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ALESSANDRO ZUCCHIATTI  
DIRECTOR OF CMAM

Alessandro Zucchiatti has a degree in Physics from the University of Genova (Italy) and a PhD from the University of the Witwatersrand (Johannesburg, South Africa). He directs the CMAM from December 2009, after having been full staff at INFN of Italy from 1975 as well as temporary research associate at the University of the Witwatersrand, research director at CNRS of France and visiting professor at Universidad Autónoma de Madrid. His professional interests go from nuclear physics, to ion beam analysis, to teaching. In both nuclear physics and IBA he has worked in prestigious international research centers, among which the ESRF, the GSI, the LNF, the C2RMF and has been responsible for several research projects. He has given courses at the University of Genoa and the University of the Witwatersrand and has directed international training schools on Archaeometry.

I’m honoured and pleased to open this fifth number of the CMAM activity report series covering the years 2010 and 2011.

The past two years have been marked by the efforts made to provide the CMAM with the most efficient and effective means of running the facility and handling research proposals, so to continue giving our users the best opportunities of doing good science at the Centre. The implantation of a Quality Management System following the ISO 9001:2008 standards makes more effective our way of working and fulfils the commitment of producing the largest possible use of the facilities by the scientific community. The quality we have achieved in the communication with the users, in the evaluation of experiment proposals, in the allocation of shifts and the delivery of beams, strengthens the role of the CMAM as a fully accessible international facility for the analysis and modification of materials. I’m sure our old and new users will appreciate the improvements as well as the fact that they have been implemented while maintaining the amount of available beamtime for both 2010 and 2011 at around 1600 hours. It is a pleasure to pay my personal tribute to the entire staff for the great work they have done to maintain the CMAM at a level of excellence in the infrastructure, the equipments and the access to them. It has not been easy in the present economical situation.

I’m also pleased to acknowledge the relevance of the work done by the scientific division in the past two years and the help of the many who, from outside the CMAM, have contributed to our activities and helped with suggestions and remarks. The scientific collaboration with national and international institutions remains one of the pillars of our policy and I commit myself to improving even further that collaborative environment from which we have obtained so many satisfactory results and the pleasure of working together.

The past achievements make me look at the future with great confidence, ready to take the responsibility of building further on what we have already consolidated to fulfil the obligations of the CMAM towards the UAM and the scientific community of which we are proudly part.

To the readers and colleagues who will receive this report, I would like to renew my appreciation for the attention put on the CMAM. I hope that this report will allow to sample the work done and motivate their support to our activities.

Thank you very much indeed.

Alessandro Zucchiatti  
CMAM DIRECTOR
INTRODUCTION

ABOUT THE CENTRE FOR MICRO ANALYSIS OF MATERIALS

The Centre for Micro Analysis of Materials (CMAM) is a research infrastructure belonging to the Universidad Autónoma de Madrid (UAM). CMAM is the result of a project financed through the FEDER program and managed, since July 1998, by a Technical Committee chaired by Prof. Fernando Aguillo López, assisted by an Advisory Committee formed by outstanding members of the Spanish scientific, cultural and academic community. At the end of 1999 both the accelerator and the building contract were signed. The construction of the CMAM began in September 2002 and was concluded with the installation and commissioning of the accelerator: an electrostatic one with a maximum terminal voltage of 5 MV. The CMAM was officially inaugurated on March 24, 2003, becoming then a university centre dedicated both to in-house materials research with ion beam techniques and to support accelerator based research projects coming from other universities, public research institutes and private companies.

CMAM aims at conducting cutting-edge research in key areas of application of ion beam techniques, such as: Materials Science, Microelectronics and Optoelectronics, Magnetism, Nanotechnology, Environmental Science, Biology and Biomedicine, Nuclear Physics, Energy, Archaeology and Heritage. We also aim at diffusing the ion beam techniques to the scientific and technological communities of Spain as well as to the business community and society as a whole. Finally we are proud of providing advanced technological support to the training, both at national and international level, of technical staff or university students through courses, masters and postgraduate studies.

The experimental equipment consists of the accelerator, several beam lines, specialized for various application areas and several ancillary equipments (micro-analytical techniques, sample preparation). The accelerator, built by High Voltage Engineering Europa (HVEE), is a tandem type and was the first in the world with a Cockcroft-Walton coaxial type accelerating system. It is provided with two sources: a plasma source for gaseous substances and a sputtering source for obtaining practically any element of the periodic table from a solid target.

CMAM is structured into three divisions: scientific, technical and administrative, to which are associated the 26 persons of the staff. The governing body is the Management Committee that includes the heads of division as well as elected representatives of scientific and technical-administrative staff. The director receives as well support from the Scientific Committee and the Centre Council. There is an external Advisory Committee, composed of six international scientists renowned in the research areas covered by the CMAM, appointed and chaired by the Vice Chancellor for Science Policy and Research Infrastructures of the UAM.

CMAM is a fully open infrastructure with different access schemes: we are committed to the widest possible collaboration with national and international research institutions.
THE UAM-FPCM COLLABORATION AGREEMENT

According to the will and policy of the UAM, the CMAM is a facility open to other universities, public research institutions and private companies to enhance the impact of UAM’s research investments on the social and industrial environment. The access to the accelerator beamtime and to ancillary instrumentation is fully open and competitive in one of three ways:

a) As a CMAM Member
b) As a scientist having established a scientific collaboration agreement with the CMAM
c) As an external user

To allow access to external users the UAM and the Madrid Science Park (FPCM, Fundación Parque Científico de Madrid) have signed, since 2004, an agreement that has reserved in 2010-2011 close to 40% of the available beamtime to the Unidad de Micro Análisis de Materiales (UMAM) of FPCM. The UMAM provides paying users with the necessary assistance as regards beamtime applications, experimental planning, use of the experimental stations and their equipment, data analysis and interpretation. From the CMAM side we provide the beam delivery and the maintenance of all the usable facilities thus giving, thank to this collaboration agreement, a complete service to any potential user of the accelerator and other ancillary equipment.

Since its inception this scheme has allowed access to about 50 research groups of which 20 in the years 2010-2011.

Information on the equipment and beamlines available can be found in the web page: http://www.cmam.uam.es/en/facilities/beamlines.

Information about the services offered by the UMAM can be found at the web page:
STRUCTURE OF CMAM

Individual charges

DIRECTOR: Alessandro Zucchiatti
DEPUTY DIRECTOR: Angel Muñoz Martín
ADMINISTRATION MANAGER: Beatriz Renes Olalla
WORK SAFETY COORDINATOR: Miguel Ángel Ramos Ruiz
QUALITY MANAGER: Angel Muñoz Martín

CAROLINA VILLASECA BLANCO (until August 2011)
MARTA GÓMEZ ESTEBAK (from October 2011)

Divisions

Administration & Human Resources Division

Head: Beatriz Renes Olalla
Ana Granados Simón, Inmaculada Sierra Martos
Mª Ángeles Gutiérrez Ortega

Scientific Division

Head: Alessandro Zucchiatti
Fernando Aguiló López, Aurelio Climent Font
David Jiménez Rey, David Martín y Marero
José Olivares Villegas, Ovídio Peña Rodríguez
Angelia Pérez Pacheco (2010), José Emilio Prieto de Castro,
Miguel Ángel Ramos, Mª Dolores Ynsa Alcalá

Technical Division

Head: Angel Muñoz Martín
Jorge Álvarez Echenique, Víctor Joco
Arántzazu Maira Vidal, Abdennacer Nakbi
Jaime Narros Fernández, Antonio J. Rodríguez Nieva
Carolina Villaseca Blanco (until 7/2011), Marta Gómez Estebar (from 10/2011)

PhD Students

Diana Bachiller Perea, Miguel L. Crespiello Almenara
Begoña Gómez-Ferrer Herrán, Javier Manzano Santamaría
Esther Punzón Quijorna, Andrés Redondo Cubero

Committees

Scientific Advisory Committee (SAC)

The international SAC was appointed in March 2011 by the Vice Chancellor for Research. Its
function is to evaluate the activity of the Centre and advise the VC Research and the CMAM
Director on scientific plans, on new experimental facilities and on improvements in managerial
and technical organization.

Prof. Rafael Garese Alarcón, Vice Chancellor for Research, UAM, Chair
Prof. Ricardo Amils Pibernat, Full professor of Microbiology, Dept. of Molecular
Biology, UAM, and associate researcher at the AstroBiology Centre (CSIC-INTA), Madrid, Spain
Dr. Jorge García Gómez-Tejedor, Director of the Restoration department of the “Centro
de Arte Reina Sofía”, Madrid, Spain
Prof. Ragnar Hellborg, Professor Emeritus of Nuclear Physics, Faculty of Science,
Lund University, Lund, Sweden
Prof. Elías Muñoz Merino, Superior Technical School of Telecommunications
Engineers, Universidad Politécnica, Director of the Institute of Optoelectronics Systems and
Microtechnology, Madrid, Spain
Dr. Carlos Rossi Álvarez, Istituto Nazionale di Fisica Nucleare, Padua, Italy
Prof. Leonardo Soriano de Arpe, Associate professor, Department of Applied Physics
UAM, Former Director of the Materials Science Institute “Nicolás Cabrera”, Madrid, Spain

Management Committee

A. Zucchiatti, Director and Chairman
A. Muñoz Martín, Deputy Director and head of the Technical Division
B. Renes Olalla, Head of the Administration and HR Division and Committee’s secretary
A. Redondo Cubero, Elected spokesperson of the PhD students until July 2010
J. Manzano Santamaría, Elected spokesperson of the PhD students from July 2010
J. Narros Fernández, Elected spokesperson of the technical and administrative staff
J. Olivares Villegas, Elected spokesperson of the scientific staff
J. E. Prieto de Castro, Elected spokesperson of the scientific staff
Scientific Internal Committee

The scientific committee is a forum of discussion on the scientific issues relevant to CMAM. It is formed by all members of the staff owning a PhD degree; they report directly to the Director. Besides the scientific division staff it includes: V. Joco, A. Maira Vidal, A. Muñoz Martín, of the technical division.

CMAM Council

The CMAM council is composed by all the members of the CMAM (staff and students) and meets at least once a year to discuss topics of general interest and to make suggestions to the direction about the organization and its improvement.

Beamtime Allocation Commission

Alessandro Zucchiatti  
Chair

José Emilio Prieto de Castro  
Secretary

Angel Muñoz Martín  
Technical advisor

Ramón Escobar Galindo  
Users representative from ICM-CSIC, Madrid

Fernando Aguillo López
PROFESOR EMERITUS AND FORMER CMAM DIRECTOR

Born in Mèrida (Spain), got his degree of "licenciado" by the Universidad Complutense de Madrid in 1956. In 1964 he obtained the PhD degree by the same University. He started his scientific career in the Nuclear Energy Commission (JEN), presently CIEMAT, creating a pioneer research group on radiation damage in insulating materials. From 1970 until his retirement in 2004 had a position of full professor in the recently created Universidad Autonoma de Madrid. He was responsible for setting up the teaching and experimental research activities in the Physics Division under the chairmanship of Prof. Nicolás Cabrera. In 1974 he was appointed director of the Department of Optics and Structure of Matter (presently Physics of Materials), leading a broad research program on: spectroscopy and optical properties of materials, defects and mechanical properties, crystal growth, dielectrics and ferroelectrics and nonlinear optics. Since 1999, he led, together with Prof. Aurelio Climent Font, a project to install a new laboratory for the analysis and modification of materials, CMAM, that was officially inaugurated in 2003. He became the first Director of CMAM in 2003. After his retirement in 2004, he continued his research activity in CMAM as emeritus professor. Most of his effort was devoted to develop a specific research line on the ion-matter interaction and on the photonic applications of ion beams with the collaboration of Dr. José Olivares from CSIC. As an output of his research activity, he has published around 350 papers, several books and monographs and has supervised 19 PhD students. He has been temporarily attached to several universities (Parma, Sussex, Dijon, Lisbon) and research centres (Brookhaven National Laboratory, IBM Zürich). He is recipient of the honour medal of the Royal Spanish Physical Society, the prize of research of UAM, and the Physical Sciences prize of CEOE for 1989.

Jorge Álvarez Echenique
COMPUTER SUPPORT ENGINEER

Jorge Álvarez Echenique works at CMAM as a computer support engineer, system administrator and webmaster and has done so for seven years. He obtained his degree in Chemistry at the Universidad Autónoma de Madrid (UAM) in 2002. He soon discovered his interest in computer science, graphic design and web design. Whilst working as a Computer Support Technician at UAM information technologies centre, he completed several courses and masters, including systems management (windows and linux environment), networks administrator, web development and graphic design. In addition to his work as IT for CMAM, he has worked as a freelance in various web projects and IT support for a wide variety of clients.

Diana Bachiller Perea
PhD STUDENT

Diana Bachiller Perea has a Degree in Physics from the Universidad Complutense de Madrid, Spain (2005-2010) and an Interuniversitary Master’s Degree in Nuclear Physics (2010-2011). Her current status is PhD student at CMAM-UAM with a FPI Fellowship of the Universidad Autónoma de Madrid. She was collaborating with the Nuclear Physics Department at the Universidad Complutense de Madrid (2009-2010) and working at the Microelectronics Institute of Madrid (IMM-CSIC) (2010-2011). Her work at CMAM started in 2011 for a master thesis related to a Coordinated Research Project of the International Atomic Energy Agency (IAEA) with the aim of creating a nuclear database containing cross sections for the most commonly demanded PIGE reactions. Her thesis work is focused on the damage produced by ionic irradiation on materials for fusion applications.
Aurelio Climent Font
FULL PROFESSOR AND FORMER CMAM DIRECTOR

Born in Tortosa (Spain), got his MSc by the Universidad Complutense de Madrid in 1973. In 1979 he obtained the PhD degree by the Universidad Autónoma de Madrid. He started his scientific career at the Department of Applied Physics studying the electric properties and conduction mechanisms of thin insulating films. In the period 1982-84 he stayed at Pennsylvania State University with a Fulbright-MEC fellowship using ion beam analytical techniques at the National Submicron facility at Cornell University for studying the damage produced in crystalline silicon by low energy ion beams and its effects on Schottky diodes and MOS structures. As an IBA scientist he has been attached to several universities (Denis Diderot-Paris VII, Lund) and research centres (Centre de Recherches Nucléaires in Strasbourg, European Commission JRC Institute for Reference Materials and Measurements in Gaest, Sandia National Laboratories in Albuquerque, Centre de Recherches sur l'Activation des Matériaux, a unit of the Direction des Sciences de la Matière at the Commission des Loisirs et des Jeux in Paris, INFN-LABEC in Florence). Involved since the beginning in the CMAM project, he has served as director of the centre from 2004 until 2009. Since 2010 is full professor of Applied Physics at UAM.

Begoña Gómez-Ferrer Herrán
PHD STUDENT

Begoña Gómez-Ferrer Herrán has a MSc in physics from Universidad de Valencia (2002 - 2007) including a year stage at Physics department of Imperial College London (2005-2006) and a European Master on Nuclear Fusion Science and Engineering Physics at Universidad Carlos iii de Madrid (2010). She was working for a year at an international consulting company, Accenture (2007 – 2008). In 2009 she started her PhD studies in IBA (Institute of Applied Physics) at the Universidad Autónoma de Madrid. Her thesis is about studying the effect of low energy ion beams on the electrical properties and conduction mechanisms of thin insulating films. Her thesis work in the CMAM consists in developing instrumentation for Surface Physics and low energy Ion Beam related methods. Besides, her work is strongly related with the beamline and experimental chamber fine tuning and release to users. She is an expert in different communication protocols, digital and analogue electronics, microcontrollers, detectors, converters, software, vacuum techniques and various scientific methods.

David Jiménez Rey
JUAN DE LA CIerva RESEARCHER

David Jiménez-Rey is currently lecturer under Juan de la Cierva contract at the Universidad Autónoma de Madrid, Departamentos de Física Aplicada. He got the Degree in Physics at the Universidad Nacional de Educación a Distancia (UNED), he then moved to the ICTS National Fusion Laboratory at CIEMAT, Madrid, Spain, where he made his PhD thesis, with a FPI Fellowship of the Science and Innovation Ministry of Spain. His thesis, defended at UNED (December 12, 2008), was focused on “Characterization of luminescent materials and its applications to the study of fast ions lost in T-J II stellarator”, under the supervision of Bernardino Zuro and Alfonso Basciò. This thesis received the extraordinary prize of PhD by UNED. As a post doc David joined the TechnoFusión Project, carrying out tasks as feasibility study & project management, and scientific researches related to the technologies needed for the fast track of the fusion program (ITER, IFMIF and DEMO), and fusion materials researches. In January 2010 he was awarded a research contract Juan de la Cierva, joining the materials for energy to the technologies needed for the fast track of the fusion program (ITER, IFMIF and DEMO), and in particular focusing on the damage and response of fusion materials by means of ions beams. David is developing a new in-situ ion beam analysis (IBA) for the damage kinetic study under irradiation, based on ionoluminescence (IL). Furthermore, he is developing the characterization studies of several scintillator materials of the Fast Ion Loss Detector of ITER. He is one of the spokespersons of the new irradiation and implantation beam line under commissioning at CMAM.

Javier Manzano Santamaría
PHD STUDENT

Javier Manzano got his degree by Universidad Autónoma de Madrid (UAM) in 2003. He started his PhD in the field of superconductors at low temperatures but had to abandon that project. In 2008 he was awarded a grant by the Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas (CIEMAT) to study superconductors at low temperatures. In 2009 he joined Fundación Parque Científico de Madrid, being addressed to the Centre for Micro Analysis of Materials, where he devoted his time to the development of new equipments.

Ángel Muñoz Martín
DEPUTY DIRECTOR

Ángel Muñoz-Martín got his degree in Physics in 1997 and, since 2002, he holds a PhD in Materials Science from Universidad Autónoma de Madrid. In 2003, Ángel joined Fundación Parque Científico de Madrid, being addressed to the Centre for Micro Analysis of Materials, where he devoted his time to the development of new equipments. In 2006 he obtained a position as Accelerator-Chief Engineer at CMAM and, since 2007, he is responsible of the whole Technical Division. In 2009 he was appointed deputy-director of CMAM. Dr. Muñoz-Martín has participated in several national and international committees for the development of new infrastructures related to ion accelerators and ion accelerator techniques, and he is actively participating in the development of new instrumentation at CMAM.
Abdennacer Nabi
TECHNICAL SUPPORT ENGINEER
Abdennacer Nabi works at CMAM as a Technical Support Engineer. He obtained his degree in Physics at Med Ben Abdullah University, Fez, Morocco in 1995, and Diploma of Electronic Engineer from the University of Granada, Spain, in 2003. His work in CMAM consists in taking part in tasks of maintenance of Ion Beam Accelerator. His work is centering in developing analog and digital electronic circuits and electric installations of the CMAM; he is also involved in development of beam lines.

Jaime Narros Fernández
ACCELERATOR TECHNICIAN
Jaime Narros works at CMAM as a technician since November of 2001. He joined the technical team of the center and keeps developing his job nowadays. His work in the CMAM consists in the operation of the accelerator, under the guidance of the supervisor of the radioactive installation. He participates in the maintenance tasks related with the accelerator, the ion sources, and the auxiliary installations. He also takes part in the development of the accelerator, the complementary equipments and beam lines in collaboration with the responsible of the installation and beam lines.

José Olivares Villegas
SCIENTIFIC INVESTIGATOR
José Olivares is currently Investigador Científico, Instituto de Optica, CSIC, and associated to CMAM-UAM. He received his PhD in Physics from the Universidad Autónoma de Madrid (UAM), Spain, in 1994, for work on the topic of proton exchanged waveguides in lithium niobate. From 1994 to 1998 he was a postdoctoral fellow at the University of Sussex, UK, working on the topics of ion implanted waveguides and of laser damage and ablation for film deposition and micro structuring. From 1997 to 1998 he worked at the University of Oviedo, Spain, in the field of integrated optics in lithium niobate. From 1999 to 2001 he worked at Instituto de Optica, CSIC, in the field of third-order optical nonlinearities of metallic nanoparticles. In 2001 he became Tenured Scientist of CSIC. Since 2003 he is working in collaboration with the Centro de Microanálisis de Materiales (CMAM) at UAM, Madrid, in the field of photonic applications with high energy ions, particularly leading the topic of novel and efficient optical waveguide fabrication with swift heavy ions and researching in the fundamental aspects of the origin of electronic damage. Recently, he is involved in understanding the ion damage in optical materials like SiO$_2$, that are also relevant to the fusion community.

Ovidio Y. Peña Rodríguez
VISITING SCIENTIST
Ovidio Y. Peña Rodríguez studied the degree in Nuclear Engineering at the Instituto Superior de Ciencias y Tecnología Nucleares (ISECYN). Cuba, Afterwards, he moved to Universidad Autónoma de México (UNAM), Mexico, where he developed a PhD work in Materials Science and Engineering. In the period between August, 2007 and December, 2011, performed successive postdoctoral fellowships in Universidad Autónoma de Puebla (UAP) and UNAM, both in Mexico, as well as in Instituto de Ciencia de Materiales de Barcelona (ICMAB-CSIC) and Centro de Micro-Análisis de Materiales (CMAM). In Spain. He has experience in various experimental techniques and on the simulation of the optical properties of metallic nanoparticles. Nowadays, Ovidio is working in the development of nanostructured coatings for the final optics of nuclear fusion facilities. More generally, he is interested in the effects of radiation on nanostructured materials and in the optical properties of plasmonic nanoparticles. He is currently a postdoctoral fellow under the PICATA program (Programa Internacional de Captección de Talentos) at Instituto de Fusión Nuclear, Universidad Politécnica de Madrid (IFN-UPM).

José Emilio Prieto
PROFESSOR UNDER CONTRACT
José Emilio Prieto is currently professor under contract (Profesor Contratado Doctor) at the Universidad Autónoma de Madrid, where he is a member of CMAM of the Dpto. de Física de la Materia Condensada and of the Instituto Nicolás Cabrera. He got a degree and a PhD in Physics from the UAM working in the field of the growth and surface characterization of thin optical films of magnetic materials. After a post-doctoral stage at the FU Berlin financed by a Humboldt fellowship, where he performed research in synchrotron-based magnetic spectrosocopies, he joined the CMAM with a Ramón y Cajal contract. His current research interests are the growth and characterization of new magnetic materials, the study of mechanisms of optical growth and the use of ion beams for characterization and modification of materials properties.

Esther Punzón Quijorna
PhD STUDENT
She is from the land of Don Quijote, Consuegra (Toledo). Graduated in Physics from Universidad Autónoma of Madrid (UAM) in 2007. She is a PhD student since 2007 at UAM, with Aurelio Climent Font as director. During her thesis she is studying ion beam techniques (with light and heavy ions, at energies from keV to MeV) for the development of micro and nano structures with optical contrast (of interest in optoelectronics), and contrast of electrical properties (specific stimulation of biological processes of differentiation). She is interested in new branches of Nanobiocience and techniques of analysis by ion beams. She has completed a research stay for eleven months, as visitor PhD student at the Joint Research Center European Commission (JRC) in Ispra (Italy).

Miguel Ángel Ramos
PROFESSOR
Along his PhD thesis he has developed the first Low-temperature Scanning Tunneling Microscope in Spain, performing tunneling spectroscopy experiments in so-called High Critical Temperature Superconductors. During his post-doctoral stage in the KPF at Gilan (Germany), he worked on the so-called Soft Potential Models which rather successfully accounts for the low-energy excitations in glasses or non-crystalline solids. His current research lines focus on the study of thermal (and also acoustic, structural and vibrational) properties of glasses and other disordered solids, at low temperatures and/or low energies. Emphasis is put on correlating these with their thermodynamic properties at higher temperatures, that is, around the glass transition region, an open and much debated unsolved topic in physics since longer than 100 years. Up to now, he has mainly studied boron oxide (B$_2$O$_3$) glasses, as well as polymorphic molecular solids made of simple monoatomic (such as ethanol, propane), butanol and their isomers which exhibit glassy phases together with crystalline ones including sometimes orientational disorder. Finally, he has begun a new research line on the possible existence of ferromagnetism in carbon materials by ion-beam irradiation, employing the 5 MV ion-beam accelerator at CMAM-UAM.

Antonio Rodríguez Nieva
ACCELERATOR TECHNICIAN
Antonio Rodríguez works at CMAM since December of 2000. He joined the technical team of the center and keeps developing his job until nowadays. His work in the CMAM consists in the operation of the accelerator, under the guidance of the supervisor of the radioactive installation. He participates in the maintenance tasks related with the accelerator, the ion sources and the auxiliary installations. He also takes part in the development of the accelerator, the complementary equipment and beam lines in collaboration with the responsible of the installation and beam lines.
SCIENTIFIC IMPACT

3

- Research Projects
- Scientific Publications
- Theses
- Scientific Contributions to Conferences
- Seminars
- Visits
- Collaborations
- Courses and Training

SUMMARY OF SCIENTIFIC ACTIVITIES

Research projects
- Projects funded by the European Union 1
- Projects funded by MICINN, CDTI 7
- Projects funded by CAM 3

Scientific publications
- Papers in SCI journals 61
- Papers in no-SCI journals 2

Theses
- PhD Theses 2
- MSc Theses 2

Scientific contributions to conferences
- Invited talks 7
- Oral contributions 7
- Posters 26

Seminars
- Seminars at CMAM 32
- Seminars of CMAM members 6

Visits
- Visits to the CMAM 26
- Visits of CMAM members 7

Collaborations
- International collaborations 22
- National collaborations 15

Courses and training
- Courses and training 11
PUBLICATIONS OF CMAM MEMBERS

3.0 < Impact factor 17 Articles 27 %


5. O. Peña-Rodríguez, U. Pal, Au@Ag core-shell nanoparticles: Efficient all-plasmonic Fano-resonance generators, Nanoscale 3 (2011) 3609-3612. IF: 4.110


22. V. Caroñen, A. Redondo-Cubero, E. Guti- érez-Puebla, J.A. Rodríguez-Velama- zan, M.A. Monge, E. Dieguez, D. Martin

CMAM ACTIVITY REPORT 2010-2011

SCIENTIFIC IMPACT

CMAM ACTIVITY REPORT 2010-2011

SCIENTIFIC IMPACT


In Physics Research Section B 269 (2011) 492-497. IF: 1.042


61. Punzon Quijorna, E.; Torres Costa, V.; Climent-Font, A.; Manso Silvan, M.; A multi-ion beam microanalysis approach for the characterization of plasma polymerized allylic films European Physical Journal A 56 (2011) 24021 IF: 0.902


Invited talks


5. A. Climent Font. Lustre ceramics. A sophisticated decoration process unravelled with the contribution of IBA techniques. COST Strategic Workshop, Florence, Italy, July 11-13 2011


7. M. A. Ramos, The Boson Peak through a model system of monohydrated alcoholics. 4th Meeting on Structure and Vibrations in Oxide Glasses, Université de Montpellier 2, Montpellier, France 25 March 2011

Oral contributions


Poster contributions
15. M. A. Ramos, J. Barzola Quiquia, P. Esquinazi, A. Zucchiatti at the Climent-Font and M. Garcia Hernández. Magnetic properties of graphite irradiated with MeV ions, VI
Reunión Nacional de Física del Estado Sólido (GEFES), Zaragoza, 3-5 February 2010.


25. A. Climent-Font, I. Arbona, M. Calviño, C. Alfonso, A. Zucchiatti. Fabrication of optical waveguides in LiNbO3 irradiated with swift heavy ions. 10th International Conference on Fusion Reactor Materials, Charleston, South Carolina, USA, 16-22 October 2010.


SEMINARS OF CMAM MEMBERS


• José Emilio Prieto de Castro, Structure studies with ion beams, including sur- faces, in Erasmus School “Engineering and characterization of nanostructures by ion beams and nuclear methods”, Leuven, Belgium, April 25th – May 4th 2010.

• Alessandro Zucchiatti, Leer el patrimonio con los instrumentos de la Física, Uni- versidad Autónoma de Madrid, Facultad de Filosofía y Letras, 24 November 2010.

• Miguel Angel Ramos. The glass transition and the universal properties of glasses, Departamento de Física Teórica de la Mate- mática Condensada, Universidad Autónoma de Madrid, 6th April, 2011.

• Fernando Aguillo López, Ions and photons to modify materials: a synergic approach First CLPU Users meeting, Salamanca, 14 December 2011.
SEMINARS AT CMAM

1. Ovidio Pería Rodríguez, Ionoluminescence induced by swift heavy ions in silica and quartz, CMAM, Madrid. (22-12-11).
2. István Bányász, Recent results in ion beam fabrication of optical elements, Research Institute for Solid State Physics and Optics, Hungarian Academy of Sciences, Budapest (Hungary). (20-12-11).
4. Aurelio Climent Font, Looking for the best ion beam to study lattice ceramics, CMAM, Madrid. (23-11-11).
5. Elías Sidera-Haddad, Interactions of Accelerated Charged Ions with Diamond, Director of MPRI (Materials Physics Research Institute), University of the Witwatersrand (Wits), Johannesburg-South Africa. (26-10-11).
8. Edgar Casanova González, Espectроскопия Raman amplificada en superficie (SERS) de colorantes mexicanos, Instituto de Fisica, UNAM, México. (22-09-11).
10. Gianluca Quarta, Applications of Ion Beam Analysis and Accelerator Mass Spectrometry to cultural heritage diagnostics at CEDAD, CEDAD (Centre for Dating and Diagnostics), Universidad de Salento, Lecce, Italy. (16-06-11).
12. Núria Gordillo García, Development of the 3D ion beam analysis at the CENBG nano-beam line: STIM and PIXE tomograph, Centre d’études Nucléaires de Bordeaux Gradignan (CNBG), Bordeaux, France. (02-06-11).
18. Alfonso Blázquez Castro, Aplicaciones biológicas del efecto fotovoltaico en volúmenes en LINO3F6, Hospital MD Anderson, Houston, TX. (20-01-11).
19. Alessandro Zucchiatti, Irradiación de objetos del patrimonio con haces de iones: ¿cuáles son los efectos?, CMAM, Madrid. (03-11-10).
20. Ulderico Santamaría, Recent polychromy studies on work of art at Vatican Museums, Università della Tuscia e Laboratorio di Diagnostica per la Conservazione e Restauro Michele Cordaro, Roma, Italy. (28-10-10).
22. Angela Pérez Pacheco, Photoacoustic Spectroscopy: some principles and applications, CMAM, Madrid. (17-09-10).
23. Ovidio Pería, Optical properties of Silicon and Germanium nanocrystals, Institut de Ciencia de Materiales de Barcelona, IMCB, CSIC, Barcelona. (10-09-10).
24. Ramón Escobar Galindo, Tayloring optical properties of Cr-Si mixed oxides by oxygen ion-beam implantation, CSIC, Madrid. (12-09-10).
25. Miguel Ángel Ramos, Magnetic properties of graphite irradiated with MeV ions, CMAM-INC, Madrid. (06-05-10).
27. José Mª Fdez Navarro, Aplicaciones funcionales de los vidrios, Instituto de Optica, CSIC, Madrid. (15-04-10).
28. Mª Dolores Ynsa, Efectos del Halo en Técnicas con Haces de Iones, CMAM. (08-04-10).
29. Antonio Rivera de Mera, Excitonic model of materials damage by ion irradiation, Institute de Fusión Nuclear - UPM, Madrid. (25-03-10).
31. D. Jiménez-Rey, Ionoluminisence and its application a plasma of fusion, CMAM, Madrid. (04-03-10).
32. Víctor Joco, Mi experiencia sobre el trabajo de desarrollo en el CMAM, CMAM. (11-03-10).

VISITS TO CMAM

Dr. Andrew D. C. Alves
Prof. István Bányász
Prof. Lucio Calcagnile
Prof. Iain Campbell
Prof. Patricia Fernández
Missis Eleonora Cuccia
Miss Rosangela Faleta
Prof. Fan Hongjin
Dr. Patricia Fernández Esquivel
Dr. Nuria Gordillo

University of Melbourne, Melbourne, Australia
Research Institute for Solid State Physics and Optics, Hungarian Academy of Sciences, Budapest, Hungary
University of Guelph, Guelph, Canada
Instituto Tecnológico e Nuclear (ITN), Sacavem Portugal
Department of Physics, University of Guelph, Guelph, Canada
Instituto de Física de Materiales de Barcelona, CSIC, Barcelona
University of the Witwatersrand (Wits), Johannesburg-South Africa
School of Physical and Mathematical Sciences, Nanyang Technological University, Singapore
Fundación Museos Banco Central de Costa Rica, San José, Costa Rica
CMAM, Madrid.

CMAM ACTIVITY REPORT 2010-2011
CMAM ACTIVITY REPORT 2010-2011
VISITS OF CMAM MEMBERS

Prof. Aurelio Climent Font Laboratorio Beni Culturali of INFN, Florence, Italy
Dr. Alessandro Zucchiatti INFN, Genova, Italy
Dr. Alessandro Zucchiatti Centre de Recherche et Restauration des Musées de France, Paris, France
Dr. Víctor Joco Institute of Experimental Physics, Johannes Kepler University, Linz, Austria
Dr. María Dolores Ynsa Alcalá Centre for Ion Beam Applications (CIBA), Singapore
Dr. David Jiménez Rey INFN, Genova, Italy
Dr. Alessandro Zucchiatti Université Moulay Ismail, Meknes, Morocco

PhD THESIS

1) Structural and compositional characterization of wide bandgap semiconductor heterostructures by ion beam analysis

Defended by Andrés Redondo Cubero at the auditorium of the CMM-CSIC on 9th July 2010.

This thesis addresses the application of ion beam analysis to the study of several wide bandgap semiconductor heterostructures. This work is motivated by the need of improving the epitaxial growth of the active and base layers composing high electron mobility transistors (HEMTs) and high-power optoelectronic devices, mainly based on GaN and ZnO. At the same time, this thesis explores the advantages and limits of ion beam techniques for the structural and compositional characterization of such heterostructures, as an alternative and complement to X-ray diffraction methods. The thesis has obtained the 2010 special PhD prize of UAM.

2) Ion beam damage by electronic excitation with swift heavy ions in lithium niobate: mechanisms and nanostructuring for photonic applications

Defended by Miguel Crespiollo Almenara at the Faculty of Sciences of the Universidad Autónoma de Madrid, on 12th December 2011.

The aim of the Thesis has been to study the origin, main features and potential applications of the damage produced by high density electronic excitation under high energy heavy ion irradiation (i.e. swift heavy ions). From the fundamental point of view it discusses the different mechanisms responsible for the observed damage. This has been addressed under some specific modelling and by studying the thermal stability of the electronic damage. The unique nanostructuring capabilities offered by the damaged ion tracks (nanometer diameter and microns length) has been exploited to study, in LiNbO₃, the fabrication of special nanopores structures and thick (up to ~40 µm) optical waveguides based on nanostructured effective optical medium obtained at ultralow fluence (~10¹¹ at/cm²).

This research has been carried out at the Centro de Micro-Análisis de Materiales (CMAM) of the UAM where most of the ion irradiations (with ion energies up to 50 MeV) have been performed. Some very high energy irradiations (~1000 MeV) have been performed at GANIL (Caen, France) and GSI (Darmstadt, Germany).
1) Ion irradiation experimental set-up for study of defect kinetics in fusion materials
Defended by Begoña Gomez-Ferrer Herrán for the European Master in Nuclear Fusion-Science and Engineering Physics at the Carlos III University of Madrid, September 2010
The thesis has focused on the experimental study of radiation damage and its evolution in materials of interest for fusion with comparison to simulations. Resistivity Recovery (RR) technique has been used. Samples have been irradiated at low temperatures with high energetic ions to produce displacement damage. After irradiation the studied materials were isochronously annealed. Evolution of the damage was followed-up through resistivity measurements of the irradiated sample after each step of the annealing. Variation of the resistivity has produced extended information on the mobility, recombination, clustering and dissociation of defects in the material. Resistivity measurement methods and techniques have been revised. Modeling methods and current state of RR investigations for pure Fe were shown and made clear the lack of experiments performed with ions.

2) Commissioning of a new setup for PIGE technique application
Defended by Diana Bachiller Perea for the Mástér Inter-Universitario en Física Nuclear at the Universidad Complutense de Madrid, September 2011.
The extended use of Particle Induced Gamma ray Emission (PIGE) as a routine technique for characterizing materials is, presently, no possible due to the lack of accurate measurement of gamma emission cross sections. A Coordinated Research Project of the International Atomic Energy Agency, (IAEA) aims to create a nuclear database containing cross sections for the most commonly demanded PIGE reactions. The Center for Micro-Analysis of Materials is participating in this project aiming at measuring a selection of those cross sections, in view of future analytical applications. The experimental work for this master thesis has been dedicated to preparing a specific setup for serial cross section measurements.
To achieve the requested measurement precision implies a detailed characterization of the experimental setup and all its parts and measurements protocols. For this reason, several measurements for the gamma ray detectors efficiency and resolution determination, accelerator absolute beam energy calibration, and characterization of the target conditions have been carried on.
### COLLABORATIONS

#### International collaborations

1. Instituto Tecnológico e Nuclear (ITN), Portugal.
2. Centre for Ion Beam applications (CIBA), Singapore.
3. Istituto Nazionale di Fisica Nucleare Genova, Italy.
4. LABEC, Istituto Nazionale di Fisica Nucleare Firenze, Italy.
5. Laboratorio Scientifico dei Musei Vaticani, Vatican City.
6. Università degli Studi della Tuscia, Viterbo, Italy.
7. Facultad de Ciencias, Universidad de Chile, Santiago, Chile.
8. Departamento de Física, Universidad Nacional Autónoma de Mexico, Mexico City, Mexico.
10. DIURAS, Università di Genova, Genova, Italy.
11. University of the Witwatersrand, Johannesberg, Republic of South Africa.
12. Thin Film Physics Division, Department of Physics, Chemistry and Biology – IFM Linköping University, Linköping, Sweden.
13. Department of Physics, Department of Materials Science and Engineering and Materials Research Institute, The Pennsylvania State University, University Park, USA.
16. Universidade de Shandong, China.
17. Università degli studi di Padova, Padova, Italy.
18. Institute of Physical Chemistry, Bulgarian Academy of Sciences, Sofia, Bulgaria.
19. Institute of Nuclear Physics, Moscow State University, Moscow, Russia.
20. Institute of Nuclear Physics, Moscow State University, Moscow, Russia.
21. Max-Planck-Institut fuer Plasmaphysik, Munich, Germany.
22. GANIL (Grand Accélérateur National d’Ions Lourds), CMIAP-CNRS, Caen, France.

#### National collaborations

1. Departamento de Física Aplicada, UAM, Madrid.
2. Departamento de Física de Materiales, UAM, Madrid.
3. Instituto de Fusión Nuclear, Universidad Politécnica de Madrid, Madrid.
4. CIEMAT, Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas, Madrid.
5. INTA, Instituto Nacional de Técnica Aeroespacial, Madrid.
6. Departamento de Biología Molecular, UAM, Madrid.
7. Museo de América, Madrid.
8. Instituto de Historia, CSIC, Madrid.
9. Centro de conservación y restauración de la Junta de Castilla y León, Valladolid.
10. Servicio de Conservación y Restauración (SECYR), UAM, Madrid.
11. Instituto de Cerámica y Vidrio, CSIC, Madrid.
12. Universidad Politécnica de Catalunya, Barcelona.
13. Departamento de Física de la Materia Condensada, UAM, Madrid.
14. Instituto de Química Física RocaSaloano, CSIC, Madrid.
15. Centro Nacional de Aceleradores, Seville.

### SUPPORT TO COURSES AND TRAINING

1. Inter-Universities PhD on Nuclear physics (2010)
   - Angel Muñoz-Martín.
2. Master on Plastics and Rubber (2010-2011)
   - Angel Muñoz-Martín.
   - Aurelio Climent Font.
   - David Jiménez-Rey.
5. Training stay FPI of Jorge Arcaz (2010)
   - Victor Joco.
6. Summer course from the Polytechnical University of Madrid (2011)
   - Angel Muñoz-Martín.
7. Inter-Universities Master on Nuclear Physics (2011)
   - Angel Muñoz-Martín.
8. Training stay of Rosangela Faieta (2011)
   - Alessandro Zucchiatti.
9. Cultural heritage course in CMAM (2011)
   - Aurelio Climent Font.
10. Ancillary Research Project of PhD student Eleonora Cuccia (2010), Italy
    - Alessandro Zucchiatti and David Jiménez-Rey.
11. Training stay of Daad Halum (2010), Syria
    - Alessandro Zucchiatti.
In addition to typical IBA techniques (PIXE, RBS, ERDA, etc.) for atomic elementary analysis of materials in a spatial scale ranging from nanometers to microns, ion-beam accelerators are also powerful tools for ionic implantation and modification of materials. When an energetic ion impinges on a solid target, important changes in the structure and properties of the solid are produced that can be used for different applications within the fields of physics and engineering, such as biochips or optical and electronic devices. Typical energy ranges employed in ion implantation are very wide, from a few tens of keV—obtained by small ionic sources—up to hundreds of MeV—produced by ion-beam accelerators—which use to be more interesting, though more expensive too, for scientific research.

Our research team is currently employing these techniques to address a hot topic within Solid State Physics nowadays: the study of the reported possible magnetic behavior in graphite, graphene and other carbon-based materials. As is well known, graphite, the stable crystalline allotrope of carbon at room temperature and ambient pressure, is known to exhibit a strong and anisotropic “textbook” diamagnetism, due to its delocalized π electrons. Nevertheless, in the last two decades more or less clear evidences of ferromagnetic behavior in carbon at room temperature have been reported, especially the presence of ferromagnetic signals in proton-irradiated Highly-Oriented Pyrolitic Graphite (HOPG) reported [1] by a group at the University of Leipzig (Germany) and led by Prof. Pablo Esquinazi (who was on sabbatical at CMAM during 2007-08).

Therefore, we have undertaken a joint research line to study this subject, by making use of the 5 MV tandem ion-accelerator at CMAM and collaborating with other research groups at the Campus of Universidad Autónoma de Madrid and of the University of Leipzig (Germany). At the same time of the ion implantation, the Particle-Induced X-ray Emission (PIXE) technique allows to determine in situ the amount of magnetic impurities in the sample, a crucial issue given the weakness of the reported ferromagnetic signals. The possible existence of ferromagnetism—or other magnetic contributions—in the samples is studied through highly accurate SQUID magnetometry and sometimes through Magnetic Force Microscopy (MFM) [2].

We have exhaustively studied [3] the change in the magnetic properties produced on HOPG samples after irradiation of H-, C- and N- ions in the MeV energy range. The use of specially made sample holders for the magnetic measurements through a SQUID magnetometer provided high reproducibility, allowing us to obtain directly the irradiation effects without any corrections or subtractions. Our results show that three main magnetic phenomena are triggered by the defects produced by the irradiation, namely Curie-like paramagnetism, ferromagnetism and an anomalous paramagnetic state that appears as precursor of the magnetic ordered state. Direct measurements of the surface sample temperature during irradiation and the decrease in the paramagnetic as well as ferromagnetic contributions after irradiation indicate that self-heating effects are one of the causes for the small yield of ferromagnetism. Taking into account the role of hydrogen, our results [3] suggest that the induced ferromagnetism appears when the average vacancy distance is around 2nm in the near surface region.

The surface science research line at CMAM is based on the study of the structure of surfaces and very thin films of new materials their relation with the properties (magnetic, electronic...) that make these materials useful. It is based on the experimental setup of UHV-surfaces beamline, which contains a powerful set of facilities for the growth of thin epitaxial films and sample characterization using several experimental techniques. The system allows the investigation based on Molecular Beam Epitaxy (MBE) and their analysis by means of the standard ion-beam techniques using the high-energy ions provided by the CMAM accelerator [Rutherford backscattering spectroscopy (RBS), elastic recoil detection Analysis (ERDA), etc.], as well as the characterization of the samples with surface-sensitive techniques like high-energy ion scattering (LEIS), low-energy electron diffraction (LEED) and Auger electron spectroscopy (AES). The equipment consists basically of a sample preparation chamber, a main analysis chamber and a load-lock system for sample introduction and transfers. A goniometer with 3 rotation axes and 2 translations is mounted in the main chamber. For sample growth by MBE, the preparation subsystem is equipped with Knudsen cells containing different elements.

For the performance of RBS/channeling and ERDA experiments in the UHV-surfaces setup, the high energy ions produced by the 5 MV tandem accelerator are transported over a distance of 15 m and focused to a spot smaller than 10 μm. A sample is bombarded by a chopped beam of noble gas ions (He, Ar, Ne...) with energies typically in the range 2-6 keV. The availability of this LEIS-ToF technique and LEED will represent a powerful combination of surface-sensitive structural techniques which will allow the determination of surface structures in a wide class of materials. For this purpose, comparison measured intensities with the results of calculations for model structures are required in both methods. Full dynamical calculations of diffracted electron intensities will be performed in the case of LEED, while the code TRIC, recently developed in our group for simulation of ion trajectories in crystalline matter, will be used for the analysis of LEIS data. Both techniques are complementary in many aspects. In favorable cases the atom position can be determined with LEIS with a precision comparable to that obtained by X-ray methods with increased sensitivity for light elements. One the other hand, the sensitivity of LEED to the few first monolayers is very convenient for the analysis of ultrathin films. In the last period, significant advances have been achieved in the commissioning of this experimental system. Research work has concentrated on the study by simulations of various aspects of epitaxial growth and on the high optical confinement of light guiding. In particular, high optical confinement can be obtained what is of great interest for nonlinear optical applications. This is achieved in the so called regime of overlapping damage tracks. The fluences needed are of the order of 10^14 cm^-2, two orders of magnitude lower than those required by light ion implantation. On the other hand, we research in the regime of isolated track impact generating nanostructured materials that also allow to produce light guiding. In this way optical waveguides are obtained with ultralow fluences of 10^12 cm^-2. A current third topic of research is the fabrication of nanopores by means of selective chemical etching of the amorphous nanotracks produced in each high energy ion impact. The potential of these templates for nanoparticle formation and for sensor in waveguides format is being explored. The main optical materials being studied are LiNbO₃, KGW, TiO₂. In this context, the electronic damage is also being carried out on optical materials that are of interest for the fusion energy field, like amorphous and crystalline SiO₂. This is made in collaboration with research groups of CIEMAT and UPM.

**ARCHAEOLOGY**

The CMAM has been an important performer in the development that ion beam techniques have seen in the last decade, being involved, as promoter, in the COST European cooperation actions, COST G1 and G8, focusing on the application of non-destructive analytical techniques to Heritage and Conservation Science.

For the study of cultural heritage the CMAM counts on a dedicated experimental station based on a focused microbeam which is referred to as the External Microbeam Line. One can therefore irradiate micrometer-sized structures of a sample or, if needed, make scans over a whole object. The analytical part includes four complementary techniques usable in parallel: PIXE, PIGE, RBS and Ionoluminescence; the latter having been incorporated recently mainly for the identification of mineral aggregates in ceramic objects. Their applications result in a wide possibility of compositional and structural characterization of complex objects, with layered structure and formed by both light and heavy chemical elements.

The archaeometry activity in the past two years has been performed exclusively on the basis of collaboration between the CMAM and other research centers since we have not specific or founded projects in this domain. We have collaborated with the University of Tuscia (Italy) and the Scientific Laboratory of the Vatican Museums, with the Centre for Conservation and Restoration of the Cultural Heritage of Castiglia y Leon, with the Institute of Ceramics and Glass of the CSIC, with the CCHS, who coordinated the research program, and with the University of Valencia and metallurgists of the Human and Social Sciences Centre (CHCS) of the CSIC of Madrid.

The largest and most important project carried on, has focused on the characterization of the exceptional pre-Colombian gold collection of the Museo de America of Madrid in collaboration with the colleagues of the CCHS, who coordinated the research program, the Museo de America, the Physics Institute of the UNAM (Universidad Nacional Autónoma de Mexico) and the Museums Foundation of the Banco Central de Costa Rica. At the CMAM accelerator we have completed, in three phases (November 2010, May 2011 and December 2011), the IBA analysis (PIXE and RBS) of 108 pieces. Given the importance of the collection and the extension and aims of the analyses, the project has received not only the attention of the scientific community but also the coverage of the media and has been dealt with in several newspapers and also in a dedicated broadcast.

**BIO-MEDICAL PHYSICS**

The CMAM has a specific interest in biomedical applications of ion beams which stems from the relevance that this research field has in the society and from the experience of some of its members.

Part of the activity of CMAM related to biomedical applications was focused on the development of the sub-micrometer beam line at +30 degrees. It is based on a set of 5 like index profile. Then, high optical confinement can be obtained what is of great interest for nonlinear optical applications. This is achieved in the so called regime of overlapping damage tracks. The fluences needed are of the order of 10^14 cm^-2, two orders of magnitude lower than those required by light ion implantation. On the other hand, we research in the regime of isolated track impact generating nanostructured materials that also allow to produce light guiding. In this way optical waveguides are obtained with ultralow fluences of 10^12 cm^-2. A current third topic of research is the fabrication of nanopores by means of selective chemical etching of the amorphous nanotracks produced in each high energy ion impact. The potential of these templates for nanoparticle formation and for sensor in waveguides format is being explored. The main optical materials being studied are LiNbO₃, KGW, TiO₂. In this context, the electronic damage is also being carried out on optical materials that are of interest for the fusion energy field, like amorphous and crystalline SiO₂. This is made in collaboration with research groups of CIEMAT and UPM.
quadrupoles which ensure demagnification up to a factor 60 so that the line has the potential of delivering e.g. proton beams between 300 nm and 25 microns. The line has been designed to provide three main nuclear analytical techniques: Scanning Transmission Ion Microscopy (STIM), Particle Induced X-ray Emission (PIXE) and Rutherford Backscattering Spectrometry (RBS) which, associated with beam scanning, are imaging-analytical techniques of proven efficacy in the characterization of bio materials and their interaction with extraneous materials. Ancillary instrumentation is dedicated to the preparation of biological samples and comprises: a cryotome, a lyophylizer, an acid digestion system with associated microwave oven, an optical microscope with digital imaging, a cutter/polisher machine for the preparation of hard tissues.

The line is completely assembled and is undergoing extended testing (122 shifts in 2010-2011) with proton beams in the aim of achieving stable and reproducible near or sub-micron performances. The major part of the work has been done on understanding the way of assuring the stability of the beam with respect to localized temperature variations and mechanical movements, on sample manipulation, on detectors test and development and on progressive improvements of the collimators mechanical set up and alignment.

Besides the instrumental developments and the research activity performed in collaboration at other centers, some results in this field have been produced with the currently available CMAM equipment by irradiating materials in our Standard beam-line, interesting results have been obtained in the domain of biosensors. Application perspectives are foreseen related to the selective growth of porous silicon produced by silicon ion bombardment according to predetermined patterns along which preferred arrangements have been observed in cell bodies, particularly the nucleus. In the CMAM it was also investigated, in collaboration with the Complutensis and Carlos III Universities and the Gregorio Marañon Hospital, the generation of PET radioisotopes with low energy threshold production, to be used as markers of irradiated volumes in proton therapy, with encouraging results from a trial experiment.

MATERIALS FOR ENERGY PRODUCTION

Materials science, applied to energy production, covers a vast field of activities and topics. At CMAM, there is, since 2004, a group dedicated to the analysis and modification of advanced materials for fusion and fission reactors.

Functional and structural materials of nuclear reactors are exposed to a hostile environment as a consequence of the intense radiation. In particular, in fusion reactors, the hot plasma generates a high flux of charged particles, high-energy neutrons and gamma rays, which will affect not only the materials of the first wall, but also other more distant equipments such as plasma heating or diagnostic systems. This irradiation, via atomic displacement phenomena and ionizing processes, will produce a number of defects in the structure of the materials, affecting their physical properties at different scales. In addition, the nuclear reactions induced by the neutrons will generate transmutation products (impurities) that may change the physical properties of the materials, and therefore, affect their reliability. The high temperatures and the intense magnetic fields may also contribute to property changes.

The use of ion beam accelerators is considered as an efficient and useful tool to study experimentally the effects of particle irradiation in metallic alloys and functional materials, even when the involved processes are different to those of neutron irradiation.

The defects created in a material structure during irradiation give rise to the modification of its properties, due to the changes on the crystalline structure, being the most important ones sputtering-erosion, swelling, thermomechanical, dielectric and optical properties and changes in light ions solubility and diffusion.

CMAM studies are concentrated on the following functional and structural materials:

- Ceramic breeder blanket materials, used for producing tritium fuel for fusion reactors.
- Radiation damage will result in microstructural changes that may affect the properties of the breeding materials, especially the tritium migration.
- Fused silica, used mainly for diagnostic systems in fusion reactors, but also with applications in other technological fields on which radiation damage could play an important role.
- Fe, Fe-Cr alloys, EUROFER and EU-ODS EUROFER steels, which are strong candidates for structural materials in fusion and advanced fission reactor. For these materials, it is still necessary to clarify degradation mechanisms induced by radiation as well as diffusion and aggregation processes of light H and He.
- Scintillators, to be used as wide range ion detectors for fusion devices.

NUCLEAR PHYSICS

The activity at CMAM related to Nuclear Physics Line is primarily focused on characterising excited states near particle threshold for their relevance in Nuclear Astrophysics. At CMAM we populate these states in low energy reactions. These studies are complementary to the ones we perform at the major European Facilities; in beta decay at SOLDE-CERN and by reactions done at energies close to the Coulomb barrier at REX-ISOLDE, Geneva Switzerland and at relativistic energies at GSI, Darmstadt, Germany.

In this context we have performed measurements in order to determine the cross section of the \( ^{\text{He}} + ^{\text{He}} \rightarrow ^{\text{Be}} \) reaction as a function of energy. The technique used in the experiment is the activation method which consist on the detection of the delayed 478 keV \( \gamma \)-ray from the first excited state in \(^{3}\text{Li}\) after the \(^{\text{Be}}\) electron capture decay.

Further, we explore excited states in \(^{12}\text{C}\), using the \(^{12}\text{C} \rightarrow ^{11}\text{C}(^{\text{Be}},^\text{He}\alpha\alpha\alpha)\) reaction, in order to obtain better understanding of the triple alpha process as well as to study \(\alpha\)-clustering in light nuclei.

In parallel we do test experiments and R&D of experimental set-ups and equipment to be used in future experiments. Especially for the development of a gamma and proton calorimeter: CEGA (Califa Endcap Phoswich Array) demonstrator composed of LaBr/LaCl Phoswich detectors, to be used in reactions experiments at relativistic energies to detect high energy gamma rays (< 30 MeV) and high energy protons (< 300 MeV) at the future European Nuclear physics facility FAIR (Facility for Antiproton and Ion Research) Darmstadt, Germany.

The Nuclear Physics Beam Line is operational since 2005, several improvements on the beam line has been made during 2011. As we operate at very moderate beam intensities (few nA) the RAMEM water cooled and computer controlled collimator has been moved to another beam line where it is of better use, and it has been replaced by two sets of manually controlled collimators. The first collimator placed just at the entrance of the beam line and a second, where the current can be measured independently (up, down, left and right), placed just before the experimental set-up. This has improved significantly the optimization of the incoming beam.
ACCELERATOR AND ION BEAM STATISTICS

The accelerator at CMAM, designed and constructed by High Voltage Engineering Europa (HVEE), was the first Coaxial High Current Tandetron Accelerator of 5 MV using the Cockcroft-Walton power supply system (previously, terminal voltages were never higher than 3 MV with this system and the power supply itself was perpendicular to the acceleration stage). It has a remarkable stability and low ripple (less than 50 V at 5 MV). Since November 2003, the accelerator tank has not been opened for service, and still the maximum high voltage value can be reached routinely.

Two ion sources, sputtering and duoplasmatron, are used to provide almost any element from Hydrogen to Lead (Fig. 1).

Figure 1. Ion sources, accelerator tank and magnetic elements of the 5MeV Cockroft-Walton tandetron accelerator

In 2010, all the thermoleaks used for the duoplasmatron source were replaced by Mass Flow Controllers (MFC), allowing for a much more stable, faster to stabilize, and easier to control gas flow. Besides, a new cylinder containing nitrogen has been added, being possible to generate NH₂⁻ beams, directly from the source.

A few new beams (namely Pb and Fe) have been extracted from the sputtering source, being Si, Br, F, C and Au the most demanded ones (see Fig. 2).

During 2010 and 2011 we have worked mainly with He and H (over 76% of the time), including ³He and ⁴He, but the demand of heavier ions has been continuously increasing, as can be seen in the Figure 3.

In 2010 we had 1335 hours of beam on target, dedicated either to experiments or for commissioning of new beamlines. In 2011, we reached 1452 hours, showing we have achieved an
operational limit. Besides, we continue slightly reducing maintenance time, as well as failures during operation (see Fig. 4).

**Figure 4** Left: the percent of shifts dedicated to maintenance. Right: the beam shifts per year.

**BEAMLINES**

Four beamlines are available by the end of 2011: Standard Beamline (STD), External Microbeam (EuB), Time of Flight (TOF) and Nuclear Physics Beamline (NUC). Although less every year, Standard beamline has continued being the most demanded beamline, mainly for classical Ion Beam Analysis (IBA) techniques, but also for Ion Beam Modification of Materials (IBMM) over small areas.

The development of new beamlines has required a certain amount of beam time for commissioning, in particular the Internal Microbeam (IuB) and the Implantation line (IMP). Figure 5 shows the distribution of beamtime use for 2010 and 2011.

Detailed information about the beamlines offer of the center can be found below.

**EXTERNAL MICROBEAM LINE**

*Responsible scientist: A. Climent-Font*

This line is mainly dedicated to studies of our Cultural Heritage. It is based on a focused microbeam produced by a quadrupole doublet (Oxford Microbeams) with high demagnification and extracted in air through a ultralinear Si$_3$N$_4$ (silicon nitride) window (200 or 150 nanometers thick) resulting in a routinely usable ion beam of nearly Gaussian spatial distribution with a FWHM of 40 micrometers. A table provided with computer controlled XYZ movements can accommodate both small samples like fragments and resin embedded sections, and whole objects of medium-large size (up to 30 kg) with a positioning precision of some tens of microns. The set up of the line combines four detection channels: two X-ray detectors for the PIXE technique, a silicon implanted blind detector, with a helium flow, for RBS, and a gamma detector for PIGE. One of the Si(Li) X-ray detectors is used for the determination of the light elements; a helium stream is applied to optimize the detection in the range of 1.0 – 12 keV. The second one is used to measure the trace elements. The particle detector is positioned in Cornell geometry for carrying on RBS analysis. For the PIGE analysis a HPGe detector can be installed temporarily for measuring very light elements like Li, Be, Na, Al or Si. The line can be provided also with a lanthanum bromide detector which has the same detection efficiency of the HPGe one but is much more compact and can offer a larger solid angle and a resolution which is excellent, amongst the scintillation detectors. Recently lonoluminescence measurements have been made possible by incorporating to the set-up a quartz fiber that collects the ion induced luminescence (or IL) spectrum of the samples from 200 nm to 1000 nm and analyses it via a compact spectrometer (Ocean Optics QE-6500).

The IL has been used so far mainly for the identification of mineral aggregates in ceramic objects. The line has also been fitted with a new nozzle to be used for a relative charge measurement, detecting the X-rays produced in the Si$_3$N$_4$ window with a dedicated compact detector (Amptek). CMAM is grateful to Brice Moignard of the C2RMF of Paris for his assistance in the design of this nozzle. The counts/charge calibration is still ongoing since it requires special care at low beam currents (<500 pA).

**STANDARD LINE**

*Responsible scientist: A. Muñoz-Martín*

Installed together with the accelerator in 2002 by HVEE, the Standard Beamline at CMAM is a multipurpose line, mainly used for “classical” IBA techniques as well as IBMM over small areas.

It has an experimental chamber equipped with a 4 axis goniometer, a HPGe detector for gamma rays and two implanted silicon detectors (one fixed and one movable) for charged particles detection. For the movable detector it is possible to define different solid angles and to put absorbent foils at its entrance. All this equipment, together with a continuously improving set of acquisition and control programs allow us to make Rutherford Backscattering (RBS), Elastic Recoil Detection and Analysis (ERDA), Particle Induced Gamma Emission (PIGE), Nuclear Reaction Analysis (NRA) and RBS-channeling experiments.

Besides, the beamline can be used for irradiating samples with a large variety of ions, from H to Au, being possible to cover areas up to 8x8 mm$^2$.

A high sensitivity optical camera and a special viewport for far-infrared (thermal) camera, complete the offer of available instrumentation of the beamline.

**Figure 5.** The annual accelerator use and distribution of beamtime per experimental line.
TIME OF FLIGHT LINE

Responsible scientists: A. Muñoz-Martín and Victor Joco

By the end of 2009, the Time of Flight (ToF) beamline at CMAM was completely assembled and the first tests were performed in 2010.

ToF beamline is at the 10º port of the first switching magnet at the exit of the accelerator, allowing for high mass, low charge state ions. In the beam line, two sets of four independent slits are located in order to define the beam spot on the sample, which is positioned by means of a 3-axis goniometer inside the scattering chamber. The incident beam current is measured continuously during the experiment by means of a Transmission Faraday Cup situated at the entrance of the chamber. A Time of Flight telescope, placed at 40º from the beam, collects the particles, measuring in coincidence both energy and time of flight for each particle. In this way it is possible to distinguish the mass of the detected particle.

To measure the time of flight, two time stations are placed inside the telescope. The length of flight between them can be adjusted, being 42cm the nominal value. When a particle crosses a time station, a fast signal is generated. This signal is used to feed Fast Preamplifiers and Constant Fraction Discriminators that determine when a particle is passing through. A time to amplitude converter is used to obtain a pulse with a height proportional to the time of flight between the two detectors. At the end of the telescope, a solid state detector measures the particle energy. Both time and energy signals are recorded in a list mode, marking every event with a timestamp. Further software treatment allows determining which events occurred in coincidence, making possible mass determination for each detected particle. For heavy particles, energy spectra obtained form time spectra are with much higher resolution than the ones obtained directly from energy detector.

LABORATORIES

Besides the accelerator CMAM has laboratories equipped for the preparation of materials and their characterization before and after their irradiation.

SAMPLE PREPARATION LABORATORY

This laboratory is dedicated to the preparation and manipulation of a broad range of samples, including biological ones. It is equipped with:

- Two extraction chambers
- Reactive closets
- Ultrasounds cleaners
- Acid pumps
- Elix water purification system, with ion-exchange resins
- Laminar flow station
- ATA Brilliant 250 diamond saw
- Carbolite 1200 °C electric oven
- Marssen 15 tons press
- Kern 770 Analytical balance (1 mg resolution)
- Mettler Toledo MX5 Microbalance (1 microgram resolution)
- Nikon SMZ800 Binocular Microscope
- Nikon Eclipse ME600 Binocular Microscope
- Telstar Cryodos Freeze dryer
- Leica CM 1510S cryotome
- Emitech Sputter Coater
- Emitech CA7625 Carbon accessory
- Struers RotoForce 4 polisher
- ATA Brillant 250 diamond saw
- Carbolite 1200 °C electric oven
- Marssen 15 tons press
- Kern 770 Analytical balance (1 mg resolution)
- Mettler Toledo MX5 Microbalance (1 microgram resolution)
- Nikon SMZ800 Binocular Microscope

COATING TECHNOLOGY LABORATORY

The CMAM has film growth facilities by a dual Magnetron Sputtering system (Fig. 6).

SAMPLE CHARACTERIZATION LABORATORY

This laboratory is equipped with:

- A Wotlam FLS300 Spectroscopic Ellipsometer for 190-1300nm range. Designed to work “in situ” connected to the irradiation chambers with special flanges (Fig. 7)
- A Veeko Dektak 150 mechanical profiler

OPTICS LABORATORY

It is equipped with the following items:

- Andor optical spectrometer with 3 grating
- Compact fast spectrophotometer Ocean Optics QE65000 that allows:
- Home-made Reflectance and Transmittance measurement set-ups, for the wavelength range 200-900 nm. Precise determination of linear optical properties (n, k)
- In-situ lonoluminescence during ion irradiation (sensor of defect creation)
- Pulsed Spectra Physics ns laser at 532, 355 and 266 nm for further processing crystal with photonic useful patterns (lines, gratings, spots)
- Micro-translational stages
- Optical detectors (Si PD, PMT, etc)

LABORATORY OF MICROSCOPY (LOMA)

- Surfaces laboratory with Scanning Tunnelling Microscope (STM)
- A Nanotec Electronica AFM system equipped with scanning modes (contact, non-contact and PLL - Phase Lock Loop - tapping mode) and KPM (Kelvin probe mode) for charge/potentials detection

MOSSBAUER SPECTROSCOPY LABORATORY

It is set in the accelerator vault (radiation controlled area) for the study of samples containing isotope $^{57}$Fe.
Irradiation and implantation

Personnel responsible: David Jimenez-Rey, Angel Muñoz-Martín

A ion implantation and radiation beam line is being developed at the CMAM, combining Ion Beam analysis (IBA), Ion Beam Modifications of Materials (IBMM) techniques, and optics studies. The main aim of the implantation and irradiation beamline (IMP) is to perform homogeneous irradiations in large areas, up to 10 x 10 cm$^2$. A reasonable homogeneous (static) beam is readily obtained, with dose control, using an electrostatic commercial Beam Sweep System made by High Voltage Engineering Europa (HVEE). This raster has four independent faraday cups (4FC) for control of the ion dose. Several irradiation geometries are possible using the 4FC system. Furthermore the IMP beam line has a cooled FC and a Beam Profile Monitor (BPM) for controlling the shape and homogeneity of beams.

Table 1. Ion beam, currents, and isotopes characterized under the beam-sweep system of the IMP beamline

<table>
<thead>
<tr>
<th>Beam</th>
<th>Charge state</th>
<th>Maximum Beam Current (nA)</th>
<th>Energies (MeV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^1$H</td>
<td>1</td>
<td>3000</td>
<td>2-10</td>
</tr>
<tr>
<td>$^4$He</td>
<td>1-2</td>
<td>-</td>
<td>2.4-5</td>
</tr>
<tr>
<td>$^{28}$Si</td>
<td>1-10</td>
<td>1480 [+3]</td>
<td>4.1-22.4</td>
</tr>
<tr>
<td>$^{35}$Cl / $^{37}$Cl</td>
<td>2-7</td>
<td>160 [+3]/200 [+3]</td>
<td>12.2-32.5</td>
</tr>
<tr>
<td>$^{35}$Br</td>
<td>3-10</td>
<td>12.3 [+3]</td>
<td>20.55</td>
</tr>
<tr>
<td>$^{197}$Au*</td>
<td>2-6</td>
<td>64 [+4]</td>
<td>6.1-14.2</td>
</tr>
</tbody>
</table>

The line has a length of 6 meters, this distance is necessary for scan 10x10 cm$^2$ with H$^+$ at 10 MeV (the most restrictive case).

Several scanning configurations with an H$^+$ at 2 MeV and 1 µA can be observed in Figure 8. The maximum ion beam focalizations, obtained with the quadrupole of the CMAM, are shown in Figure 8c. This beam can be focused to just a few mm$^2$, without slit collimations. Even so, motorized slits for high power have been installed in the second magnet HE2. These slits are necessary with ion beams with high magnetic rigidity.

A large irradiation chamber (Fig. 9) has been designed and optimized for Materials Science and Technologies research with the following main characteristics:

- Compatible with ultra high vacuum operation (all flanges are built as CF).
- Compatible with advanced optical measurements, in particular, with Andor Optical spectrometer and Woollam Ellipsometer, available at CMAM and capable of in-situ measurements. Specific ports at 75º are installed for that aim.
- Compatible with pulsed laser excitation (Nd:YAG at λ (nm) = 266, 355, 532, 1064), through glass/silica windows.
- Compatible with load-lock loading system for transfer of 3 inches wafers between vacuum chambers (via "vacuum suitcase") without air exposure.
- Provided with solid state detectors for RBS and ERDA measurements.
Figure 9 CAD Design of the IMP vacuum chambers from different lateral points of view. In a) the 4FC and insulated ceramic can be observed.

Nowadays, the experimental vacuum chambers (principal and 4FC) are under commissioning, in collaboration with CIEMAT. The current status is shown in the Figure 10. The coupling between the beam line and experimental vacuum chamber will be held during 2012.

Figure 10 Pictures of the current status of the IMP (left), and vacuum chamber (right).

UHV-SURFACES BEAMLNE

Development plans for the UHV-surfaces beamline concentrate on improvements of the performance of the LEIS-ToF system (ion source, beam production and detection, etc.), the installation of detectors for experiments of the standard ion-beam techniques using the high-energy ions provided by the CMAM accelerator [Rutherford backscattering spectroscopy (RBS), elastic recoil detection (ERDA), etc.], as well as the completion of the load-lock system for sample introduction and transfers, which will allow the full use of the preparation subsystem equipped with several Knudsen cells containing different elements.
PIXE and PIGE techniques to study the Roman glasses of Duratón (Spain)

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Abstract

Close to the village of Duratón in the Segovia province (Spain) is the archaeological site of "Los Mercados", where have been disclosed the remains of an urban Roman settlement, perhaps the city of Confluenta mentioned by Ptolomeus. Recent field surveys [1-2] tried to define the real extension of the site and the controversial chronology (1st to 3rd century A.D.) of its occupation. The survey in a gridded area (about 80 ha) based on an intensive surface collection of material, like pottery, tegulae, mud bricks, metal and glass, was chosen for a spatial and functional analysis of the site. PIXE and PIGE simultaneous measurements where performed at our external micro-beam line [5] on 56 glass fragments of different shapes and colours to quantify major (Na, Si, Ca), minor (Mg, Al, K) and trace elements (Ti, Mn, Fe, Co, Ni, Cu, Zn, As, Sr, Sb and Pb). A beam of 3 MeV protons, with a current of a few nA and a diameter of 100 microns FWHM was delivered on the target placed 3 mm downstream the 0.2 mm-thick SiN exit window. The small beam diameter allowed limiting to 1mm² the surfaces that had to be polished with the sand. In Figure 2 a linear correlation of slope 0.2 (R² = 0.75) between the first two oxides has been suggested [12]. The last group (full diamonds, despite they have compositions close to the natron type group but with higher magnesium content) have a chemical composition close to one another and close as well to those already reported to plant-ash glass [9]. Not all the Duratón glasses have been produced with the same flux. The calcium oxide was provided in Roman times by calcareous coastal sand or shells and the elements titanium, iron and aluminium are associated with the Duratón glass used MnO as decolourant (Fig. 4). MnO is a component of the plant ash, used for this in the 19th century we suppose a modern origin for this last group. Some of the samples employed a mixture of manganese and antimony and the glasses with low manganese content were discoloured by arsenic. Since arsenic is normally included in the glass recipes of the 19th century we suppose a modern origin for this last group.

The analysis confirms that the majority of glasses from Duratón are of Roman type, as one would expect from a site dated mainly from I to II AD, and are not dissimilar from the many that have been studied and reported in literature. In a site where the material studied and reported in literature, it is not surprising to have found glasses of the plant ash type and also some which can be, by the purity of the main components and the use of arsenic as decolourant, classified as modern.

References

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Study of a glazed ceramic lunette belonging to Buglioni’s workshop

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³ Restaurator, Art Novae of Emanuele Ioppolo

The so called Della Robbia glazed terracotta sculptures are a form of art developed in a few Florentine workshops during the Renaissance. The glaze on surface was capable to confer these works of art durability, brilliant colours and elegance and produce their remarkable commercial success. For this reason all the subtleties and secrets of this art were jealously protected to guarantee the “monopoly” in its production [1].

The recent restoration of a glazed lunette made by Benedetto Buglioni gave the opportunity to study the constituent materials and the execution techniques, providing a further insight into the activities of Benedetto during his stay in Bolsena.

The lunette (fig. 1) sits on the portal of the Oratory of St. Leonard, annexed to the Renaissance façade of the Basilica of Santa Cristina in Bolsena (Viterbo province, Italy) and is part of a group of eight works conserved inside and outside the building [2,3]. It was commissioned by the then cardinal Giovanni De’ Medici, son of Lorenzo the Magnificent and future Pope Leo X and was made between 1493 and 1494.

The lunette is inserted in the tympanum of the Oratory’s portal and features St. Leonard between two devotees. The Saint is portrayed half-length and presents his traditional iconographic symbols: the fetters of the prisoners and the Gospel. St. Leonard is represented in the guise of a young deacon but the presence of tassels on his dress could allude to his future Episcopal Mission.

The two devotees are smaller compared to the Saint. They are identified as members of the Brotherhood of the Flagellates; in fact they are fitted with a hood and hold in their hands the scourge, an instrument of passion. As usual in this kind of artifacts, the modeler has divided the object into five pieces to avoid any deformation of the panel during the drying and firing.

Due to the limited structural stability of the artifact, it was necessary to temporarily remove the lunette from its seat for restoration [4] and this gave access to lots of information (tool traces, fingerprints, etc.) to understand the execution technique used by the artist. The artist probably created the relief by modeling (not...
sodium increase the brightness of the surface. The lead component respects the percentage of feldspars. Aluminum oxide contributes to opacifying the glaze and the oxides of potassium and the other elements give the well worked and purified clay a nearly uniform thickness of 3-4 cm. The ceramic molding) figures on a single sheet of clay supported by wooden boards. The back was emptied to 20% of SnO₂. The other contaminates are vitreous covers rich in lead and tin, being silica the element that forms the glass, lead the major element (Fig. 5) indicated that the measurements SCO 5a and SCO 5b are quite cer want to investigate. From the concentration of major elements its evident that the brown layer as seen from the CaO content of the order of 30%. Looking at the minor elements (Fig. 4) we find the higher percentage of PbO (40%) which should have resulted in a glaze brighter in line with the compositions of the time. The blue color is obtained through a cobalt mineral, Co being present in oxide concentrations between 0.42 to 0.70% associated with Fe, Ni and Cu. The use of blue with arsenic is documented only from 1520 onwards [14]. In the blue glazes of the lunette, in addition to the normal proportion of silica, tin and lead, there are quite homogeneous glazes with a vitreous phase well separated from the biscuit by an interphase with abundant “dark” secondary crystals that assure the adhesion to the support [6].

The chemical elemental analysis was conducted non destructively using the PIXE technique [7] on fragments; no polished section has been analyzed or scanned by PIXE. The extraction of oxide concentrations has been done with the GUPIX package and the reliability of the spectra deconvolution has been checked with the NIST standard 621 [8-10]. The clay used by Benedetto Buglioni was rich in carbonate (around 20% of CaO, figure 2), as was typical of Della Robbia’s workshop and his competitors, in order to achieve a clear colour and dilatometric properties similar to those of the glaze to enhance its brilliance [11, 12]. The glaze analysis was made complex by the fact that we analyzed raw glazes and that the samples were, as said, erraticaly collected. This caused that some of the PIXE analyses on fragments of glazes came out to concern materials different from the glaze itself, so we wanted to investigate. From the concentration of major elements its evident that the brown layer (Fig. 3) measured in SCO 1a and SCO 1b-2 is not from a glaze since in both the Si, Pb and Sn content is too low. The measurement SCO 1b-2 is quite certainly associated to some calcite layer as seen from the CaO content of the order of 30%. Looking at the minor elements (Fig. 4) we find the sample SCO 1a has very large amounts of metals Mn, Fe, Cu, which associated to the low amount of Si, might indicate a paint instead than a glaze. In the white samples the analysis of major elements (Fig. 5) indicated that the measurements SCO 5a and SCO 5b are quite cer clearly associated to some calcite layer as seen from the CaO content exceeding 25. An extended optical microscope examination of polished cross sections showed quite homogeneous glazes with a vitreous phase well separated from the biscuit by an interphase with abundant “dark” secondary crystals that assure the adhesion to the support [6].

The recent restoration work made on the relief of St. Leonard opened the possibility to start a detailed study of the lunette and also to confirm, thanks to the analytical approach, that the production features strongly already associated with the Florence workshop of Buglioni, were maintained during his stay in Bolsena. In spite of the limited financial resources, he was able to compete with the rival workshop of the Della Robbia, offering a lower quality but still appealing product. The way Buglioni worked is reflected in the Bolsena production and in the execution technique of the lunette of St. Leonard, which shows little attention to details. This suggests the hurry and the urgency that animated the workshop, probably due to the pressure that an important order such as that of Cardinal Giovanni de’ Medici, future Pope Leo X, produced on the artist. BIBLIOGRAPHY
Technological characterization of pre-Hispanic gold metallurgy has a wide deficit of analytical data upon which to build a synthesis, comparable to that made for ancient Europe. Our research tries to fill the gap between the well established stylistic classifications and the scientific identification of the production processes. Our concern is mainly with gold and tumbaga alloys, lost wax technological processes, and depletion gilding.

The Museo de América (Madrid) keeps an important collection of prehispanic gold, the main part of which comes from Costa Rica (Fig. 1) and Colombia. The so called Quimbaya’s treasure (Fig. 2) is a funerary set made up of 123 gold objects (originally near 200) dated between 500BC-600 AC. It was found in two tombs in La Soledad, near the municipality of Finlandia (Department of Quindio, Colombia) and after its exhibition in Madrid during the 4th Centenary of the Discovery of America in 1892, it was offered as a present to the Queen regent of Spain by the president of the Colombia Republic.

In the frame of a three years research project funded by the Spanish Ministry of Science and Innovation (Ref.: HAR2009-09298) we have designed a strategy for the study of the gold alloys, manufacturing techniques and surface treatments combining three analytical methods: SEM, PIXE-RBS and XRF, in order to measure the thickness of the gold enriched surface layer, and to allow access to high resolution, high magnification topographic images when possible. At the CMAM accelerator we have completed, in three phases (November 2010, may 2011 and December 2011), the IBA analysis (PIXE and RBS) of 108 pieces for a total of 494 measurements (including standards) which means the characterization of a significant portion of the collection from Costa Rica and the Quimbaya treasure. Despite the problems produced by the abrasive cleaning methods, used during the long history of the collection, that have eroded the gilded layer almost to its disappearance in more than one case, we know from our preliminary results that it is possible to determine the metal composition and the sequence of layers in all objects, which gives information on the manufacture technology and the type and purity of the metals. Although the quantification of all the analyses (a total of 1500 spectra) and the integration of IBA results with those obtained by other analytical techniques and by the topographic study of the objects is in progress and will evidently take time to be completed we can give a few examples of the study of the objects and draw some preliminary conclusions.
The PIXE analysis gives for example convincing evidence of the jewellery technique adopted in the construction of an earring that functions as well as a rattle (Fig. 3). The piece is cast using a lost wax casting technique that leaves in the inside of the piece a sphere of about 6 mm diameter. The comparison of the composition of the sphere and that of the outer surface of the earring puts in evidence (Fig. 4) that the casting has been made from a gold-copper alloy whose composition corresponds to that of the sphere and then the earring has been treated by an acid attack that has removed copper from the outer surface leaving it much richer in gold.

As regards the composition of gold for the two largest groups analysed in this project: Costa Rica and Quimbaya, we have performed a quantitative PIXE analysis only on a small subset of samples at 3 MeV proton energy (Fig. 5, 6 and 7).

The RBS data analysis and the quantitative PIXE analysis at 5 MeV proton energy are still in progress. It must be observed that during the data taking, the PIXE spectra appeared to be remarkably constant for the Costa Rica set with only a few exceptions, the same did not occur for the Quimbaya treasure. Preliminary data point to depletion gilding as a standard finishing process in the Costa Rica production, resulting in a gold rich surface alloy, with gold correlated to Ag and anti-correlated to Cu. This is the case of Qimbayas’s treasure piece 17436 (Fig. 8) (see photograph). The RBS spectrum shown obtained with 5 MeV proton energy is representative of spectra measured in other points of the object.

After analysis of the RBS spectrum shown in the (Fig. 9) adjacent figure, using the RBS simulation program SIMNRA [1], the composition depth profile can be obtained. The figure shows the relative atomic composition values for the main elements gold, silver, and copper, showing clearly an enrichment of Au with a very thin layer, about 500 x 10^15 gold atoms/cm^2 (that is about 90 nm) of pure gold, followed by a transition region, with Au, Ag and Cu, stepwise modelled with a first step 6500 x 10^15 atoms /cm^2 (about 1.1 µm), a second step 48 000 x 10^15 atoms /cm^2 (about 8.3 µm), where the Cu increases slightly while Au and Ag both decrease, and finally the bulk region with the alloy composition 43 % gold, 25 % silver, and 32 % copper, (Fig. 10).

We can conclude after this study that Quimbaya objects show, on their side, a variety of raw materials and technologies, from almost pure gold to tumbaga alloys, in masterly lost wax castings of big size, complex objects.

References

Modeling and simulation of the interaction of swift heavy ions with dielectric materials

F. Agulló-López in various collaborations with:
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3 Laboratory of Synchrotron Light (Cells-Alba), Barcelona, Spain.
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The group at CMAM has continued and extended its research activity in those areas during the period of the report and it has focused the attention on the following topics:

1. Non-radiative exciton model for point-defect generation

Based on a suggestion by Itoh and Stoneham a theoretical model for damage and amorphization by swift-heavy ions (SHI) in LiNbO3 was proposed in ref [1]. It relies on the non-radiative decay of localized (possibly self-trapped) excitons, triggered by the synergy between the thermal and excitation spikes generated after the swift ion impact. The model was suggested to apply, also, to TiO2 [2] and KGd(WO4)2 (KGW) in accordance with experimental observations. During the present two-year period we have been describing the application of the model to NaCl as a typical alkali halide crystal where the existence of excitons and their role on the damage by purely ionizing irradiation (UV light, X-rays) is well ascertained. The model has been applied to understand data obtained by the group of prof. M. Toulemonde in the Polytechnical University in Madrid.

2. Ionoluminescence Modeling

Stimulated by the novel and interesting ionoluminescence (IL) phenomena observed in CMAM on the amorphous (silica) and crystalline (quartz) phases of SiO2 [4], we are developing a theoretical model aiming to describe the kinetics of the two main light emissions (1.9 eV and 2.7 eV). Our analyses have suggested that the two emissions are generated as a consequence of STE formation and recombination in Si-O bonds. The model developed by us proposes that the relative intensity of the two emissions is determined by the strain state of the Si-O bonds at the STE’s. For quartz, where bonds are initially unstrained, the main emission is the red band at 1.9 eV. During ion-beam irradiation the intensity of this emission decreases in favour of the blue emission at 2.7 eV that becomes dominant at high fluences. In fact, it has been proposed that the blue emission can be considered as a sensitive sensor to monitor the quartz amorphization during irradiation (see Fig. 1). On the other hand, for silica the bonds are heavily strained and the dominant emission throughout the whole fluence range occurs at 2.7 eV. However, we have obtained possible evidence for the formation of a metacmic amorphous phase (similar to that generated by ion-beam irradiation of quartz) from the evolution of the IL spectra with fluence.

3. Stress-strain halo around amorphous tracks in LiNbO3

The stress-strain haloes around an amorphous track in LiNbO3 have been calculated taking into account the real symmetry of the crystal lattice (trigonal C3m symmetry). The calculations assume an isotropic approximation for the strain fields but use the full elastoplastic tensor to calculate the stress profiles [5]. The stress-strain fields drop off with distance from the track axis as 1/r2, i.e. much faster than for a crystal dislocation. In a second stage, a method is developed to approximately account for the effects of crystal anisotropy for tracks oriented along the principal crystal axes (X, Y, Z). The results provide justification for some experimental observations related to the role of crystal anisotropy on irradiation effects. They include the dependence of radiation-induced disorder and magnitude of surface swelling on crystal cut, fracture patterns and morphology of pores after chemical etching.

4. Kinetics of amorphization: MonteCarlo and analytical models

The theoretical model proposed by us to understand the formation of single tracks by ion impacts predicts, in a natural way, their complex structure including a central amorphous core surrounded by a defective halo. Considering such structure we have put forward theoretical tools to describe the amorphization kinetics as a cumulative process associated to track overlapping. On one side, we have developed a MonteCarlo approach [6] that takes into account the statistical (random) spatial distribution of ion impacts on the crystal surface. It has been applied to LiNbO3 and it has been able to illustrate the comparison between sub-threshold and above-threshold irradiations as shown in the accompanying Figure 2. Moreover, by computing the overall generated amorphous areas we have obtained a good representation of the Avrami-type kinetic curves determined through experiment (Fig. 3).
On the other hand, we have, also, developed an analytical model that assumes that the superposition of a fixed number $N$ of haloes turns the crystal into an amorphous state. In spite of its simplicity, it describes many relevant features of the kinetic behaviour, both for $\text{LiNbO}_3$ and crystalline $\text{SiO}_2$ (quartz).

5. Trapping of micro and nano-particles by PV fields in $\text{LiNbO}_3$

In collaboration with the group led by prof. M. Carrascosa in the Department of Physics of Materials (where the experiments have been performed) we have developed a theoretical model to calculate the evanescent photovoltaic (PV) fields generated by visible illumination at the surface of a crystal plate. These fields have been shown to allow for trapping and patterning of micro- and nano-particles on the surface and even provide a tool to kill cells for cancer therapy.

References


Measurement of Activity Produced by Low Energy Proton Beam in Metals Using off-line PET Imaging

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External beam radiotherapy using protons has been used extensively for more than forty years. Protons show an increasing energy deposition with the penetration distance, giving rise to the maximum of the energy loss –the Bragg peak– close to the end of the range of the protons. This physical feature causes an advantage of proton treatment over photon or electron irradiation since the region of maximum energy deposition can be well positioned within the target for each beam direction. This opens up the possibility of achieving a highly conformal high dose region, created by a spread-out Bragg peak (SOBP), and thus the potential of covering extended tumour volumes with high accuracy and low collateral damage to healthy tissue.

The most promising method for in vivo and non-invasive monitoring of proton radiotherapy is positron emission tomography (PET). During the proton irradiation, positron emitters, such as $^{13}$C and $^{15}$O, are produced by nuclear interactions along the proton beam path, and can be imaged as a spatial imprint of proton trajectory. Due to the energy threshold of the proton beam of about 15 to 20 MeV for the creation of such isotopes, it is not possible to obtain an image of the treated region, where protons have a much lower energy.

![Figure 1 Denenzo-inspired pattern activated with the proton beam at CMAM](image1.png)

![Figure 2 PET reconstruction of the Denenzo-inspired pattern, 2 hours after irradiation (first frame of the acquisition). Without range correction (left) and with range correction (right)](image2.png)
However, other so-called metal β\(^+\) isotopes, suitable for PET imaging as well, can be produced at lower energies than \(^{11}\text{C}\) and \(^{15}\text{O}\) via proton induced reactions on appropriate targets. Some of these alternative PET nuclides are isotopes of Ga and Cu. If a given tumor-specific molecule is labeled with the target isotope for these reactions, the interesting β\(^+\) PET isotopes will be then produced in proton therapy by protons reaching the target volume with low energy.

In 2011, we investigated PET imaging with \(^{68}\text{Ga}\) and \(^{66}\text{Ga}\) after proton irradiation on a natural zinc foil. Profiting from the low energy reaction threshold for production via \((p,n)\) reactions, both \(^{68}\text{Ga}\) and \(^{66}\text{Ga}\) gallium isotopes were produced by activation on a natural zinc target by a proton pencil beam. We created detailed Derenzo-inspired patterns (Fig. 1) with a proton beam produced by the accelerator at CMAM. The energy of the beam (up to 10 MeV) is similar to the residual energy of the protons used for therapy at the distal edge of their path. The activated target was then imaged (Fig. 2) in a small animal PET/CT scanner and reconstructed with a fully 3D iterative algorithm, with and without positron range corrections.

Resistivity Measurements at Cryogenic Temperatures
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In the context of development of advanced radiation resistant materials for application in fusion reactors, measuring the irradiation-induced changes to the microstructure provides the link between irradiation and mechanical and other bulk property changes. Fundamental knowledge about defect production, their diffusion and clustering is required. Computational simulations are one of the most important tools used nowadays for understanding of radiation effects in materials.

We are facing the analysis of structural materials, whose main candidates in fusion technology field are ferritic-martensitic steels. At present, simulations are powerful enough to study pure materials or very dilute alloys. Groups dedicated to modelling of defect kinetics have combined \textit{ab-initio}, kinetic Monte-Carlo (kMC) and rate theory (RT) methods to reveal information on radiation effects of pure irradiated iron\(^{1,2}\). Modelling codes require some model experiments which may be able to validate input parameters, such as migration energies, which are usually taken from different computational approaches and sometimes may not be accurate enough.

Resistivity Recovery (RR) measurements provide indirect information on population and mainly dynamics of vacancies, interstitials and their clusters. Samples are ion-irradiated at cryogenic temperatures where defects are “immobile”. Subsequent thermal annealing reveals important information about defect kinetics. This method has been used in the past by other researchers using electron irradiation but still it is not clear its capability and scope. The goal of our work is to study this analysis technique and evaluate its possibilities of reproducibility and reliability, to study its limitations and define the information that it can contribute.

Resistivity Measurements at Cryogenic Temperatures experiment has been commissioned in 2010 as a result of a collaboration CMAM/CIEMAT, and its development has continued at Center of Micro-Analysis of Materials (CMAM) along 2011.

The experimental set-up basically consists of a continuum-flow cryostat which permits the accommodation of temperature sensors, resistivity measurement connections and heating devices. A sample has thickness of about 30 to 70 microns and it is placed at the end of the cold-finger of the cryostat. Samples studied are typically pure Fe, and Fe alloys Fe10\%Cr and Fe14\%Cr. Heating Thermocoax device is placed in the sample holder. And temperature sensors are inside the cold finger as well as on the sample holder near the sample. During operation, the cooling is done by continuous aspiration of liquid nitrogen (LN), from a large storage vessel through a flexible vacuum-insulated transfer line. Cryostat design and LN vessel allow replacing the storage without warming the samples and thus continuous operation\(^3\). Figure 1 is a picture showing a general view of the cryostat with the sample mounted. For enhance the cooling down a thermal radiation shield is mounted over the cold finger and ensuring collimation of the ion beam.

Throughout 2011 continuous improvements on the experimental system have being developed:
- Type E thermocouple, 1 mm diameter with steel sheath and mineral insulation was replaced by two type T thermocouples (0.123 mm diameter) with PFA (Fluorocarbon Polymer) insulation. New thermocouples, unlike the former type E which was placed touching the sample-holder, are welded directly to the samples which are ~50 µm thickness.
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- Brass spring-like connection system was replaced by spot-welded wires system with a small pin intermediate connector (Fig. 1) which has improved and simplified the process of removing and replacing the sample.

- It was installed a relays system which makes possible switching of electrical connections to a second position in such a way that resistivity measurements can be done by using Van der Pauw method. Also with this purpose there was added an extra step on the sample preparation process which consisted of 4 spark-erosion cuts (see Fig. 1). Van der Pauw method is better for measuring samples with small surface and circular geometry given that it diminishes wire welding effects and delimits the measurement area and thus the irradiation area.

- Irradiation, tests and measurements were possible cooling the set-up with LHe. In order to manage the flow of LHe through the continuum-flow cryostat, aspiration system was replaced by a pressure injector system in LHe dewar.

General view of the upgraded set up can be seen in Figure 2.

The experimental procedure for RR type experiments is as follows: system is refrigerated with liquid helium (LHe), minimum temperatures reached in sample are about 20 K and rise up to ~50 K during irradiation with 4.5 - 5 MeV protons and currents between 5 - 40 nA. Sample thickness (30 - 70 microns) determines energy (implantation should be avoided) and thus its increase in temperature during irradiation determines the current. Thickness is very difficult to control at this scale with the method used for preparation which is mechanical polishing. After irradiation the sample is subjected to annealing steps and its resistivity is measured after every step at irradiation base temperature (~50 K). Resistivity of the material should increase with the radiation and decrease as the annealing temperature rises (RR).

Along 2011, 7 irradiations were carried out, 2 of them were crucial for defining and testing set-up improvements and from the remaining 5 we have been able to get experimental results related to the resistivity recovery. Irradiated materials and its dose are shown in the Table 1.

![Figure 1](image1.png)

**Figure 1** Frontal view of the sample holder, housing a ~50 µm iron sample with the four spark-erosion cuts, the 4-wire Van der Pauw configuration, the spot welded thermocouples and the new intermediate pin connector.

![Figure 2](image2.png)

**Figure 2**. Experimental set-up at the end of ERDA-TOF line.

<table>
<thead>
<tr>
<th>Material</th>
<th>Thickness (µm)</th>
<th>Eproton (MeV)</th>
<th>Ibeam (nA)</th>
<th>Dose (10^-6 dpa)</th>
<th>Fluence (cm^-2)</th>
<th>RR (Ω cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe 1 (plate)</td>
<td>38.0 ± 0.6</td>
<td>4.5</td>
<td>38</td>
<td>220</td>
<td>7.9E15</td>
<td>(52K) - 250 (33K) - 240</td>
</tr>
<tr>
<td>Fe10Cr (EFDA)</td>
<td>52.0 ± 2.0</td>
<td>5</td>
<td>39</td>
<td>170</td>
<td>7.5E15</td>
<td>(52K) - 720 (33K) - 800</td>
</tr>
<tr>
<td>Fe10Cr (EFDA)</td>
<td>49.2 ± 0.4</td>
<td>5</td>
<td>35</td>
<td>220</td>
<td>9.1E15</td>
<td>(54K) - 546 (33K) - 667</td>
</tr>
<tr>
<td>Fe14Cr (EFDA)</td>
<td>65.8 ± 0.4</td>
<td>5</td>
<td>10</td>
<td>83</td>
<td>3.6E15</td>
<td>(54K) - 363 (33K) - 400</td>
</tr>
<tr>
<td>Fe 2 (plate)</td>
<td>23.6±0.1</td>
<td>4.5</td>
<td>21.2</td>
<td>23</td>
<td>0.92E15</td>
<td>(54K) - 196 (22K) - 180</td>
</tr>
</tbody>
</table>

**Table 1.** Experimental results of the Resistivity Recovery at cryogenic temperatures

The derivative of resistivity recovery with respect to temperature shows peaks referred to as recovery stages. For some stages the change-of-slope technique allows to deduce effective activation energies of defects in solids. Real experiments just measure resistivity before and during annealing as explained. Computational simulations calculate the number and kind of defects produced during irradiation and associate a certain increase in resistivity to each defect (Δρi), the sum of all Δρi leads to a resistivity curve as a function of temperature. Afterwards derivation of this curve provides the information on recovery stages. Real experiments are complemented by computing simulations and the other way around.

**References**

Commissioning of a new setup for PIGE technique application

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Centro de Micro-Análisis de Materiales

The extended use of Particle Induced Gamma ray Emission (PIGE) as a routine technique for quantitatively characterizing materials is, presently, no possible due to the lack of accurate measurement of gamma emission cross sections.

A Coordinated Research Project of the International Atomic Energy Agency, (IAEA) aims to create a nuclear database containing cross sections for the most commonly demanded PIGE reactions. The Center for Micro-Analysis of Materials, is participating in this project aiming at measuring a selection of those cross sections, in view of future analytical applications.

Experimental work has been dedicated to preparing a specific setup for serial cross section measurements and incorporated in a master thesis.

To achieve the requested measurement precision implies a detailed characterization of the experimental setup and all its parts and measurements protocols. For this reason, several measurements for the gamma ray detectors efficiency and resolution determination, accelerator absolute beam energy calibration, and characterization of the target conditions have been carried on.

To calibrate the accelerator in energy the well known resonances at 991.74 keV [1] and 1316.83 keV of the nuclear reaction \(^{27}\text{Al}(p,n){}^{28}\text{Si}\) have been used. The gamma rays are emitted with an energy of 9-12 MeV. The yield was of gamma rays from 3-8 MeV detected by a HP-Ge detector and a LaBr\(_3\) detector, and normalized to the total charge in the sample. The result of this new calibration was the following:

\[
V_{\gamma}\text{max} = A + B V_{\text{acc}}\text{eV}
\]

\[
A=4.5\pm0.6, B=1.018\pm0.001\text{ keV}
\]

Figure 1 Vacuum chamber developed and used for this work.

References


SCIENTIFIC RESULTS

Shower approach in the simulation of ion scattering from solids

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The results of the final phase of the development and testing of the computer program TRIC (Trajectories of Ions in Crystals) has been published in Ref.[1]. An efficient approach for the simulation of ion scattering from solids, including the calculation of energy spectra and angular distribution of scattered and recoiling particles, has been proposed. A high efficiency is crucial to achieve accurate structural determinations of materials by means of comparisons with measured data. The essential novel idea of TRIC is to take, for every atom encountered by the ion, multiple samples of its thermal displacements among those which result in scattering with high probability to finally reach the detector.

As a result the detector is illuminated by intensive “showers”, where each event of detection is weighted according to the actual probability of the considered atom displacement. The computational cost of such simulation is orders of magnitude lower than in a direct approach and a comprehensive analysis of multiple and plural scattering effects becomes possible. The method can successfully applied for any combination of crystal lattice and ion species with energies for which the scattering potential and the electronic energy loss are sufficiently well known and the binary collision approach is applicable. The present strategy bears similarities with the method of VEGAS Monte-Carlo integration of multi-dimensional functions as proposed by G. P. Lepage et al. [2], based on importance sampling. Here sampling points are concentrated in the regions that make the largest contribution to the integral.

The program TRIC produces output that is equivalent to that of programs like Marlowe [3] or other “brute force” approaches, but at a speed that is a factor of tens of thousands higher. Energy spectra of scattered or recoiled particles measured in any geometry for a given atomic arrangement can be well reproduced. This implies that also angular distributions of scattered or recoiled particles sampled over a defined energy range can be reproduced. Such distributions reflect the arrangement of atoms in the crystal structure. Unknown arrangements of atoms can be determined by comparing measured angular scans with simulated scans for a large number of trial atomic arrangements in the crystal. The program has been shown to work properly for low, medium and high energies of incoming ions [1].

We have used the new method for two main purposes [1]. First, the accuracy of approximate approaches, developed mainly for ion-beam structural analysis, is verified. Second, the possibility to reproduce a wide class of experimental conditions is used to analyze some basic features of ion-solid collisions: the role of double violent collisions in low-energy ion scattering; the origin of the “surface peak” in the scattering from amorphous samples; the low-energy tail in the energy spectra of scattered medium-energy ions due to plural scattering; the degradation of blocking patterns in 2D angular distributions with increasing depth of scattering. As an example of simulation for ions of MeV energies, we verify the time-reversibility for channeling/blocking of 1 MeV protons in a W crystal. The possibilities of analysis that our approach offers may be very useful for various applications in particular for structural analysis with atomic resolution.
In the figure above is illustrated the principle of selection of the “hot” region of atomic displacements used in the shower generation. The density of the Gaussian distribution of atomic displacements is represented by the gray cloud while the “hot” region is indicated as the tube enclosing the region of impact parameters for scattering into the chosen angular cone (left panel). Analogously, the picture at the right panel illustrates the generation of showers of recoiled atoms. \( W_i \) and \( w_i \) are the weights ascribed to the trajectories of primary and secondary ions, respectively, while \( P_i \) designates the integral probability of an atom displacement into the “hot” region.

References


Effect of the lattice misfit on the equilibrium shape of strained islands in Volmer-Weber growth

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Nanotechnology is currently an active research field due to the exciting properties of materials when system sizes approach the nanometer scale. One way to fabricate nanostructures is the spontaneous development (self-assembly) of three-dimensional islands when growing a thin film epitaxially on a crystalline substrate with a substantial lattice mismatch between the two materials. Here it is essential to understand the key factors controlling island sizes and shapes. After several works published on coherent quantum dots grown on a wetting layer of the same material, we have studied the effect of the misfit on the equilibrium shape of three-dimensional pyramidal islands grown on a foreign substrate in the case of incomplete wetting (Volmer-Weber mode of growth) [1]. By means of atomistic simulations using anharmonic interaction potentials, we find that tensile islands have smaller aspect ratios compared with compressed islands owing to their better adhesion to the substrate. The average strains of consecutive layers decrease faster with thickness in compressed than in tensile islands. The strains decrease rapidly with thickness, with the consequence that above a certain height, the upper layers of the pyramid become practically unstrained and do not contribute to a further reduction of the top base. As a result, the truncated pyramids are not expected to transform into full pyramids. Our results are in good agreement with experimental observations in different systems [2-5].

References

Second-layer nucleation in coherent Stranski-Krastanov growth of quantum dots

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In order to control the mechanisms behind the formation of epitaxial islands which lead to the formation of self-assembled nanostructures, it is important to understand the key factors controlling island nucleation. By means of atomistic simulations using anharmonic interaction potentials, we have studied the mono-bilayer transformation in the case of the coherent Stranski-Krastanov growth \([1]\). We have found that the energy of formation of a second layer nucleus is largest at the center of the first-layer island and smallest on its corners. Thus nucleation is expected to take place at the corners (or the edges) rather than at the center of the islands as in the case of homoepitaxy. This is confirmed by experiments \([2-5]\). Furthermore, we have found that the critical nuclei have one atom in addition to a compact shape, which is either a square of \(i^2\) or a rectangle of \((i^2 - 1)\) atoms, with \(i > 1\) an integer. When the edge of the initial monolayer island is much larger than the critical nucleus size, the latter is formed always by a rectangle plus an additional atom, adsorbed at the longer edge, which gives rise to a new atomic row in order to transform the rectangle into the equilibrium square shape.

In the figure above are shown locations of second layer nuclei. From top to bottom and from left to right: initial 20x20-atoms, monolayer-high island; 13-atoms second-layer cluster nucleated at the terrace center, at an island edge and at an island corner of the initial monolayer island.

The color scale denotes the height of the considered atom. The height is biggest at edges and corners due to the atoms “climbing up” on their neighbours underneath due to strain relaxation. The lattice misfit is -7%.

In the Figure are shown heights of the nucleation barriers as a function of the value of the lattice misfit (main plot: positive misfits; insert: negative misfits). The figures at each point denote the number of atoms in the critical nucleus. A cluster size of 400 atoms was considered.

References

\[1\] J.E. Prieto and I. Markov, Phys. Rev. B 84, 195417 (2011)
Optical waveguides obtained by swift-ion irradiation on silica (a-SiO₂)

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The preparation of optical waveguides for visible light in silica (a-SiO₂) by means of swift heavy-ion irradiation (F at 5 MeV, Cl at 20 MeV) was reported in an article presented in REI-2009. Swift heavy-ion irradiation originate from the refractive index enhancement (compaction) brought about by the electronic excitation. Their main features are discussed in relation to those prepared by light ion irradiation (H, He) and implantation. In particular, irradiation fluences to achieve waveguiding may be considerably reduced. On the other hand, the measured refractive index profiles have been used to discuss the mechanisms of electronic damage. Moreover, it is shown that the measurement of the index profiles provides a novel optical method, alternative to IR spectroscopy, to estimate the radius of the irradiation-induced tracks.

Electronic damage in quartz (c-SiO₂) by MeV ion irradiations: Potentiality for optical waveguiding applications

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The damage induced on quartz (c-SiO₂) by heavy ions (F, O, Br) at MeV energies, where electronic stopping is dominant, has been investigated by RBS/C and optical methods in this work. The two techniques indicate the formation of amorphous layers with an isotropic refractive index (n = 1.475) at fluences around 1e14 cm⁻² that are associated to electronic mechanisms. The kinetics of the process can be described as the superposition of linear (possibly initial Poisson-type) and sigmoidal (Avrami-type) contributions. The coexistence of the two kinetic regimes may be associated to the differential roles of the amorphous track cores and preamorphous halos. By using ions and energies whose maximum stopping power lies inside the crystal (O at 13 MeV, F at 15 MeV and F at 30 MeV) buried amorphous layer are formed and optical waveguides at the sample surface have been generated.

References:
Recrystallization of amorphous nano-tracks and uniform layers generated by swift-ion-beam irradiation in lithium niobate

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2 Instituto de Fusión Nuclear, Universidad Politécnica de Madrid, Spain.
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4 Instituto de Optica Daza de Valdés, Consejo Superior de Investigaciones Científicas, Madrid, Spain.

The thermal annealing of amorphous tracks of nanometer-size diameter generated in lithium niobate (LiNbO3) by Bromine ions at 45 MeV, i.e. in the electronic stopping regime, has been investigated by RBS/C spectrometry in the temperature range from 250 ºC to 350 ºC. Relatively low fluences have been used (< 1e12 cm-2) to produce isolated tracks. However, the possible effect of track overlapping has been investigated by varying the fluence between 3e11 cm-2 and 1e12 cm-2. The annealing process follows a two-step kinetics. In a first stage (I) the track radius decreases linearly with the annealing time. It obeys an Arrhenius-type dependence on annealing temperature with an activation energy around 1.5 eV. The second stage (II) operates after the track radius has decreased down to around 2.5 nm and shows a much lower radial velocity (See Fig. 1 Left). The data for stage I appear consistent with a solid-phase epitaxial process that yields a constant recrystallization rate at the amorphous-crystalline boundary. HRTEM has been used to monitor the existence and the size of the annealed isolated tracks in the second stage (See Fig. 1 Right). On the other hand, the thermal annealing of homogeneous (buried) amorphous layers has been investigated within the same temperature range, on samples irradiated with Fluorine at 20 MeV and fluences of ~1e14 cm-2. Optical techniques are very suitable for this case and have been used to monitor the recrystallization of the layers. The annealing process induces a displacement of the crystalline-amorphous boundary that is also linear with annealing time and the recrystallization rates are consistent with those measured for tracks (Fig. 2 left and right). The comparison of these data with those previously obtained for the heavily-damaged (amorphous) layers produced by elastic nuclear collisions is summarily discussed.

Figure 1 Left. Arrhenius-type plot of the radial velocity data for the stage I for the three studied fluences indicated in the figure with their corresponding activation energies.

Figure 1 Right TEM images of a LiNbO3 sample irradiated with Br 45 MeV ions at a fluence of 3e1011 ions/cm2 after annealing in air for 30 min at 275 ºC. b) High resolution image shows that the radius of the tracks (of about 2.5 nm) are consistent with data obtained from RBS/C.

Figure 2 Left Disordered fraction obtained from RBS/C versus annealing time for the fluences a) 3e1011 cm-2, b) 5e1011 cm-2 and c) 1e12 cm-2, and for the temperatures 250, 275, 300, 325 and 350 ºC, as indicated in the figures labels. The insets show esquematic illustrations of the covered area, for each of the three fluences, simulated with random impacts of estimated track radius of 4.4 nm.

Figure 2 Right Evolution of the track radius with annealing time for the fluences a) 3e1011 cm-2, b) 5e1011 cm-2 and c) 1e12 cm-2, and for the temperatures 250, 275, 300, 325 and 350 ºC, as indicated in the figures labels. The track radius is obtained for each case from the corresponding disordered fraction (shown in Fig. 2 Left) as described in the text assuming a linear dependence of the cross-section o a track multiplied by the impact density.

References

He(He,γ)Be, cross sections by measuring the induced γ radiation

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In collaboration with University of York and The Weizmann Institute in Israel, we have performed measurements at the Nuclear Physics Beam Line in order to determine the cross section of the He+He → Be reaction as a function of energy. The technique used in the experiment is the activation method which consists of detecting the delayed 478 keV γ-ray from the first excited state in 7Be. The activation is the method used in the detection of the delayed radiation. We use a 3He beam impinging onto a 4He gas target. The set-up is shown in the figure below.

Two complementary techniques are used to determine the incoming beam particles: through the ions detected in a silicon detector placed at 45° and by integrating the incoming charge. The 7Be recoils were collected in a Cu catcher, and the subsequent gamma delayed radiation is measured off-line using a low-background HPGe detection station. We have thus determined the astrophysical factor for different center of mass energies. Our main results are shown in red in the figure below. A paper summarizing the present results is being sent to the journal Phys. Rev. Lett.

Figure 1: Left photo: sample holder ready for being mounted into the irradiation chamber. Graphite sample is fixed to the specially designed sample holder made of golden quartz for the SQUID; in turn it is attached to a setup made of MACOR designed to fit into the irradiation chamber. Right photo: quartz (foreground) and graphite sample (background) already mounted in the irradiation chamber. A rotary feedthrough allows placing them at the irradiation position from outside of the chamber without breaking vacuum.

Magnetic properties of graphite irradiated with MeV ions

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In this work, we have exhaustively studied the change in the magnetic properties produced on highly oriented pyrolytic graphite (HOPG) samples after irradiation of H-, C- and N- ions in the MeV energy range. The use of specially made sample holders for the magnetic measurements through a SQUID magnetometer provided high reproducibility, allowing us to obtain directly the irradiation effects without any corrections or subtractions (Fig. 1). Our results show (Fig. 2) that three main magnetic phenomena are triggered by the defects produced by the irradiation, namely: paramagnetism, ferromagnetism and an anomalous paramagnetic state. The induced ferromagnetism appears when the average vacancy distance is around 2nm in the near surface region.

Figure 2: Paramagnetic parameter C from the Curie law, obtained from the fits to the magnetic moment difference data as a function of the nominal vacancy number produced by the irradiation for all measured samples. The labels show the sample number and the label "V" in brackets means that sample shows induced ferromagnetism by the irradiation. The point with the label "V" refers to the samples in virgin, nonirradiated state, with similar volumes. The points from the samples 513-515 have a larger error in the C values because of the different misalignment in the SQUID holder since the samples were taken out of the holders for the lower temperature irradiation. The continuous line is the function $C=0.075 \times 2.08 \times 10^{-15} (N_{V} + 6 \times 10^{16})$. The dashed and dotted lines follow the same function but with the first numerical coefficient equal to 0.085 and 0.065, respectively.

SCIENTIFIC RESULTS
The main conclusions of this work are:

- Ferromagnetic states can be induced independently of the ion used, in agreement with published results in literature. However, the results presented in this study indicate that there is a rather narrow window of parameters where this effect can be triggered using MeV ions. Apart from heating effects during irradiation this narrow window is probably related to the mean vacancy distance and the high hydrogen concentration at the near surface region. For the samples where this state was induced, a linear temperature dependence of the ferromagnetic moment is found in agreement with ferromagnetic excitations in a quasi-2D lattice.

- Heating effects during irradiation appear to be important and can induce a decrease in the paramagnetic as well as ferromagnetic initial states of the samples. Self-heating, the further relaxation of defects and hydrogen diffusion at room temperature are some of the reasons for the small yield of ferromagnetic mass using ion irradiation at MeV energies.

- The Curie-like paramagnetic contribution increases proportional to the nominal induced vacancy number with an effective Bohr magneton number \( \mu_B = 0.27\pm0.02 \).  

- Heating during irradiation appears to be important and can induce a decrease in the paramagnetic as well as ferromagnetic initial states of the samples. Self-heating, the further relaxation of defects and hydrogen diffusion at room temperature are some of the reasons for the small yield of ferromagnetic mass using ion irradiation at MeV energies.

- The Curie-like paramagnetic contribution increases proportional to the nominal induced vacancy number with an effective Bohr magneton number \( \mu_B = 0.27\pm0.02 \).
Porous silicon micropatterns as a tool for cell migration research

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Irradiation of crystalline Si wafers with MeV high-energy ions has been shown to increase its resistivity up to the point where porous silicon (PSi) growth is inhibited. Thus, the localized irradiation of Si allows a precise patterning of PSi at the microscale. Due to the unique chemical properties of PSi and its ease of biofunctionalization by different means, these structures can result in patterned contrasts of surface chemistry and/or hydrophobicity, which are of special relevance for cell migration studies. In the present work, PSi/Si micropatterns have been fabricated using He and Si MeV ion beams, and their viability as microstructured substrates for cell adhesion and proliferation assays has been studied.

Structural characterization shows that PSi patterns with precisely defined geometry can be fabricated by copper masks for MeV ions. PSi growth is well defined, both laterally and in-depth, at the sub-micrometer scale. Preliminary culture assays with human mesenchymal stem cells (hMSCs) show that cell behavior is strongly affected not only by the pattern design and size, but also by the type of ion used for the patterning of PSi. The experimental results suggest that this kind of PSi/PSi structures have a great potential for cellular assays, since they provide a reliable procedure, structurally and chemically customizable patterned substrates for such studies.

Irradiation of materials with MeV energy ions has been used in the last decades to synthesize new materials and to induce luminescent or magnetic properties. Irradiation on insulators also changes the optical properties of the damaged area, being possible to make waveguides and optical devices based on waveguides. The resistivity of crystalline silicon, and hence its electrochemical properties, are also affected by ion irradiation, up the point where growth of PSi in the irradiated area is inhibited. Defocused MeV ion beams have been used in combination with inexpensive cooper masks to create micrometric PSi/Si patterns, and their use as microstructured substrates for cell migration research has been explored.

Boron doped p-type <100> crystalline silicon wafers, with a resistivity of 0.05-0.1 Ω•cm, were irradiated using the accelerator facility at CMAM with 20 MeV Si and 1 MeVHe ions. Implantation fluence was set at $10^{14}$ ions/cm² for both type of ions in order to inhibit PSi formation. The ion beam was defocused to achieve a homogeneous irradiation area of about 3x3 mm² defined by a set of slits. Commercial TEM copper grids with different motifs were used as masks to selectively cover/expose the crystalline substrate to the ion beam. After irradiation, the copper grids were removed and the samples were anodized in HF-based solutions to obtain nanostructured porous silicon layers, following a well established procedure.

Figure 1 shows a typical SEM image of a PSi/Si micropattern consisting of 35 micron wide PSi stripes with a 60 micron pitch. It can be observed that the fabrication process results in well-defined PSi areas both on surface and in-depth.

Figure 2 shows fluorescence microscopy images of hMSCs cells cultivated on striped micropatterns formed with a 1 MeV He beam with different PSi/Si width ratios. hMSCs are preferentially located on Si areas for micropatterns consisting on 100 µm wide Si stripes and 25 µm wide PSi stripes. However, the adhesion behavior of hMSCs was found to dramatically change by reducing the width of the Si stripes down to 40 microns and the width of the PSi stripes to 20 microns. In this case, the actin skeleton (in green in figure 2) tends to preferentially locate on Si areas, although the nuclear environment (in blue) is preferentially located on the PSi areas. The absence of hMSCs localized in PSi areas for wide Si stripes and the inhibition of focal adhesion formation on PSi for any pattern suggest that these surfaces behave as antifouling platforms.

Figure 2 Fluorescence microscopy images of hMSCs cultivated on PSi/Si micropatterned substrates. Cell proliferation behavior is strongly influenced on the PSi/Si width ratio (fluorescence of the PSi stripes is also visible).
Modification of the electrochemical properties of crystalline silicon by MeV Si implantation

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It is known that irradiation of silicon with H and He ion beams in the MeV energy range modifies the electrochemical properties of the material, as is evidenced by the inhibition of porous silicon growth in the irradiated areas \([1]\). MeV ion beams of heavier elements with higher stopping force will be more effective in the modification of the physical and chemical properties of the material under irradiation. We have studied this alteration effect of the electrochemical properties of crystalline \(<100>\) silicon p-type substrates using MeV Si ion beams with the electrostatic accelerator of CMAM facility. Thus, besides irradiating the material with an ion heavier than H or He, the ion is of the same elementary nature as the irradiated material, ruling out the possibility of ascribing any contribution of the observed effects to the implanted species. A drastic resistivity increase of the irradiated silicon substrate is observed for growing implantation fluence, which can be pointed out as the mechanism responsible for the inhibition of pore formation (Fig. 1). This change in resistivity may be attributed to defects in the crystalline lattice produced by the irradiation producing new energy levels in the gap of the semiconductor that might act as traps for the charge carriers and also the deactivation of dopants \([2]\).

![Figure 1](image1.png)

**Figure 1.** Evolution of the I-V characteristics through the Si samples under different fluences.

![Figure 2](image2.png)

**Figure 2.** He 3 MeV channeling spectra of a Si control sample (black) and a Si irradiated sample (red). See text for details.

The damage on the crystalline structure of silicon produced by ion irradiation has been studied with ion beam channeling at CMAM. Figure 2 shows the channeling spectrum performed with 3 MeV He on a Si sample irradiated with 5 MeV Si ions with a fluence of 5x10\(^{13}\) atom/cm\(^2\) (red spectrum). For purpose of comparison, the channeling and random spectra of a reference Si sample, that is non-irradiated, are also shown. With a 3 MeV He channeling experiment the depth probed by the He beam and shown in the spectrum is about 3.2 µm, while the implantation range of 5 MeV Si into Si is of about 3 µm. Therefore the damaged region can be seen in the spectra in the channel region from 50 to 150. It can be noticed that in this region the spectrum of the irradiated sample is slightly higher than the control one and can be attributed to displaced atoms in the irradiated sample due to the nuclear stopping force.

![Figure 3](image3.png)

**Figure 3.** Planar TEM image of irradiated Si showing a region oriented \(<110>\). The inset shows the corresponding diffraction diagram.

![Figure 4](image4.png)

**Figure 4.** The mask pattern, 40 µm spacing copper grid, is transferred to the substrate as porous silicon.

This work is in progress and recent experiments at higher fluencies with channeling back-scattering experiments made with 4 MeV He are confirming these first results.

TEM experiments on Si irradiated samples show patterns of orientation different from the original \(<100>\), namely \(<110>\) as shown in the planar TEM image in Figure 3 or the corresponding diffraction pattern (Inset Fig. 3).

Patterns of localized porous silicon have been obtained irradiating Si samples covered with TEM copper grids as masks. Porous silicon growth is precisely confined to non-implanted areas (Fig. 4), producing abrupt porous silicon/silicon lateral interfaces. These patterns of several µm in size, and with marked different physical properties, can affect the dynamics of biological cell growth.

In conclusion, the irradiation of Si with MeV Si ions at fluencies as low as 5x10\(^{12}\) /cm\(^2\), can inhibit the subsequent formation porous silicon. Broad MeV silicon beams can be very effectively used to form lateral micro sized patterns with different electrochemical properties using appropriate masks. The decrease in conductivity necessary to inhibit the formation of porous silicon may not imply complete amorphization. Point defects may deactivate or passivate the dopants. Other properties induced by Si irradiation (wetting ability or resistance to chemical etching) can be exploited as well for surface functionalization of Si. Of interest in biomedical applications.

References


Irradiation damage response of fused silica for fusion devices

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As it is known amorphous SiO2 is an important material widely used in different technology fields like optics, electronics, nuclear or space. Optical and electrical properties of silica are strongly influenced by defects, which are introduced during the manufacturing process or produced by energetic photons (UV light, X-ray or γ ray) and/or particles (ions, electrons or neutrons)1.

The optical degradation (radiation induced optical absorption and light emission) imposes severe limitations on the use of any optical material within a radiation field. In fusion devices fused silica is a key element for optical diagnostic and remote handling components (windows, fibers). High purity fused silica with different OH content: KU1 (high OH) and KS-4V (low OH), highly radiation resistant and considered as reference materials in ITER , were irradiated with Helium, Oxygen and Silicon ions at several fluences and also a commercial silica (Infrasil 301) with higher metallic impurity content (see Fig. 1). To study the fluence effect and correlate with defect creation induced by ion irradiation, the irradiations were performed at several fluences by: O ions at 13.5 MeV energy, Si ions at 24 MeV and He ions at 1.7 and 2.5MeV energy.

The ionoluminescence (IL) spectrum was measured during ion irradiation in order to study the evolution of different defects. An example of the samples (KU1, KS-4V, I301 and reference) mounted on a sample-holder, together with the luminescence emitted during the ion implantation (right), can be seen in Figure 1.

The accumulated ionoluminescence spectra of the KU1, KS-4V and Infrasil 301 are shown in Figure 2. The IL response provides rich information of damage kinetic evolution in intrinsic and extrinsic defects.

After ion implantation the samples were characterized at CIEMAT by optical absorption. The absorption spectrum was measured in a wide range, from the VUV to IR, and the behaviour of defects (as E’, ODC, NBO-HC, POR) created by the irradiation at different doses was analysed, mainly in the VUV range. An example of the obtained spectra is shown in Figure 3.

The optical absorption measured after O implantation for the three silica grades studied is similar, independent of OH and impurity content of the sample, while it is known that gamma induced optical absorption depends on the material grade2.

References

Irradiation effect in structural EU-ODS EUROFER for fusion applications

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The fusion structural materials will be exposed to high heat loads and high flux 14 MeV fusion neutrons. These neutrons will produce atomic displacement damage and nuclear transmutations atoms (H, He) within the irradiated materials. Accumulation of data on unirradiated condition is sufficiently complete. However, the database on irradiation effects associated with higher He and H generation is still unclear. Reduced activation ferritic/martensitic (RAFM) steel is the leading candidate structural material for theblanket system. In Europe the denominates EUROFER and EU-ODS EUROFER steels have been selected as reference structural materials for the Test Blanket Modules (TBM) for ITER and possibly for DEMO reactor. Both alloys have a basic composition of Fe-9Cr1WVTa with the difference that the EU-ODS EUROFER contains 0.3 % Y2O3.

It is expected that the He transmutated will affect the mechanical properties and dimension stability. These effects are expected to eventually be clarified by IFMIF irradiation experiments, but will have to be estimated from simulation irradiation such as ion irradiation. With this objective a systematic study has been defined to evaluate the He effects on the nano-mechanical properties on reference structural materials for fusion.

During 2010-2011 part of a series of experiments was performed in order to carry out a systematic study of the irradiation effects on structural materials (Eurofer’97 and EU-ODS EUROFER) steels for fusion applications. The studies not only include experimental irradiations with He at different irradiations conditions; irradiations with Fe ions to simulate neutron damage and subsequent ion implantations with He, Fe and H will also be performed. In addition, irradiation and test temperature will be evaluated. After irradiations a detailed microstructural (TEM) and mechanical investigation will be carried out in order to evaluate the hardening behaviour.

Figure 1 Sample holder (left) and geometry of irradiated specimens (right).

Figure 1 Sample holder (left) and geometry of irradiated specimens (right).

Figure 2 He concentration profile from 2 to 15 MeV.

Figure 2 He concentration profile from 2 to 15 MeV.

Figure 3 Load vs nano-indentation depth for the Eurofer 97 steel.

Figure 3 Load vs nano-indentation depth for the Eurofer 97 steel.

Figure 4 Hardness variation in function of He concentration.

Figure 4 Hardness variation in function of He concentration.

In all the cases, hardness increase was observed as compared with the unirradiated average value. The maximum hardness values corresponded to the zones with higher He content. The hardness values fit completely with the simulated helium diffusion profile at all tested loads.

Nowadays, nano-indentation tests are being carried out on EU-ODS EUROFER steel. The preliminary results on irradiated samples show less variation on hardness with respect to the Eurofer 97. In addition, microstructural observations of irradiated samples will be performed by TEM.

Irradiations with Fe ions have also been performed. The damage level was 0.05 and 0.2 dpa.

References
Ion behavior on Lithium-based ceramics for fusion breeding blankets

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The choice of a suitable breeding material for fusion reactors is actually an important issue in fusion. Lithium-based ceramics such as Li₂O, gamma-LiAlO₂, LiSiO₄, LiZrO₃ and Li₂TiO₃ have been considered as candidates for T breeding materials in D-T fusion reactors. The breeder materials have to produce tritium from lithium while fulfilling the following conditions: to release the produced tritium, to possess physical and chemical stability at high temperatures, to display compatibility with other structural components, and to exhibit optimum irradiation behavior. In a fusion environment, irradiation damage will be caused by fast neutrons, energetic tritons and He ions generated during \(^6\)Li(n,He)\(^3\)H reactions. Radiation damage will result in microstructural changes that may affect the properties of the breeding materials, especially the T migration. Then, the study of structural or compositional changes occurring after materials irradiation will be needed to an effective correlation with their physical behavior.

The retention of light atoms, which can be produced by transmutation or introduced by interaction with the plasma, is one of the crucial problems. At least in insulating materials, ionizing radiation induces valence changes and charged vacancies, which modifies their activation energies for diffusion. In addition, the induced damage creates vacancies enhancing also the diffusion. The effect of ionizing radiation on H isotope and He behaviour is one of the main objectives to achieve on lithium ceramic materials.

He diffusion experiments on fusion Li-based candidate ceramics were performed at CMAM experimental set-up (see Fig. 1). Lithium titanate and lithium silicate, pellet-shaped, thinned and polished ceramic samples of about 10 mm in diameter and 200 µm thicknesses were supplied. Ceramic disks will be sintered to achieve a porosity of 10-15% (90-85 % of the theoretical density). These roughly polished samples were implanted at the multipurpose experimental line at the 5 MV terminal voltage tandem accelerator at CMAM facility in Madrid, Spain. \(^4\)He ions with energy of 1.365 MeV and a fluence of 1E16 ions/cm\(^2\) were implanted at room temperature on the ceramic targets. As the \(^4\)He beam energy is too high, the samples were partially covered with a 4 µm thick Al sheet to reduce the depth penetration of the incident beam. The thickness of the metal beam stopper was calculated by the SRIM Monte Carlo code for the ions to project up to about 650 nm. Following the implantation, the sample was characterized by ERDA using 25 MeV \(^5\)Li ions (see Fig. 1). A low beam current (up to 20 nA) was selected to avoid target overheating. The beam spot was 5 x 2 mm\(^2\). Proton backscattering spectrometry (p-BS) was also used for the samples characterization before and after implantation (Fig. 2 Left), using 20-100 uC dose of 2015 keV proton beams with 17 nA. The backscattered ions were collected at 165º and 170º in IBM geometry.

During both He implantation and Si ion characterization an intense luminescence emission was registered (see Fig. 1 and 2). The emitted light was transmitted through a silica window port placed at 45º to the ion beam direction and focused with a 25 mm diameter, 4 cm focal length silica lens into a silica optical fiber of 1mm diameter. The light is guided to a compact spectrometer QE6500 (Ocean Optics Inc) configured with a multichannel array detector for measuring the whole spectrum simultaneously in the wavelength range 200-850 nm with a spectral resolution better than 2 nm (see Fig. 2 Right). The light integration time was varied between 1 and 5 s.
Magnetic field influence in the irradiation with ions at structural fusion materials

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To date, studies of structural and functional materials for fusion have focused on material behaviour as a function of irradiation dose, energy, temperature, etc. However, the behaviour of materials under such conditions when a magnetic field is applied simultaneously is poorly understood at present. In principle, the microstructure and mechanical properties of materials are modified by the propagation of defects produced by irradiation, and it is hypothesized that this propagation may be affected by the strong magnetic fields present in the fusion devices currently under design. Therefore, better insight into the mobility, recombination, clustering or dissociation of defects, has been the aim of the irradiation experiments of Fe and fusion alloys under magnetic field carried out during 2011.

The Fe ion irradiation of Ultra High Pure Fe (UHP-Fe) samples in a magnetic field of 0.8-1 T was carried out. The magnetic field was obtained with a permanent rare-earth magnet and at a temperature of ~173 K (using a liquid-nitrogen cooled cold finger). The main reason for the use of Fe ions on Fe samples is the simulation of the displacement damage produced by neutrons in fusion reaction conditions (neutron fluxes of ~10\(^{19}\) n m\(^{-2}\) s\(^{-1}\) with energy of 14 MeV and a power flux up to 2 MW m\(^{-2}\)). A copper sample holder has been specially designed for these experiments. This sample holder allows irradiating two samples consecutively. One disc shaped sample can be irradiated while submitted to the magnetic field generated by the permanent magnet embedded underneath. The other sample is inserted in a cavity far enough from the magnet to avoid the influence of the magnetic field. A copper ribbon connecting the set-up to an liquid nitrogen (LN) finger guarantees sample low temperature in spite of the incident power deposited by the ion beam during irradiation. The research carried out on the development of a proper Fe ion source has permitted to obtain beam currents as high as 20 to 250 nA with energies of 1 to 10 MeV and charge state of +1 to +3. Some experiments were carried out with different ion energies, currents and charge states with irradiations dose of 3 dpa to 8 dpa. A TEM characterization was carried out post irradiation in the sample with and without magnetic field, in order to compare the damage induce by irradiation.

CEPA the forward End-Cap of CALIFA

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The goal of this work is to test an array of LaBr\(_3\)(Ce) +LaCl\(_3\)(Ce) Phoswich crystals for the front (forward cap) of the calorimeter CALIFA sensitive to gammas and protons of high energy. One of the difficulties in the design and development of the calorimeter for PB is the special requirements of the small angle forward cap. The innovative solution our research team has proposed is the development of a detector consisting in a dual layer of a new generation scintillator material crystals (LaBr\(_3\)(Ce) + LaCl\(_3\)(Ce)) in a Phoswich configuration with a common readout. Monte Carlo simulations show that gamma rays interact with high probability within the first few cm of detector material. By combining two materials, one could distinguish at which depth the interaction happens. The second layer is used to absorb the total gamma energy or if the first interaction takes place there, to veto that event.

We have during 2010-2011 studied the Phoswich approach both with simulations and with experimental tests. The analysis and simulations have advanced considerably and we now have an extensive understanding of the Phoswich and how it can be used. The first prototype was tested first at CMAM for high energy gamma rays, where a standard NIM electronic set-up was used and secondly with high energy protons at the Svedberg Laboratory in Uppsala Sweden.

Figure: The test at CMAM producing high energy γ-rays in the reaction p+\(^{19}\)F → \(^{20}\)Ne+π\(^0\) gamma, the figures show electronic set up and resulting gamma spectrum. Due to differences in light yield, the γ stopped in the LaBr\(_3\)(Ce) show up at twice the channel number.

At Uppsala a low intensity proton beam at 180 MeV was provided by the Gustaf Werner Cyclotron and collimated to a few millimeters. A flash ADC was used to digitize the entire pulse using a 1ns resolution for off-line analysis. The energy spectra obtained is shown in the figure. The spectrum is overlaid in red with the resulting energy spectra obtained from a GEANT4 simulation of the full set-up including beam tube, degrader and Si detector, an excellent agreement is obtained.
As a flash ADC was used and the full pulse shape was recorded; one can make an pulse shape analysis of the data, comparing the full integral of the digitized pulse with that of the tail, as marked in the figure.

Figure: Total energy in Phoswich. Comparison of exp. Data in red with MonteCarlo simulation in blue.

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Figure: The plot on the left depicts $I_{\text{tail}}$ vs $I_{\text{total}}$ for the phoswich detector when irradiated with two discrete proton energies (180 and 150 MeV). Protons depositing the full energy in the phoswich combination, acting as a $E-E$ telescope, correspond to the two main spots. The other patterns seen in the plot all correspond to protons depositing only parts of their energy due to scattering out of or into the active volume. Scattering out of the LaBr, leading to partial energy deposit in the first part, form the line A in the plot. With increasing energy deposition, the line bifurcates at a break-out point where the maximum energy loss in 1st crystal is obtained.

A new CEPA demonstrator, consisting of 4 phoswich LaBr/LaCl clusters optically isolated in one common enclosure, was delivered in December 2011. and we have built a special support in order to be able to use it not only for testing its own characteristics but also as a very efficient gamma ray detector that can be used in several of the experiments.

Figure: The figure shows the CEPA demonstrator mounted as standalone detector that either be used in air or can be inserted into the vacuum chamber in close geometry for the reaction studies at CMAM.

DISSEMINATION ACTIVITY

- Dissemination Activity
- The Fifth GUPIX Training School
DISSEMINATION ACTIVITIES

As a university research centre, the CMAM is fully committed to give support to teaching, training and dissemination of science. Therefore we regularly carry on several activities based on our research infrastructures (accelerator, ancillary equipment, laboratories) addressed to a broad range of persons.

For students of any degree, from undergraduate to master, who come from the Spanish universities accompanied by their professors, we conduct guided visits of our accelerator and experimental areas giving emphasis to the aspects of ion beam materials analysis and modification more pertinent to the topic dealt in their course.

We host in our laboratory scientists and short term students in the framework of international collaborations (e.g. Università della Tuscia, University of the Witwatersrand, …) and schemes (e.g. IAEA, …).

We are actively participating in dissemination events like the nationwide “Science Week” when the CMAM is open for visit to a broad public, from individuals to societies and schools. All year round we have a specific scheme for visits of groups and organizations of which we would like to recall the Programa Universidad para los Mayores (PUMA) of the Universidad Autónoma.

We give special attention to motivating the future scientists and to promoting science within the Universidad Autónoma. In close collaboration with the Department of Applied Physics, directed by prof. Carmen Morant, we have included the practical use of the accelerator among the demonstrative activities organized by different research groups for the high school students of Northern Madrid. Hundreds of students have had a first contact with the scientific world thanks to these initiatives and have expressed their appreciation for the program offered.

In collaboration with the Scientific Communication Unit of UAM, directed by prof. Carmela Cales, we have participated in July 2011 to the “Summer Science Camp”, sponsored by the FECYT (Spanish foundation for Science and Technology). Four groups of seven students each selected from all Spain, spent a whole week at the CMAM in a “hands on” activity that drove them through all the steps of a basic IBA experiment of which they were the principal actors under the guidance and with the assistance of Begoña Arribas, Begoña Gómez-Ferrer, Marta Ramos Rodrigo, David Jiménez Rey, Javier Manzano Santamaría, Salvador Saura and Alessandro Zucchiatti.

We host in our lab students from secondary technical education institutes of Madrid to complete their education with three months professional training stages where they design and execute, under the guidance of our engineers, a technical project, typically in electronics and data logging.

We offer short tutorials on IBA analysis in the framework of training programs managed by the local authorities (e.g. requalification of unemployed people).

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THE FIFTH GUPIX TRAINING SCHOOL

By the initiative of Aurelio Climent Font, the CMAM and CNA (Centro Nacional de Aceleradores), in collaboration with the Universidad Autónoma de Madrid and The University of Seville, have organized from September 27th to September 29th the fifth GUPIX Training School. The event took place in El Escorial (the historical residence of the kings of Spain), 45 km north of Madrid at was hosted by the Guest House “San José”. The school was structured very similar to the schools that took place in 2005 in both Paris and Florence, especially as regards their interactive nature, with the “students” solving PIXE programs on individual computers. The school was run by Professor J.L. (Iain) Campbell, from the University of Guelph, Ontario, Canada, assisted by Dr. Novella Grassi, at the time researcher at the LABEC (Laboratorio Beni Culturali) of INFN Florence. They presented a detailed exploration of the capabilities of GUPIXWIN, and assisted students in analyzing real spectra with GUPIXWIN. Emphasis was put on the many special features of the program which make it a versatile tool for PIXE, but which are sometimes not used to their full capacity. Attendees have worked on their own laptops, using demo copies of the software and have had the widest opportunity to discuss all the details of the spectra deconvolution and to present their ideas for possible improvements to the existing software.

Figure 1 Professor Campbell follows the work of Andres Redondo Cubero during a practical session

Figure 2 The group of participants in the gardens of the San José Guest House in El Escorial
THE CMAM IS CERTIFIED ISO 9001

The CMAM has implanted a Quality Management System (QMS) according the ISO 9001:2008 standard with the aim of making more effective our way of working and achieve a high standard in the communication with the users, in the evaluation of experiment proposals, in the allocation of shifts and the delivery of beams. Our QMS system has been certified by the company SGS until March 2015. It is focused on the "Delivery of Beamtime to the Accelerator Users" and is organized following the scheme below.

The backbone of the system are the Operational Processes which we follow to handle the beamtime requests (Beamtime Assignment PG 400 01), to set the accelerator working plan and deliver beams (Beam delivery PG 200 02) and finally to follow up the delivery, both from the CMAM and the user side (Follow up PG 500 01). In other words all the procedures that begin with the information made available to a potential user on our web page, until the end of the planned experiment, when the user fills a customer satisfaction enquiry, passing obviously through the technical protocols that assure the correct working conditions and the delivery of beam as planned, are documented, certified and constantly controlled by registers and indicators. This is obviously completed by processes that the ISO 9001 norm requires for managing the system and for supporting the operational processes.

The users can find all the information related with our quality policy, the access to beamtime and the CMAM commitments in the documents “Quality Policy” and “Information to the users of beamtime” which are available from our web page (www.cmam.uam.es).

As a result of the implantation of the Quality Management System we have created in-house a users Portal where we receive and process the beamtime requests. The Portal is accessible through our web page and is self-instructing. It detects errors or incomplete parts and it directs the user to all the information he should know at the moment of the application (commitment of the CMAM, requested compliances, evaluation criteria, CMAM contact address for queries and suggestions). The portal is also accessible to our referees who can download the applications and the related documents as well as the information they need about our evaluation criteria, our beam lines and beam features. The referees use obviously the portal to drop their evaluation in such a way that any application is fully traceable by the applicant, the secretary of the beam assignment commission, the referee and the head of our technical division. The communication with the user takes place via e-mail and our web page.

To make the access to our accelerator as easy as possible we are posting on the webpage the calendar of beamtime periods (normally 8 per year) at the beginning of each year so that our users can plan their activities early in advance and in the best possible way.

The communication with our users and their feedback about the perceived quality of our services is fundamental to maintain and continuously improve the CMAM QMS. For that purpose we are regularly conducting Customer Satisfaction Enquiries and handling queries and suggestions through a dedicated mail address calidad.cmam@uam.es.

We are convinced that the implantation of a QMS puts the CMAM at the level of a fully accessible international facility for the analysis and modification of materials. We are proud of the results obtained in the organization of our work in terms of quality and we are sure that our old and new users will appreciate the improvements. We are grateful to all those who have, are and will cooperate with us in the forthcoming years to achieve the best possible quality in the use of our facilities.