, Training student, U.I
- P. MATOS, Training student, ESAS

Technical Personnel

- H.M. MARCOS
- P. PEREIRA
- V. DAMAS
- J. VENÂNCIO
- A. P. MARQUES ²

¹ On leave from University of Uppsala.

² ITN Grant.

Funding (€)

Research Projects:	5 652
Expert Services:	1 174
UTR Services:	12 230
Total:	19 056

Publications

Journals:	3	and 2 in press
Proceedings:	1	
Conf. Communications:	4	
Other publications:	4	
Theses: Bch	1	

Food Irradiation

S. Cabo Verde, M.L. Botelho, R. Tenreiro¹ and M.J. Trigo²

Objectives

The use of ionising radiation, from radionuclides such as ⁶⁰Co, in conjunction with good manufacturing practices, its well established safety, and the absence of residues creates a solid scientific background for the implementation of radiation processes for specific food products as an effective means to improve safety of our food supply. In this field two projects are being developed:

1 – Application of Ionising Radiation to Eggs – The aim of this project is to develop and validate methodologies for applying irradiation technology to eggs in order to get a safe product, free of pathogenic microorganisms such as *Salmonella* spp. and *Campylobacter* spp..

2 - Application of Ionising Radiation to Vegetables – The aim of this project is to evaluate the effectiveness of irradiation on microbial levels as well as on colour, texture and sensorial parameters in some products.

Results

1 – “Application of Ionising Radiation to Eggs” – Techniques and methods were validated for the determination of total bioburden in/on eggs belonging to five stage chicken age, to find out whether chicken age influences egg bioburden. Results suggested that the egg bioburden is between 10³ to 10⁶ cfu/egg for the shell and <50 to 10⁴ cfu/egg for white and yolk. Based on an Approximate Test of Equality of Means applied to bioburden values, the results highlight that there is no significant differences between the bioburden values of the five stages of chicken age (p<0,05). Thus, we can assume an average bioburden value of 2x10⁵ ± 3x10⁴ cfu/egg for the shell eggs analysed. The natural contaminants of eggs were morphologically characterized; two major morphological types were found, gram-positive rods, catalase positive (42%) and gram-positive cocci, catalase positive (40%). Until now, neither microorganisms of *Salmonella* or *Campylobacter* genus were found in the samples. HACCP studies (in feed, drinking water, environment and faeces samples) indicated that the feed should be assumed as a critical point.

Initial irradiation studies are being developed; dosimetry studies were carried out in a gamma facility (UTR, ITN) and in an e-beam facility (Centro Oncológico, Carnaxide) to find the best geometry and dose rate for irradiation process. Eggs were artificially

Published, accepted or in press work

1. S. Cabo Verde, H. Marcos, A. Marques, M.L. Botelho, R. Tenreiro. “Contaminantes Naturais dos Ovos e suas Possíveis Origens” presented in Food

contaminated, externally and internally, with control strains of *Salmonella typhimurium* (CECT 443) and *Campylobacter coli* (DSM 4689^T, Portugal) and irradiated in the gamma facility at sub-lethal doses (0.2 to 1 kGy) with a dose rate of 1.0 kGy/h. The D_{values} for each strain, for shell and yolk+white egg, were calculated based on the number of survivors: 0.31 ± 0.06 kGy for shell; 0.26 ± 0.04 kGy for yolk+white contaminated artificially with *Salmonella typhimurium* [Fig. 1] and 0.21 ± 0.02 kGy for shell; 0.178 ± 0.001 kGy for yolk+white contaminated artificially with *Campylobacter coli* [Fig. 1].

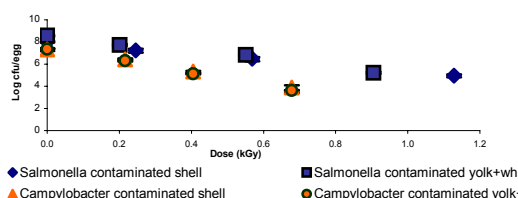
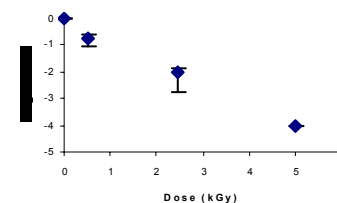


Fig. 1 – Inactivation curves of *Salmonella typhimurium* (n = 144) and *Campylobacter coli* (n = 72) control strains.

The D_{value} of total natural shell contaminants of a sample of eggs was found to be 1.29 ± 0.02 kGy for the total micro flora of the shell Fig. 2. The resistant and similar non-resistant strains are being morphologically characterized and molecular methods will be applied to these bacteria to confirm their genetic similarity.

Fig. 2 – Inactivation curve of total natural contaminants of



shell eggs (n = 72).

The viscosity, physical and chemical properties and the nutritional properties of eggs, before and after irradiation in the γ facility, are being studied by two training students (Escola Agrária de Santarém).

2 - “Application of Ionising Radiation to Vegetables” – Several samples of minimal processed vegetables were irradiated in the gamma facility. Organoleptic and microbiological studies are being developed.

Protection 2002, February, Monte da Caparica, Portugal.

¹ Departamento de Biologia Vegetal, Faculdade de Ciências de Lisboa, Portugal

² Instituto Nacional de Investigação Agrária

Expert Services

S. Cabo Verde, M.L. Botelho

Objectives

- a) This field aims to provide specific service related irradiation/sterilization technology and microbiology for pharmaceutical industries and has built a bridge between the industry and the scientific research. Thus microbiological study on Mucoflux syrup was carried out at the request of Delta Laboratories.
- b) As an internal service, at the request of the ITN, the microbiological quality of the drinking water of all ITN Departments was monitored.

Results

- a) The Membrane Filtration technique was validated as a substitute method for the Incorporation technique (used by the Delta Laboratories) to calculate the total mesophilic bioburden of the Mucoflux syrup. The Membrane Filtration results were reproducible and in agreement with the Incorporation technique. The average bioburden calculated for the 150 ml samples flasks studied was 8 ± 2 cfu/ml and for the 60 ml flasks samples was 2 ± 1 cfu/ml. The natural contaminants obtained were morphologically characterized and the most common ones were identified. The most frequent microorganism in the 150 ml and 60 ml syrup flasks analysed (94% and 92% respectively), was characterized as gram-negative rods, oxidase positive and identified as *Pseudomonas cepacia*, Fig. 1. Neither this microorganisms or the others isolated from the syrup flasks don't are referred to in the Pharmacopée Européenne as being potentially harmful.

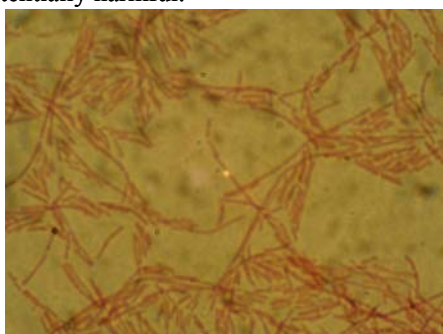


Fig. 1 – Gram stain of the most frequent strain identified as *Pseudomonas cepacia*, isolated from the Mucoflux syrup samples.

- b) The experimental procedure was based on the “Standard Methods for the Examination of Water and Wastewater – 20th Edition”. The detection of microorganisms of the coliform group was carried out using the Most Probable Number technique, the detection of potentially pathogenic bacteria such as *Salmonella* spp. (ISO 6579) and the determination of total mesophilic bioburden was carried out using the Direct Plating and Membrane Filtration Techniques. From the 17 samples analysed none of them have presented detectable contamination by coliforms or *Salmonella* spp.. The hygieno-sanitary quality of the studied water was in conformity with the recommended values by law (Decreto-lei nº236/98 1of August). All the samples presented total bioburden values below the maximum value recommended by law. In the entire sample totality it was found six different types of colonies were found, the most frequent (76%) was characterized as gram-positive cocci, probably belonging to the *Staphylococcus* genus. 18% of the colonies were gram-negative rods from the *Enterobacteriaceae* family. These results do not present any sanitary gravity, so microbiologically, the ITN water is suitable, for drinking.

Activity	Quantity	Client	Price (Euros)
HACCP	2	Delta Lab.	933
Project Audi	1	AdI	300
Water Analysis	1	ITN	250
Total			1483

Published, accepted or in press work

1. S. Cabo Verde, A. Marques, M.L. Botelho. Estudo Microbiológico do Xarope Mucoflux. *External report* to Delta Laboratories, November 2002.
2. S. Cabo Verde, A. Marques, M.L. Botelho. Controlo Microbiológico das Águas de Consumo do ITN. *Internal report ITN*, April 2002.

Thermal Analysis Evaluation of Mechanical Properties Changes Promoted by Ionising Radiation on Surgical Polymeric Textiles

L.M. Ferreira, M.H. Casimiro, C. Oliveira¹, M.E. Cabeço Silva², M.J. Marques Abreu², A. Coelho³

Objectives

To develop methodologies, involving experimental thermal techniques (DSC and TGA) and data analysis based on materials molecular organisation.

Results

This work is part of a study on the influence of ionising radiation (gamma and e-beam) on the properties and performance of disposable materials and products for hospital protection.

The use of DSC and TGA techniques provide rapid knowledge about the material's behaviour and enable the identification of critical doses since these properties are directly dependent on the materials molecular organisation.

As a consequence of results obtained with the polymeric textiles samples irradiated with gamma

radiation⁽¹⁾, the same experimental and data analysis methodology is being applied on samples irradiated at e-beam facility (IONMED/Madrid).

The most relevant mechanical properties were identified based on the results obtained previously by thermal analysis¹ on gamma irradiated samples. These are *tensile strength* and *elongation* with the fibre material direction.

Mechanical properties of samples irradiated by electron beam (at the same doses obtained with gamma radiation) were performed.

Results point out that after 25 kGy absorbed dose, the mechanical properties of laminate material showed differences after irradiation for the two types of radiation (see the table).

The thermal analysis evaluation is in course.

Table. - Effects on the mechanical properties (tensile strength and elongation) of based material, with type of irradiation (25 kGy) and the fibre material direction. (Data obtained at the University of Minho).

Material		Laminate							
		Fmax (N)		Emax (%)		Fbreak (N)		Ebreak (%)	
		MD	CD	MD	CD	MD	CD	MD	CD
Without Radiation	Average	59,56	9,18	8,94	19,06	57,64	7,07	9,45	22,76
Gamma Radiation	Average	51,68	8,43	7,22	14,56	51,01	7,09	7,54	21,21
	Fo	3,96	2,05	3,46	4,64	5,93	1,29	3,06	2,50
	to	12,29	3,05	9,06	4,78	8,34	0,05	7,54	1,41
		S	S	S	S	S	NS	S	S
Electron Beam Radiation	Average	58,48	8,43	8,19	15,97	47,27	8,71	6,91	23,75
	Fo	2,77	1,54	2,34	1,73	37,50	9,62	62,66	1,46
	to	0,87	1,87	2,17	1,30	2,28	2,81	0,87	0,67
		S	NS	S	NS	S	S	S	NS

LEGEND: S - Significant; NS - Non Significant

Published, accepted or in press work

1. L.M. Ferreira, M.H. Casimiro, C. Oliveira, M.E. Cabeço da Silva, M.J. Marques Abreu, A. Coelho, Thermal Analysis Evaluation of Mechanical

Properties Changes Promoted by Gamma Radiation on Surgical Polymeric Textiles, *Nucl. Inst. and Meth. B*, 191 (2002) 675-679.

¹ Nuclear Instruments and Matter Physics group, ITN

² Dpt. Textil Engineering, University of Minho, 4800-058 Guimarães Portugal

³ FAPOMED, Indústria de Confecção de Produtos Médicos-Cirúrgicos, S.A., 4610-108 Felgueiras, Portugal.

Gamma Radiation Facility (UTR)

L.M. Ferreira, P. Pereira, J. Venâncio, V. Damas

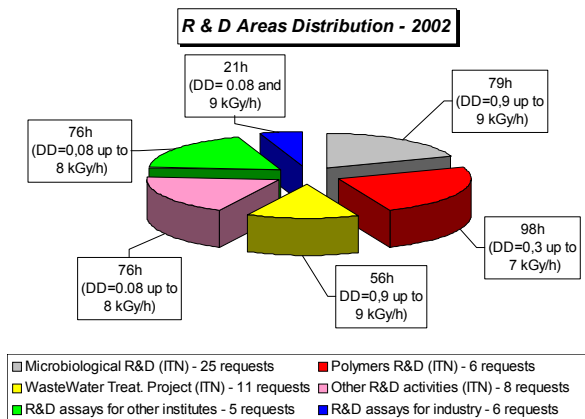
Objectives

To supply gamma irradiation services such as radiosterilisation of medical devices and pharmaceuticals, and radiodecontamination of raw materials, herbs, etc, to institutions and companies.

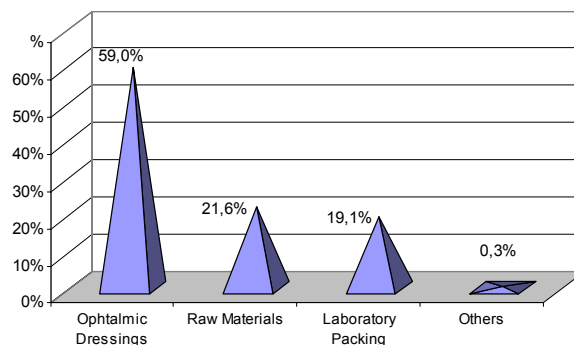
Another of the UTR's objectives is to support R&D projects in the field of gamma radiation interaction with matter (live matter and inert matter).

Results

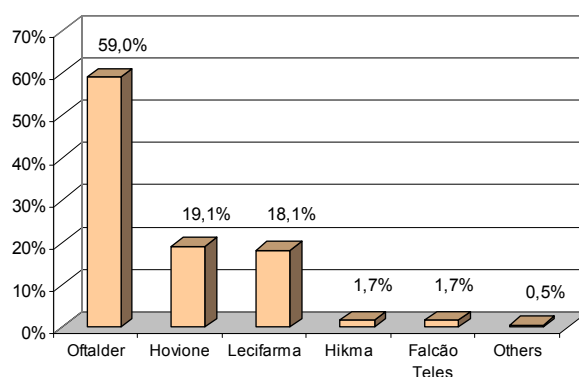
During this year the facility has supplied gamma irradiation services to several national and foreigner companies, namely, *Oftalder S.A.*, *Hovione S.A.*, *Lecifarma Lda*, *Hikma Farmacêutica S.A.*, *Falcão Teles Lda*, *Lorca Marin* (Spain), as well as to a few others organizations of lower economical expression. The R&D irradiation services for internal and external projects were improved in order to make radiation technology transfer a reality. However, due the low activity of the facility (47 kCi in December/2002) the operating limitations have increased. Thus this continuing decrease in performance has lead to very long irradiation times, which turn meeting real industrial needs very difficult. Furthermore, for some products, long irradiation times can be dangerous for the final product's quality.



Industrial Irradiation Services Distribution - 2002



Industrial Clients Occupation - 2002



The figures show a summary of the irradiation services distribution during 2002 (29.Nov.2002).

Published, accepted or in press work

- L.M. Ferreira, M.H. Casimiro, C. Oliveira, M.E. Cabeço da Silva, M.J. Marques Abreu, A. Coelho, Thermal Analysis Evaluation of Mechanical Properties Changes Promoted by Gamma Radiation on Surgical Polymeric Textiles, *Nucl. Inst. and Meth. B*, 191 (2002) 675-679.
- C. Oliveira, L.M. Ferreira, I.F. Gonçalves, J. Salgado, Monte Carlo Studies of the Irradiator Geometry of the Portuguese Gamma Irradiation Facility, *Radiat Phys. Chem.* 65 (2002) 293-295.
- C. Oliveira, Hélio Yoriyaz, M.C. Oliveira, L.M. Ferreira, Monte Carlo Simulation for Dose Distribution Calculations in a CT Based Phantom at the Portuguese Gamma Irradiation Facility, *V Inter. Topic Meeting on Indust. Rad. and Radioisotope Measurement Application*, Jun. Bologna, Italy (2002).
- A.N. Falcão, M. Carrapiço, J.Santos Sousa, F.M.A. Margaça, L.M. Ferreira and F.G. Carvalho, "Investigation of Organic-Inorganic Hybrid Materials Prepared by Irradiation", *Journal of Sol-Gel Science Techn.* 26, (2003) 349-352.

Impact of e-beam and γ - Radiation on the Treatment of Wastewater and Drinking Water: comparative studies

M.L. Botelho, M.C. Freitas, P. Ferreira¹, S.C. Verde, M. Ventura, L. Alves, M. H. Casimiro, A. Costa, P. Matos

Objectives

The long term purpose of this study is to implement the ionizing radiation technology in the wastewater treatment in Portugal. The response of microbiological and chemical factors must be studied according to the type of equipment used and the main characteristics parameters of this equipment in order to plan the future geometry Plant.

Results

Previous studies on two kinds of municipal wastewater effluent samples showed that total wastewater microorganisms inactivation at lower dose rates could be seen to be somewhat less efficient than at higher dose rates. This fact was demonstrated by the determination of D_{10} (90 % of inactivation) of the population irradiated at three dose rates, with an approximate difference of a factor of 10.

Bacteria present a higher D_{value} when irradiated at a Lower Dose Rate than at a Higher Dose Rate. One of the possible reasons is that conditions might be conducive to microbial growth, which could be dependent on the action of irradiation namely if growth kinetics is equal to or greater than inactivation kinetics. Furthermore, repair mechanisms of bacteria could respond more easily.

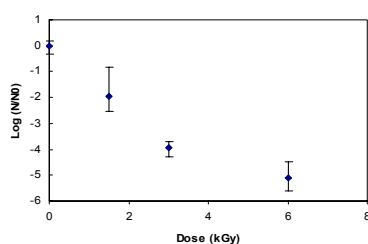


Fig. 1 - Inactivation curve of total microorganisms of pea washing water by ϵ - radiation. (n = 3 each point $\alpha = 0.05$).

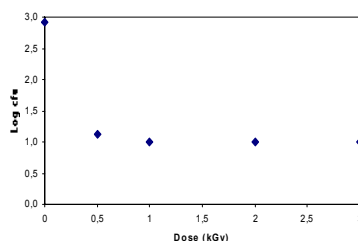


Fig. 3- Inactivation curve of total microorganisms of drinking water by γ - radiation. (n = 6 each point).

From the FTIR analysis one can suggest that there are no substantial alterations in the solid components; the spectra from the filtrate show that there are some alterations (≈ 1420 cm⁻¹) after irradiation which can be related to the presence of either thiol groups or nitrogen groups. INA analysis also indicates that changes in the chemical state of a few chemical elements appear, either by observation of precipitation or dissolution.

Based on these results, the comparative studies of gamma radiation and electron beam impact on the microbiological and chemical dynamics of waste water samples were developed, during 2002, at a similar dose rate (e-beam 1,3; gamma 1,0 kGy/h) and the same geometry of irradiated sample. Studies were carried out using drinking water samples and pea wash water of peas from an Agro-Industrial factory.

Microbiology studies show that the number of cfu/ml obtained, at equivalent doses, in both inactivation processes (γ and e- beam) are not significantly different ($P > 0,05$ - Anova) for the two samples studied.

The graphics presents the inactivation curves for the two kinds of samples irradiated at the accelerator (e-beam) and UTR facility (γ -radiation).

Chemical studies are still underway.

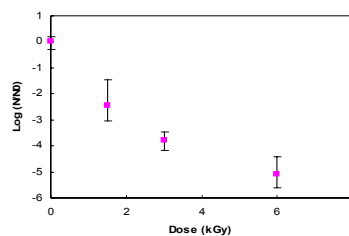


Fig. 2 - Inactivation curve of total microorganisms of drinking water by ϵ - radiation. (n = 3 each point $\alpha = 0.05$).

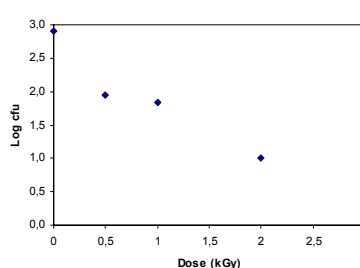


Fig. 4 - Inactivation curve of total microorganisms of drinking water by e- beam. (n = 6 each point).

Published, accepted or in press work

1. M. L. Botelho, M. C. Freitas, P. Ferreira, S. C. Verde, M. Ventura, M.H. Casimiro, L. Alves, A. Costa, P. Matos, presented on workshop

“Wastewater Treatment by Ionizing Radiation”, IAEA, 1st Report CRM, ITN-October 2002.

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