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FOREWORD

Celebrating 10 years of CMAM activity
This sixth number of the CMAM activity report series coincides with the tenth anniversary of the inauguration of our centre. An important step in the life of CMAM and a special occasion indeed, that we would like to share with you through these pages.

I would like first of all to express my deep admiration and esteem for all the people who have conceived at the end of the 90’s the idea of giving the Universidad Autónoma de Madrid and Spain a modern laboratory for the analysis and modification of materials with ion beams. To that idea, to their determination to defend and support it, to the tremendous work that they have done to make CMAM a reality in March 23rd 2003, I like to dedicate the appreciation of the whole staff and my personal one.

From the first steps promoted by Fernando Agulló López and Aurelio Climent Font, both former directors of the centre, the CMAM has become, in a few years, a mature, open and highly competitive research infrastructure dedicated to support the UAM, the national scientific community and many international collaborators.

The tenth anniversary is a first important step of an expectedly long walk. The quality of our staff, the infrastructure that we have been capable of building, our excellent accelerator (the world’s first of its kind) makes me believe that the CMAM will not miss the opportunity to continue growing along this path and contribute to maintain the UAM in an internationally recognized position in materials science.

However, the conditions in which we are working today are quite different from those of ten years ago. The general situation of the research appears complex and difficult in the short period. Since four years I have the responsibility and commitment to seek, in the conditions we are given, the growth opportunities that CMAM deserves and requires. Hold the route of development and innovation depends more than ever and in large measure on the ability to act with the force of the organization and the collaboration of all. It is a demanding task which could not be done without counting on the constant support we receive from the UAM’s government and on the extraordinary dedication of the CMAM colleagues that I deeply acknowledge.

We hope to count as well, readers and colleagues, on your attention, encouragement and advice: they are very important and motivating to face the challenges that are awaiting us. Thank you very much indeed for you support.

Alessandro Zucchiatti
CMAM DIRECTOR
About CMAM
The Centre for Micro Analysis of Materials (CMAM) is a research infrastructure belonging to the Universidad Autónoma de Madrid (UAM). It is part of the research axis “Nanoscience and Advanced Materials” of the CEI UAM+CSIC, a campus of international excellence established in 2009 jointly by the UAM and the Consejo Superior de Investigaciones Científicas (CSIC). Its main tool is an electrostatic ion accelerator that, in 2002, was the first in the world to reach 5MV with a coaxial Cockcroft-Walton acceleration system.

CMAM mission is to conduct cutting-edge research in key areas of application of ion beam techniques, such as: Materials Science in general, Microelectronics and Optoelectronics, Magnetism, Nanotechnology, Environmental Science, Biology and Biomedicine, Nuclear Physics, Materials for Energy Production, Archaeology and Cultural 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. As a university centre we also provide advanced technological support to teaching and training activities, at various academic levels and both within national and international schemes.

CMAM is the result of a project financed through the FEDER program and managed, from July 1998 until the official inauguration on March 23rd 2003, by a Technical Committee chaired by Prof. Fernando Agulló López, assisted by an Advisory Committee formed by outstanding members of the Spanish scientific, cultural and academic community.

The experimental equipment consists of the tandem accelerator, 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. It is completed by several beam lines, specialized for various application areas and by a set of ancillary equipments (micro-analytical techniques, sample preparation). The advanced construction parameters of the accelerator confer it characteristics among the most competitive as regards: deliverable ion range, maximum achievable energy (0.8 to 50 MeV), accuracy and stability of energy setting, fast operations and easy maintenance.

CMAM is structured into three divisions: scientific, technical and administrative, to which are associated the staff members and collaborators. The governing body is the Steering Committee that includes the director, the heads of division, the elected representatives of scientific and technical-administrative staff and a Ph.D. student’s representative. The director receives as well support from the Scientific Committee (all the doctors of the centre) and the Centre Council (the whole staff). 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 Rector for Science Policy and Research Infrastructures of the UAM.

CMAM is a fully open infrastructure: the instrumentation and techniques developed are made available also to external researchers to perform studies within the field of materials science. We are committed to the widest possible collaboration with national and international research institutions. Ion beam delivery, the CMAM main activity, is certified according to ISO9001:2008. Our quality management system, deals with the different access schemes, fully open and competitive (thanks to a board of international referees), that are supervised by a commission (CATH) including a users representative.
Organization and People

Centro de Micro-Análisis de Materiales
Organization chart

Director

Vice-Rector Research

Scientific Advisory Committee

Management Committee

Scientific Committee

Centre Council

Operative Structure

Head Technical Division

TECHNICAL DIVISION

Head Scientific Division

SCIENTIFIC DIVISION

CMAM

Deputy Director

Administration Manager

ADMINISTRATION & HUMAN RESOURCES DIVISION

Quality Manager

Laboral Security Coordinator

Responsible of Radiological Protection

Support Structure

Operative Structure
STRUCTURE OF CMAM

DIRECTOR: Alessandro Zucchiatti
DEPUTY DIRECTOR: Ángel Muñoz Martín
HEADS OF DIVISION Beatriz Renes Olalla, Ángel Muñoz Martín, Alessandro Zucchiatti

Divisions
Administration & Human Resources Division

Head: Beatriz Renes Olalla
María Teresa Aparicio Villarroel
Ana Granados Simón
Inmaculada Sierra Martos

Scientific Division

Head: Alessandro Zucchiatti
Fernando Agulló López (Honorary member former CMAM director)
Aurelio Climent Font (Former CMAM director)
David Jiménez Rey (Until May 2013)
David Martín y Marero
José Olivares Villegas
Rafael Pérez Casero (From May 2013)
José Emilio Prieto de Castro
Miguel Ángel Ramos
Mª Dolores Ynsa Alcalá

Technical Division

Head: Ángel Muñoz Martín
Jorge Álvarez Echenique
Marcos Benedicto Córdoba (from June 2013)
Manuel Díaz Hijar (from June 2013)
José Miguel Fernández Ampuero
Victor Joco
Arantza Maira Vidal
Abdennacer Nakbi
Jaime Narros Fernández
Antonio J. Rodríguez Nieva
Individual Charges

ADMINISTRATION MANAGER: Beatriz Renes Olalla
WORK SAFETY COORDINATOR: José Miguel Fernández Ampuero
RADIOLOGICAL PROTECTION: Ángel Muñoz Martín
QUALITY MANAGER: José Miguel Fernández Ampuero

Ph.D. Students
Diana Bachiller Perea                     Javier Manzano Santamaria
Begoña Gómez-Ferrer Herrán               Esther Punzón Quijorna

Master Students
José María González Gutiérrez             Victoria Tormo Márquez

Committees

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

Chair: Prof. Rafael Garesse Alarcón,
Members:
Prof. Ricardo Amils Pibernat
Dr. Jorge García Gómez-Tejedor
Prof. Ragnar Hellborg
Prof. Elías Muñoz Merino
Dr. Carlos Rossi Álvarez
Prof. Leonardo Soriano de Arpe

Vice Chancelor for Scientific Policy and Research Infrastructures, UAM
Chair of Microbiology, Dept. of Molecular Biology, UAM, and associate researcher at the Astro-Biology Centre (CSIC-INTA), Madrid, Spain
Director of the Restoration department of the “Centro de Arte Reina Sofía”, Madrid, Spain
Professor Emeritus of Nuclear Physics, Faculty of Science, Lund University, Lund, Sweden
Superior Technical School of Telecommunications Engineers, Universidad Politécnica, Director of the Institute of Optoelectronics Systems and Microtechnology, Madrid, Spain
Istituto Nazionale di Fisica Nucleare, Padua, Italy
Full professor. Department of Applied Physics UAM. Former Director of the Materials Science Institute “Nicolás Cabrera”, Madrid, Spain
Management Committee

Members:

<table>
<thead>
<tr>
<th>Name</th>
<th>Position/Role</th>
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<tr>
<td><strong>A. Zucchiatti</strong></td>
<td>Director and Chairman</td>
</tr>
<tr>
<td><strong>A. Muñoz Martín</strong></td>
<td>Deputy Director and head of the Technical Division</td>
</tr>
<tr>
<td><strong>B. Renes Olalla</strong></td>
<td>Head of the Administration and HR Division and Committee’s secretary</td>
</tr>
<tr>
<td><strong>Diana Bachiller Perea</strong></td>
<td>Elected spokesperson of the Ph.D. students from February 2012</td>
</tr>
<tr>
<td><strong>J. Manzano Santamaría</strong></td>
<td>Elected spokesperson of the Ph.D. students until February 2012</td>
</tr>
<tr>
<td><strong>J. Narros</strong></td>
<td>Elected spokesperson of the technical and administrative staff until October 2012</td>
</tr>
<tr>
<td><strong>J. Álvarez Echenique</strong></td>
<td>Elected spokesperson of the technical and administrative staff from October 2012</td>
</tr>
<tr>
<td><strong>J. Olivares Villegas</strong></td>
<td>Elected spokesperson of the scientific staff until October 2012</td>
</tr>
<tr>
<td><strong>J. E. Prieto de Castro</strong></td>
<td>Elected spokesperson of the scientific staff</td>
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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 owing a Ph.D. degree; they report directly to the Director. Besides the scientific division staff it includes:

Members:  

<table>
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<tr>
<th>Name</th>
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<tr>
<td>V. Joco</td>
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<td>A. Maira Vidal</td>
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<td>A. Muñoz Martín</td>
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<td>M. Benedicto Córdoba</td>
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</table>

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

Members:  

<table>
<thead>
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<th>Name</th>
<th>Role</th>
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<tr>
<td><strong>Alessandro Zucchiatti</strong></td>
<td>Chair</td>
</tr>
<tr>
<td><strong>José Emilio Prieto de Castro</strong></td>
<td>Secretary</td>
</tr>
<tr>
<td><strong>Ángel Muñoz Martín</strong></td>
<td>Technical advisor</td>
</tr>
<tr>
<td><strong>Ramón Escobar Galindo</strong></td>
<td>Users representative from ICMM-CSIC, Madrid</td>
</tr>
</tbody>
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PEOPLE

Scientific Division

Alessandro Zucchiatti
Director of CMAM

Alessandro Zucchiatti directs the CMAM since December 2009 after having served INFN of Italy from 1975 onwards. He holds a degree in Physics from the University of Genova (Italy) and a Ph.D. from the University of the Witwatersrand (Johannesburg, South Africa). During his career he has been seconded as temporary research associate at the University of the Witwatersrand, as research director at CNRS of France and as visiting professor at Universidad Autónoma de Madrid. He has begun to work in nuclear physics, which has been his field of interest for many years before he progressively moved into ion beam analysis and its applications, in particular to the atmospheric environment and the cultural heritage. In both nuclear physics and IBA he has worked in prestigious international research centres, among which the ESRF, the GSI, the INFN-LNF, the INFN-LNL, the C2RMF and has been responsible for several research projects. He has also maintained a strong and constant interest to teaching; he has given courses at the University of Genoa and the University of the Witwatersrand; has directed international training schools and has supervised students work in nuclear and applied physics.

Fernando Agulló López
Professor Emeritus and Former CMAM Director

Born in Mérida (Spain), got his degree of “licenciado” by the Universidad Complutense de Madrid in 1956. In 1964 he obtained the Ph.D. 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 Autónoma 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 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 Ph.D. students. He has been temporarily attached to several universi-
ties (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.

Aurelio Climent Font
Full Professor and Former CMAM Director

Born in Tortosa (Spain), got his M.Sc. by the Universidad Complutense de Madrid in 1973. In 1979 he obtained the Ph.D. degree by the Universidad Autónoma de Madrid. He started his scientific career at the Department of Applied Physics studying the electrical 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 Geel, Sandia National Laboratories in Albuquerque, Centre de Recherches et Restauration des Musées de France 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.

David Jiménez Rey
Juan de La Cierva Researcher

David Jiménez-Rey 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 Ph.D. thesis, with a FPI Fellowship of the Science and Innovation Ministry of Spain. His thesis, defended at UNED (12 December, 2008), was focused on “Characterization of luminescent materials and its applications to the study of fast ions lost in TJ-II stellarator”, under the supervision of Bernardo Zurro and Alfonso Baciero. This thesis recieved the extraordinary prize of Ph.D. by UNED. As a post doc David joined the TechnoFusión Project, carrying out tasks as feasibility study and project management, 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 research line at CMAM, and in particular focussing on the damage and response of fusion materials by means of ions beams. David developed a new in-situ Ion beam analysis (IBA) for the damage kinetic study under irradiation, based on ionoluminescence (IL). Furthermore he developed the characterization studies of several scintillator materials of the Fast Ion Loss Detector of ITER. On May 2013 he returned to CIEMAT but continues cooperating with the CMAM on energy related research.
José Olivares Villegas
Scientific Investigator

José Olivares is currently Investigador Científico, Instituto de Optica, CSIC, and associated to CMAM-UAM. He received his Ph.D. in Physics from the Universidad Autónoma of Madrid (UAM), Spain, in 1994, for work on the topic of proton exchanged waveguides in lithium niobate. From 1994 to 1996 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 nanocomposites. In 2001 he became Tenured Scientist of CSIC. Since 2003 he is working, in collaboration with the Centro de Microanálisis de Materiales (CMAM) of 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.

Rafael Pérez Casero
Professor

Rafael Pérez Casero has recently joined the scientific staff of CMAM. He is currently professor at the Department of Applied Physics at the Universidad Autónoma de Madrid. He obtained the Ph.D. degree by the Universidad Autónoma de Madrid in 1989 and has developed his scientific activity at the Department of Applied Physics, the Groupe de Physique des Solides de l’École Normale Supérieure de Paris and the Institut de Nanosciences de Paris. His research activity has been principally focused on the study of the mechanisms of ordered growth of complex oxide thin films by laser evaporation and on the growth of organic materials by laser assisted evaporation. Rafael Pérez Casero has also an extensive experience in the compositional and structural characterization of thin films by high energy ion beam techniques, mainly, Rutherford Backscattering Spectrometry and Nuclear Reaction Analysis.

José Emilio Prieto
Professor Under Contract

José Emilio Prieto is currently professor under contract (Professor 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 Ph.D. in Physics from the UAM working in the field of the growth and surface characterization of thin epitaxial 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 spectroscopies, 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 epitaxial growth and the use of ion beams for characterization and modification of materials properties.
Miguel Ángel Ramos

Professor

Born in Madrid (Spain), he graduated in Physics in 1985 and got his Ph.D. degree in 1990, both at Universidad Autonoma de Madrid. Along his doctoral thesis, he developed the first Low-temperature Scanning Tunneling Microscope in Spain (and the third in the world), performing tunneling spectroscopy experiments in so-called High Critical Temperature Superconductors. During a post-doctoral stay in the KFA at Jülich (Germany), he worked on a theoretical model (the Soft-Potential Model) 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. In the last years, his group has mainly studied polymorphic molecular solids made of simple monoalcohols (such as ethanol, propanol, butanol and their isomers) which exhibit glassy phases together with crystalline ones including sometimes orientational disorder, as well as extremely stable glasses, such as hyperaged geological amber or physical vapor deposited indomethacin. Finally, he has also started 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.

Technical Division

Ángel Muñoz Martín

Deputy Director & Head of Technical Division

Ángel Muñoz-Martín got his degree in Physics in 1997 and, since 2002, he holds a Ph.D. 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 infrastructure and local assistance to external users. 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.
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 nine 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 and graphic design projects and IT support for a wide variety of clients.

Marcos Benedicto Córdoba  
Development Engineer

Marcos Benedicto Córdoba got the bachelor’s degree in Chemistry, in 2008, and a Master’s degree in Advanced Materials and Nanotechnology in 2010, both from the Universidad Autónoma de Madrid (UAM). In 2008, he started working at the Instituto de Ciencia de Materiales de Madrid (ICMM-CSIC), obtaining a Ph.D. in Applied Physics in 2012, as a result of his research on the development of the active parts of a new generation of transistors. The Ph.D. thesis was funded by Intel Ireland and defended at the UAM. During 2012, and part of 2013, he teachted as Associate Professor at the Universidad Antonio de Nebrija and as Science Teacher in support academies. Since July 2013, Marcos works as responsible of the implantation beamline at the CMAM accelerator.

José Miguel Fernández Ampuero  
Quality Manager

Born in Madrid (Spain), he got his degree in Geology (speciality geotechnical engineering, geophysics and hydrogeology) in 2002 from Universidad Complutense de Madrid. Since 2002, he has worked in companies connected with civil and quality control engineering (TRAGSA, S.A, FCC, S.A). In 2010-2011 he completed a Master in Geological and Geotechnical Engineering from Universidad Complutense de Madrid. In 2012, José Miguel joined Universidad Autónoma de Madrid, as Quality Assurance Manager and Safety Coordinator of the Centre for Micro-Analysis of Materials.
Victor Joco
Research & Development Engineer

Victor Joco works at CMAM as a Research & Development engineer. He obtained his degree in Physics at Babes-Bolyai University, Cluj Napoca, Romania in 1999, and the Ph.D. in Physics from the Universidad Autónoma de Madrid (UAM), Spain, in 2008. The work in CMAM consists in developing instrumentation for Surface Physics and Ion Beam related methods. Besides, his work is strongly related with the beamline and experimental chamber fine tuning and release to users. He is an expert in different communication protocols, digital and analogue electronics, microcontrollers, detectors, converters, software, vacuum techniques and various scientific methods. He actively participates in training and educational activities.

Arantza Maira Vidal
Technical Support Engineer

Arantza Maira Vidal works at CMAM as technical support engineer. She obtained her degree in Physics at Universidad Autónoma de Madrid (UAM) in 1997 and the Ph.D. in condensed materials physics at Universidad Autónoma de Madrid (UAM) in 2005. Before she started to work at CMAM, she was working in the fields of astrophysics, condensed materials science and nuclear physics. Her work in the CMAM consists in the operation of the accelerator, the supervision of the radioactive installation, the participation in the maintenance tasks related with the accelerator and with the ion sources, the supervisión of the auxiliary installations (as the refrigerated water circuits or the compressed air circuit) and the development and improvement of the accelerator and the complementary equipments. She actively participates in training and educational activities.

Abdennacer Nakbi
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; centering in developing analog and digital electronic circuits and electric installations of the CMAM and also involved in development of beam lines.

Jaime Narros Fernández
Accelerator Technician

Jaime Narros works at CMAM as a technician since November 2001. 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 equipments and beam lines in collaboration with the responsible of the installation and beam lines.

**Antonio Rodríguez Nieva**  
Accelerator Technician

Antonio Rodríguez works at CMAM since December 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 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.

**Ph.D. Students**

**Diana Bachiller Perea**  
Ph.D. 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 Ph.D. 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.

**Begoña Gómez-Ferrer Herrán**  
Ph.D. Student

Begoña Gómez-Ferrer Herrán has a M.Sc. 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 Ph.D. studies in Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas (CIEMAT) in collaboration with CMAM at Universidad Autónoma de Madrid. Her thesis is about studying radiation damage on fusion structural materials, more specifically is based on developing Resistivity Recovery (RR) experiments at cryogenic temperatures which might be able to verify computing simulations in the context of multiscale modeling.
Javier Manzano Santamaría
Ph.D. Student

Javier Manzano got his degree by Universidad Autónoma de Madrid (UAM) in 2003. He started his Ph.D. 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 insulator materials for fusion reactors (ITER). More specifically, he studies damage processes under ion irradiation in the electronic regime in order to clarify the color centers creation processes. He got his Ph.D. in November 2013.

Esther Punzón Quijorna
Ph.D. Student

She is from the land of Don Quixote, Consuegra (Toledo). Graduated in Physics from Universidad Autónoma de Madrid (UAM), she finished the M.Sc. in “Advanced Materials and Nanotechnology” in 2010 at the Applied Physics Department (UAM). She initiated a Ph.D. granted by Ministerio de Ciencia e Innovación (FPI grant), at Applied Physics Department and Center for Micro Analysis of Materials (CMAM) 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 Nanobioscience and techniques of analysis by ion beams. She has completed a research stay for eleven months, as visitor Ph.D. student at the Joint Research Center European Commission (JRC) in Ispra (Italy).

Administration & Human Resources Division

Beatriz Renes Olalla
Administration and Human Resources Manager

Beatriz Renes joined the Universidad Autónoma de Madrid in 1994. She has a wide experience in administration and foreign trade and she has worked for several years as Import/export manager in an American multinational corporation. During the first years at the university she worked as the Administrator assistant at the Faculty of Science. Afterwards she joined the Instituto Nicolás Cabrera, where she was in charge of the institute administration, and also coordinating summer schools, congress and scientific events. In 2002 she was appointed Administration Manager of the Centro de Micro Análisis de Materiales (CMAM), hired by Fundación Parque Científico de Madrid. She is responsible for the entire administration control of the centre, including support for external scientific projects, and she takes care of the auxiliary building maintenance. From 2007 she is also officially in charge of the Human Resources of the CMAM and she supervises the external staff attached to the centre.
Teresa Aparicio Villarroel
Reception Desk Assistant

Teresa Aparicio has been working at the Universidad Autónoma de Madrid for more than 15 years, mainly as general service assistant in different centres and faculties. She joined the CMAM in 2012. Her main work consists on reception tasks, such as switchboard and visitors attention, external and internal mail, parcels and incoming orders registration and keys distribution and control. She also does the control of the office material and equipment and the reservation of the meeting room.

Ana Granados Simón
Administration Assistant

Ana Granados works at CMAM as administration assistant since January 2005. Before that, she was doing secretarial, translation and assistant jobs for years. Her main work in CMAM consists in issuing, keeping updated and filing all the administrative documents of the centre, especially those of the order’s processing system in accordance to the Quality Management System. She gives support and advice to the staff in matters related to their travels to Congresses, and to external visitors, being also in contact with the different departments of the university as well as suppliers. She issues the orders for the general service and maintenance of the building and she also takes care of the library.

Inmaculada Sierra Martos
Reception Desk Assistant

Inmaculada Sierra works at the Universidad Autónoma de Madrid since 2008. Prior to that she worked for private and public companies, doing administrative, reception and lay out works. She joined the CMAM in 2011. Her main work consists on reception tasks, such as switchboard and visitors attention, external and internal mail, parcels and incoming orders registration and keys distribution and control. She also does the control of the office material and equipment and the reservation of the meeting room. Due to her experience and knowledge in lay out works, she actively participates in the graphic design of the activity reports of the CMAM.
10 Years
CMAM CELEBRATES TEN YEARS OF ACTIVITY

The tenth anniversary of the inauguration of our centre fell on March 23rd 2013. An important moment in the life of CMAM, which we celebrated in an two days event that remembered, on the first day, the early stages of the conception and construction of our laboratory and reviewed, on the second day, the evolution of our scientific production in the past ten years. It was also the occasion to meeting for the International Advisory Committee, chaired by the UAM rector, prof. José María Sanz Martínez.

We were delighted to be accompanied by friends and colleagues, quite a few of which had a role in the construction of the centre and the establishment of fruitful scientific collaborations in the whole of our activity.

The first CMAM director, Fernando Agulló Lopez, was the keynote speaker of the celebration ceremony and recalled how the idea of giving the Universidad Autónoma de Madrid and Spain a modern accelerator was born and how the CMAM people were able to create, in a few years an open and highly competitive research infrastructure dedicated to modification and analysis of materials with ion beams.

The vice-chancellor for science policy and research infrastructures of the UAM, prof. Rafael Garesse Alarcón, underlined the role that CMAM plays in the framework of the UAM+CSIC campus of international excellence and its contribution to the Nanoscience and Advanced Materials research platform.

The UAM rector, prof. José María Sanz Martínez made emphasis on the progress that the CMAM was able to achieve in the past ten years and acknowledged the efforts and contribution of all the people who participated in making the CMAM a continuously growing reality, closing the celebration.

On the second day, the CMAM scientist and invited speakers, participated to a special seminars session, dedicated to review our scientific activity and its main results. Some abstracts of those seminars are given in the following pages.
Introduction and Background

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.

We are 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).

Highly Oriented Pyrolytic Graphite

During several years, we have exhaustively studied [2] 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. 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 [2] suggest that the induced ferromagnetism appears when the average vacancy distance is around 2 nm in the near surface region.

Figure 1. Obtained paramagnetic Curie coefficients as a function of the produced number of vacancies by ion-beam irradiation using different ions and fluences.
The main conclusions of this work were:

- 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 \( p = 0.27 \pm 0.02 \ \mu_B \).

- We found a new intermediate magnetic state in samples where the ion irradiation did not induce any relevant ferromagnetic contribution. This state is neither pure paramagnetic nor superparamagnetic. Phenomenologically speaking, its temperature dependence resembles that obtained from the mean-field theory where a “molecular” field proportional to the magnetization is included as well as a “critical” temperature \( T^* \) above which this magnetic contribution vanishes.

![Figure 2. Left: Net magnetic-moment curves at different temperatures, after subtracting the signal before proton irradiation for a HOPG sample. Right: Obtained ferromagnetic saturation magnetic moments as a function of the average distance between vacancies for the different samples studied.](image)
Looking for Magnetism in Diamond

Diamond is a fascinating material, both for fundamental research and technical applications, because of its unique and extreme properties, such as its hardness, chemical inertness, irradiation resistance, high thermal conductivity or transparency to light in a wide frequency range from ultraviolet to infrared. In the last two decades, diamond and diamond-related materials have attracted renewed interest due to findings of surprising properties that may make them also promising materials for electronic applications. Among those very interesting properties, we could mention reported superconductivity at low temperature for boron-doped diamond [3] or ferromagnetism in nanosized diamond particles implanted with nitrogen and carbon ions [4].

We have therefore started a research project to explore the possible existence of magnetic order in diamond, either pure or doped. This project is being performed in collaboration with the group of Prof. Elias Sideras-Haddad at the University of Witwatersrand (Wits) in Johannesburg (South Africa), who provides both synthetic and natural diamond samples. They have also performed preliminary irradiation and magnetic characterization measurements (unpublished) which indicated apparent magnetic behaviour in some samples.

In a first stage, we conducted H⁺ irradiation at 2.2 MeV, with different fluences ranging from $10^{17}$ to $10^{18}$ ions/cm², which have been calculated to provide in diamond an amount of defects' density equivalent to the optimal values found by Esquinazi’s group and by us in HOPG. After the corresponding SQUID measurements to assess the magnetic response of the samples (labeled J1 and J2), they were re-irradiated and measured again to follow the evolution of the possible magnetization with the amount of induced defects.

Interestingly, some ionoluminescence on diamond samples was observed, that helped to monitor the position and size of the ion beam onto the diamond samples. The emission was always blue at the beginning, then it faded away, and finally it turned reddish:

![Figure 3. Evolution of ionoluminescence with proton irradiation in ultrapure diamond.](image)

Later on, two different samples (named B1 and B2) of Ib-type diamond, with ~100-200 ppm of nitrogen impurities, were also studied. In comparison to our earlier findings by irradiating II-a pure diamond, we observed that proton irradiation is more efficient to produce paramagnetic centers in Ib diamond.

In all cases, we subtracted the magnetization of the pristine sample from the irradiated ones to obtain the net magnetization as a function of temperature, and plot as a function of $1/T$ to make a fit to the Curie equation in the temperature range where the paramagnetic behaviour seems well fulfilled:

$$M_{\text{diff}} = C/T + b$$

The obtained Curie coefficients for the different irradiations are shown in Fig. 4.
We summarize now our main results and conclusions.

Several MeV proton-irradiation of II-a pure diamond samples J1 and J2, with different irradiation parameters have apparently produced a significant paramagnetic signal added to the initial diamagnetic behaviour, but with no traces of cooperative ferromagnetism. The Curie-like paramagnetic signal increased with increasing fluence and hence number of vacancies produced, but it is more effective when keeping the density of defects lower and one distributes the defects in different regions of the sample. The effective Bohr magneton number per nominally produced vacancy was found to be around $0.1-0.2 \mu_B$ in these experiments, hence some 50% of that previously found by us in graphite. Nonetheless, this number surely depends on the irradiation parameters employed, such as fluence, ion current, etc.

Then, annealing the sample J1 at 800 °C for 1 h was found to reduce the number of vacancies in J1 from $1.61 \cdot 10^{18}$ (after the 2nd irradiation) to $0.96 \cdot 10^{18}$, by interpolating in the observed linear correlation.

On the other hand, 2.2 MeV proton irradiation on two similar I-b nitrogen-rich (100-200 ppm) diamond samples B1 and B2 was performed, leading to the following findings:

- No room-temperature ferromagnetism is observed, though a small magnetic hysteretic loop may appear below $T_g \approx 55$ K.
- Superparamagnetic contribution is observed at 300 K superposed to the expected bulk diamagnetism, that remains essentially constant after irradiation, and could be understood by the sample preparation methods.
- Pristine I-b samples before irradiation exhibit a Curie paramagnetic contribution of 0.0123 emu·K/g.
- After irradiation, these I-b samples modestly increase that paramagnetic coefficient, though seemingly in a more efficient way per nominal vacancy than when irradiating II-a samples.

References

Modification of the optical properties of materials with ions at CMAM

José Olivares Villegas

In the last ten years (2003-2013) we have been extensively researching in the subject of the interaction of high energy heavy ions with optical materials, exploring the potential development of unique nanostructuring and photonic applications like novel optical waveguide fabrication. The main mechanism we exploit by using ion irradiation is the very high levels of electronic excitation obtained from the electronic energy loss caused by such heavy ions along their ion tracks.

The amount of excitation (that can reach very high values of several keV/nm!) can be tuned selecting the appropriate ion and with the absolute energy also the shape of the stopping curve can be adjusted so as, for example, to place the maximum value a few microns inside the material. Since the electronic excitation create damage in the materials, we can select whether this damage layer is on the surface or buried [1-4].

One of the optical materials that we have studied in more detail is the reference material for Photonics lithium niobate (LiNbO₃). It is worth mentioning that our first trial to produce optical waveguides by means of ion “implantation” using silicon ions failed. It was soon discovered that we were using so much electronic excitation density that amorphous layers were formed from the surface, as discussed in detail in ref [1].

A strong modeling effort has been made to start understanding the damage creation and accumulation, key issues of the damage process [2,6].

Soon we realized and proposed a novel method to fabricate optical waveguides based on generating buried amorphous layers, by means of placing the maximum stopping power inside the crystal. This is the case of irradiations with fluorine ions with energies in the range of 15-30 MeV. Fig. 2 summarizes the main characteristics of such waveguides. A key result is that the fluence needed in the range 10¹⁴-10¹⁵ at/cm² are several orders of magnitude lower than those required by standard light ion implantation. More details can be seen in refs [3-5].
Figure 2. (a) Refractive index profiles of the waveguides for some fluences indicated in the labels, (b) Microscope photograph of the polished end, showing darker the buried amorphous layer and also some additional damage at the depth of the nuclear end of range depth, (c) Photograph of the light scattering from the guide mode coupled and being propagated.

The irradiation in the regime of isolated tracks has shown a quite unique and attractive way of nanopatterning, since very high aspect ratio amorphous tracks are can be produced, as shown in the TEM photograph (Fig. 3a). In addition, these tracks can be etched to fabricate nanopores as shown in the AFM image (Fig. 3b).

Several Thesis have been carried out in the last ten years at the CMAM working with this experimental approaches in combination with the development of appropriate models (mainly proposed by Fernando Agulló-López):

Most of the research work relative to optical materials for photonic applications have been developed in collaboration with the group of Nonlinear Optics of Depto. Física de Materiales, leaded by Mercedes Carrascosa. We have published together several articles and we have joined projects listed at the end.

On the other hand, we started in 2007 a collaboration with the CIEMAT to study the damage under irradiation in the optical/functional material SiO₂ (both amorphous and crystalline) for the field of Fusion energy; the results obtained are collected in the recently defended Thesis mentioned above. Some illustrative results are shown in the Figs. 4, 5 and 6 and in ref [44].

In the last few years a strong effort is being carried out to perform in-situ optical measurements such as ionoluminescence, reflectance and transmittance so as to improve the efficiency and capability of the data analysis. Some results of the publication [39] are shown in Fig. 7.
Figure 7. (Left): IL spectra, and analysis into their component bands, during irradiation of quartz with Br at 25 MeV. a) Low irradiation fluence (2.5 x 10^11 cm^-2) and b) high irradiation fluence (6.5 x 10^13 cm^-2). The insets represent color photos of the samples showing the transition from red to blue on progressing irradiation. (Top right): Overall kinetics for the normalized yield of the blue (2.7 eV) band (see text) as a function of the irradiation fluence. The curves correspond, from bottom to top, to the ions O (4 MeV), Cl (5 and 10 MeV), F (5 MeV) and Br (15 and 25 MeV), Bottom right): Initial slope for the growth of the blue band as a function of stopping power S_e. The threshold stopping value (≈ 1.7 keV/nm) is determined by extrapolating the linear plot to zero yield.

Projects Related To The Photonics Research Line:


3 “Daño por excitacion electronica de materiales opticos no lineales con iones pesados de alta energia para micro- y nano-procesado”. Proyecto MAT2008-06794-C03-03/MAT del M. Ciencia e Innovacion. 2009-2011. IP: José Olivares. I. Óptica, CSIC.

4 Desarrollo del programa de actividades I+D multidisciplinares de la instalación científico-técnica singular del centro de tecnologías para la fusión. Proyecto S2009/ENE-1679; “Programas de actividades de I+D entre grupos de investigación” de la Comunidad de Madrid. 2010-2013. IP: A. Ibarra, CIEMAT.


6 “Validacion de los etalones de niobato de lito como filtros espectrales para la mision solar orbiter.” Accion Complementaria AYA 2011-15060-E, MICINN ; 2012. IP: José Olivares. I. Óptica, CSIC.
Publications Related To Photonics In The Period 2003-2013


Applications of ion beam techniques in modern semiconductor materials

A. Redondo-Cubero¹

During the last years, and thanks to several collaborations with ISOM (Madrid), IST (Lisboa), and HZDR (Dresden) among other institutions, CMAM has intensively participated in the study of semiconductor materials. Due to the great interest of wide bandgap semiconductors, such as Gallium nitride (GaN) or Zinc oxide (ZnO), a considerable attention has been paid to the analysis of these systems, which are the current basis for the development of light emitting diodes (LEDs), laser diodes (LDs), and high electron mobility transistors (HEMTs). These materials present different challenges from the point of view of the growth due to the alloying with other binaries (sometimes leading to phase separation), the formation of heterojunctions, the growth in non-polar planes, the reduced crystal quality, etc. This has motivated an extensive characterization of several heterostructures, from epitaxial thin films to as-processed devices.

Ion beam analysis (IBA) techniques have proved to be powerful methods to obtain depth-resolved and lateral information about composition, defects, impurities, phase separation, strain, etc.. Rutherford backscattering spectrometry in random (RBS) and channeling mode (RBS/C) has been systematically used for the elemental depth-profiling and defect characterization of these materials. Fig. 1 shows an example of the random and aligned spectra of an a-plane MgZnO heteroepitaxial film grown by molecular beam epitaxy [1]. This study is a paradigmatic case, where an accurate determination of the composition can be carried out thanks to the individual analysis of the three elements of the layers (Mg, Zn, O). Taking advantage of resonant conditions to O (at 3035 keV), the crystal quality and the lattice site location of the different atoms can be also analyzed along different crystallographic directions [1,2].

The valuable information provided by RBS/C, in terms of defect depth-profiling, has proved to be very helpful in the case of GaN-based compounds too. In particular, RBS/C experiments helped to clarify that spontaneous phase separation can also take place in relaxed AlInN layers [3]. The determination of strain in these ternary layers is difficult due to the lattice-matched conditions with the substrate, what leads to small shifts in the RBS/C angular scans. However, a series of high-energy experiments carried out at

Figure 1. (Left) RBS/C spectra of a-plane Mg0.52Zn0.48O heteroepitaxial layer. (Right) Lattice site location of Mg by ion channeling indicating the substitutional character on Zn-sites.
CMAM showed that, even for such critical cases, it is possible to analyze the strain state by breaking the anomalous channeling effects at the interface with the substrate [4].

The constant miniaturization of the electronic devices is also forcing the development of IBA methods beyond the current limits. However, the analysis of as-processed devices is feasible in many cases when using special set-ups with micrometer lateral resolution (which can be attained in the internal microprobe beam line at CMAM). Fig. 2 shows an example of such kind of analysis for Au/Ti/Al/Ti ohmic contacts that evidenced crater formation during the thermal annealing treatment [5]. The elemental mapping from RBS and particle induced X-ray emission (PIXE) spectra demonstrated the lateral segregation of the metals during the processing.

References:

(*) various collaborations with: R. Gago2, K. Lorenz1, A. Hierro3, M. Vinnichenko4, M.D. Ynsa5, E. Alves1, E. Muñoz3
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Ten years of IBA of Cultural Heritage at CMAM

Alessandro Zucchiatti

The CMAM strategy in CH applications of IBA

Since the foundation of CMAM the promotion and development of IBA in the field of Cultural Heritage (CH) has been one of its main activities. This is based on a specialized beamline (Fig. 1), on the participation to cooperation schemes and on several multi-disciplinary national and international collaborations and has experienced a continuous evolution in the past ten years.

The focused micro-beam produced by a Oxford quadrupole doublet, with high demagnification and extracted in air, has been the second beam line installed in the CMAM, just after the so called Standard beam line, supplied at origin with the 5 MV accelerator. That first set-up has been upgraded quite a few times. The first Si(Li) detectors have been replaced with models offering better resolution and peak to valley ratios as well as a more compact design. The exit kapton foil has been replaced by a Si₃N₄ (silicon nitride) window that now acts also as a relative charge monitor thanks to a dedicated SDD counter that measures de Si X-rays. An electromechanical mass flow controller has been associated to the He flow in front of the low energy X-ray and the RBS detectors and a control software for the additional instrumentation (motors, flow-meter, pointing laser, etc.) has been implemented. The experimental station is capable of providing ion beams of nearly Gaussian spatial distribution with typically a FWHM of 40 micrometers and currents from a few hundred pA to a few nA. Our system can manipulate and analyze large size (up to 30 kg) objects with a positioning precision of some tens of microns and includes complementary techniques usable in parallel: PIXE, PIGE, RBS and, recently, Iono-Luminescence.

Figure 1. Samples of many different kinds have been successfully analyzed at the external microbeam line of CMAM
The instrumental characterization of a CH object (in other words archaeometry) is fully relevant only if it is part of a multidisciplinary project. It could involve scientists of several disciplines (typically physics, chemistry and geology) as well as curators, conservators, restorers, art historians. When a project aims at linking the object with the history, the human feelings, the artistic moods, the economy and the technology of its epoch, the impact of its outcome is definitely much enhanced compared to a simple analytic approach. This productive and maybe necessary approach in CH, drives the research activity of non-dedicated laboratories (as is the CMAM with its broad range materials research), mainly towards the development of instruments and methods and the coordination and promotional actions. The analysis, on the other hand, is heavily conditioned by the occasional availability of objects and the constraints imposed on their transport, manipulation and storage. Therefore the mission of CMAM, as a potential scientific partner in the general study of the CH, was and is twofold. The first aim is to develop a cluster of IBA and associated techniques and protocols that can demonstrate effective in answering the many questions that a CH object can raise. The second aim is to offer its potential partners (museum, archaeologists, etc.) the most extensive guarantees about the protection of the object, the validity of results and the assistance of the team in all the analytical steps and beyond. To reach its goals the CMAM has based its CH activity on:

1. A constant attention to the development of instruments and methods for IBA application to CH.
2. The promotional and coordination activity. To mention are the participation to the COST European Cooperation actions, COST G1 and G8, focused on the application of non-destructive analytical techniques to Heritage and Conservation Science and the inclusion in the national network of CH professionals of two CMAM researchers.
3. The establishment of cooperation links with national and international institutions, cemented by common work, seminars, visits and agreements.

On these bases it has been possible to build a significant and recognized activity that has allowed unique advancements in the field and has brought to the CMAM outstanding objects from the most important museums like the Luca Cambiaso ink drawings, the Torredonjimeno treasure, the golden objects Costa Rica collection and the Quimbaya (Colombia) treasure. The activity is reflected in 27 publications produced on archaeometry topics from 2002 to 2013, participation to conferences, seminars, publication of monographies, and the obtention of a Ph.D. by Carolina Gutierrez Neira in November 2009.

**Outstanding applications: The gold treasure of the Museo de America, Madrid**

An unprecedented combination of techniques, of which PIXE (and RBS) has been applied in two phases, to a large set of object from the Museo de América (Madrid), the institution that keeps an exceptional collection of pre-Hispanic gold, including the Quimbaya’s Treasure, Colombia (QT), with a total of 135 gold items, and the Costa Rica Collection (CRC), with 49 gold items (Fig. 2). This has been a research that has had quite some impact in the media, including the recording of a divulgation documentary emitted by the national chain TVE2. In the first instance both collections were characterized on site at the museum with portable equipment, performing an examination of the surfaces by optical microscopy (OM), an XRF elementa analysis and, in the case of sixteen of the biggest, hollow items from the QT, also radiography. Subsequently, the CRC was moved to the MicroLab, CCHS-CSIC (Centro de Ciencias Humanas y Sociales), Madrid, for electron microscopy examination and elemental
microanalysis (MEB-EDS). With the above information at hand a set of 65 Quimbaya items and 38 CRC objects was selected for analysis by PIXE and RBS at our Centre.

While handling the QT vessels, known to be used to mix coca leaves and as cinerary urns, some of the ashes dropped out and were collected for AMS dating. This extraordinary surprise allowed us to approximately date this treasure to between 410-590 AD. On the contrary, the CRC has no archaeological context whatsoever. A beam of 3 MeV protons (in a few cases 5 MeV) was used with currents around 3 nA and measurement times of 600 s in the CMAM external microbeam line. A total of 420 points were measured on the 103 gold (and 2 reference objects). The PIXE spectra gave so far the most relevant information. The analytical procedure has been checked with the help of two gold reference objects (Fig. 3).

Figure 2. A pendant from the Costa Rica Collection analyzed at CMAM

Figure 3: The elemental concentrations of a 17 carats gold ring are constantly reproduced

Figure 4. The PIXE and XRF gold concentrations agree with them in a large set of objects
The PIXE objects composition is in agreement with that given by the on-site XRF analysis of the entire collection (Fig. 4). Even if the IBA could only be applied to a subset of objects, the richness of PIXE data allows a step forward in the understanding of the construction technology. PIXE is reproducible (Fig. 3), can be easily applied to different points of the same object to verify the homogeneity of the alloy, the spectra can be analyzed with different complementary choices of the elemental X-ray lines to probe at least qualitatively different depths of the object. With the exception of Fe and Zn that appear as minor or trace elements in some cases, but without any significant pattern, the whole collection if composed of ternary Cu-Ag-Au alloys.

In the majority of Costa Rica objects (Fig. 5) the alloy is homogeneous within each object, quite homogeneous within the set of objects received at CMAM and generally very rich in gold. Levels above 80% in weight are seen, associated to relatively low levels of Ag. The Ag content has an average of 5% in weight with a standard deviation of 2%, quite small compared with that of Cu (standard deviation of 15%). This would indicate that copper was added, in different quantities, to a native gold-silver material to produce the jewellery alloy which was worked afterwards by the lost wax process. This datum is compatible with the composition of native gold in the region. Costa Rica, and its extension towards the Panama strait, is a region of great mineral wealth, with gold, silver and copper deposits. The gold-bearing rivers of the Osa and Burica peninsulas and the deposits near the border with Nicaragua are still worked by artisan miners (coligalleros). Although the primary composition of the gold varies both in terms of the depth of the primary deposits and along the course of alluvial deposits, the average silver content can be estimated as around 5% in weight.

The Quimbaya treasure is definitely heterogeneous (Fig. 5). PIXE allows identifying objects in which the alloy is quite homogeneous although different in composition from object to object. However several objects show patches and repairs by casting on, which are reflected in the composition of the corresponding areas. The ternary Au, Ag, Cu alloys have the characteristic features of the so called Tumbaga. Three groups of objects can be seen (Fig. 5): one is characterised by low Ag (<6% in weight); the second by high Ag (>27% in weight) and the third and largest by a broadly distributed composition with an intermediate roughly constant Ag content. The key for the interpretation of this data is, rather than the heterogeneity of the treasure, the masterly use of the Cu/Au ratio to control the object colour so to associate it with the use and function of the object. Utilitarian objects fall in the group of copper rich “reddish” objects. Ritual and personal “status symbol” objects, like pendants and collars, fall in the group of gold rich “shining” objects. The mastery of colour through the alloy composition reaches its top in the QT with polychromy obtained on the same object, like in the case of some pins.

Figure 5. The Au-Ag-Cu ternary diagram defines the different alloy compositions. Blue full dots, Red full squares, green full triangles and black open squares identify outlier objects in the Quimbaya set.
Use wear and repeated abrasive cleaning have strongly affected the surfaces, more deeply in the obverse, to the point of almost eliminating the gilded layer in quite a few cases. That is why only a few of the RBS spectra, regularly taken together with the PIXE ones, could be clearly interpreted as generated by a gold-rich layer on the surface sitting on top of a copper-gold alloy. Nevertheless the analysis of the PIXE spectra, and in particular the \(\text{CuK}_\alpha/\text{CuK}_\beta\) ratio gave some qualitative evidence of depletion gilding as a standard finishing process in the Costa Rica production, resulting in a gold rich surface alloy, while most of the Quimbaya objects do not show clear evidence of depletion gilding. The calculated \(\text{CuK}_\alpha/\text{CuK}_\beta\) ratio generated by 3 MeV protons, from a Au-Cu alloy in various proportions from 100% Au to 100% Cu with a pure gold layer on top of thickness up to 5 microns is compared (Fig. 6) to the experimental histogram (y-axis) corresponding to either the QT or the CRC. We see how the CRC distribution is compatible with a Au-rich (within 70 and 100 %) alloy topped by a 1-2 microns of pure gold while only part of the QT is compatible with such description. The Majority of QT is on the contrary compatible to a homogeneous Au-Cu alloy (40% Cu 60% Au in weight) not enriched on the surface.

![Figure 6. Comparison of the Cu Kα/Cu Kβ experimental and calculated ratio (see text for explanation)](image)

**Outstanding applications: The ink drawings of Luca Cambiaso**

The Genoese painter Luca Cambiaso (1527-1585) was one of the most famous masters of his time. His notoriety extended well beyond the regional boundary. In 1583, at his apex of success in Genoa, he was requested by the king of Spain Phillip II to the Escorial, near Madrid, to execute some of the frescoes and altarpieces of that royal monastery and there he died two years after. Chief of a huge and well organized workshop, he was an innovative and copious draftsman. His corpus of ink drawings is by no doubt the largest among those of the Renaissance, counting thousands of pieces distributed over several collections. The technical and stylistic studies may be at times quite difficult in such a rich context. The need of the support of an objective, analytical classification of the Cambiaso immense production led to two pilot, combined studies focused on the collections of the Musei di Strada Nuova in Genoa (analysed at INFN Florence) and of Prado in Madrid (analysed at CMAM). From the two museums were made available 28 drawings (Fig. 7) in pen (or brush) and ink on unprepared papers, with application of dilute ink, in form of broad washes. PIXE was used at both labs for the characterization. A relative beam monitor that counted the X-rays from a dedicated mono-elemental target (a rotating Ni platelet in Florence and a Au coating over the kapton exit window in Madrid) allowed to produce a calibration curve for the detectable inorganic elements in the ink strokes and on the paper.
Both sets are iron-gall inks. The sum of elemental thicknesses from Na to Pb measured on ink strokes averages (Figs. 8a and 8b) at about 150 \( \mu \text{g/cm}^2 \) (st.dev. about 80 \( \mu \text{g/cm}^2 \)) with peaks up to 450 \( \mu \text{g/cm}^2 \). The sum of the elemental content measured on paper is in the order 30-50 \( \mu \text{g/cm}^2 \) (Figs. 8c and 8d). The drawings from Genoa have higher average value on paper probably because they have all been restored with a consequent dispersion of ink elements on the blank paper.

Iron dominates the ink composition in black or brown strokes (Figs. 9a and 9b); its amounts are quite diluted in the washes (Figs. 9c and 9d) and there is little in the paper (Figs. 9e and 9f). Sulphur is distinctively present in the inks (Figs. 9a and 9b) although its concentrations are very dispersed. The ratio S/Fe in the ink averages at 0.17 ± 0.23 for the Genoa inks and 0.23 ± 0.25 for the Prado inks. The average ratio is lower than the stoichiometric weight ratio (0.57) expected from the basic ingredient of iron-gall inks i.e. \( \text{FeSO}_4 \) (vitriol). However on paper the ratio S/Fe is close to 1 as shown by the similar distribution of Figs. 4e and 4f. In the washes the ink is diluted and the sulphur content is partly provided by the ink and partly by the paper. Calcium is abundant everywhere. The average values on the ink are larger than those on the paper and washed areas; however its distribution is quite dispersed and makes it difficult to subtract the contribution of the paper in a correct way.
Minor and trace elements allow specific considerations about individual drawings. Light elements are often below MDL: Na (55% of cases), P (more than 20%), Cl (more than 20%). Titanium, chromium and mercury are occasionally present. Chromium is only present in a note of the collector Santo Varni (1807-1885) added to The Holy Family (SFA), mercury is present in the washes of The Circumcision of Abraham’s Tribe (CTB) but is not present in the ink strokes (Fig. 10).

Lead requires some attention because it has a very peculiar behaviour. It is present in the paper and therefore uncertainties in the subtraction may result in variations of results from point to point in the same drawing. However in a few cases, A winged monster (ARA) or Arpia (ARC) the excess of lead in some points corresponds evidently to a lead rich ink. A possible explanation could be a retouch or perhaps the effect of some sedimentation in the ink pot which at the time was most probably made of lead (Fig. 11). The lead variability is in summary large in all the drawings: in The Sacrifice of Isac (ISC) and Niobe’s impiety (NIO) lead can be as high as 50% of the iron content.
Other specific problems could be addressed. The Circumcision of Abraham’s Tribe and the Galatea riding dolphins with Amorini (CTA,CTB and GTA,GTB respectively) came in two versions. The versions CTB and GTB are clearly different from the drawings CTA and GTA, believed to be autograph. CTB, as already mentioned, is quite peculiar in the washes made with a mercury containing ink while the ink strokes in CTA are much richer in Mn and Cu as well as in Ca and Pb than are those of CTB. GTB differs from GTA mainly because of the higher copper content. The drawing representing Minerva (MIN) bears at the verso the handwriting “Cambiasi+”. The analysis reveals that it was not written with the same ink as the drawing. It is characterised by more copper and zinc than the drawing; therefore this is a confirmation that the inscription is certainly a collector note rather than a signature.

This was the first, exploratory, PIXE (and IRR analysis) of an important set of 28 Luca Cambiaso ink drawings. The combination of non destructive techniques and the selection of drawings have proved the remarkable potential that this combination has in distinguishing school copies and imitations from the original production. The possibility that was offered to receive artefacts both from the collections of Marcello Durazzo and Santo Varni, hosted in Musei di Strada Nuova of Genoa, and from the collection of the Prado Museum has enhanced the outcome of such a study in particular in what concerns the followers work and the techniques of drawings. Our study has put in evidence that, on the long range, the construction of a specific database of ink composition is desirable to fully exploit the analytical benefits in the attribution and indirect dating of the production of Luca Cambiaso, the most prolific drawer in the history of art.

**Outstanding applications: Luster ceramics**

Lusters are a composite material consisting of thin layers of coinage metal nanoparticles in glass. They display peculiar optical properties and were obtained, from the 9th century AD by a process that involves, as we know it today, ionic exchange, diffusion, and crystallization. The origin of the high reflectance (golden-shine) shown by those layers has been debated at length. It has been attributed to either the presence of larger particles, thinner multiple layers, or higher volume fraction of nanoparticles. The CMAM has been involved for quite some time in a cooperation work aimed at understanding the nature and origin of luster by applying Rutherford backscattering spectroscopy, transmission electron microscopy, x-ray diffraction, and ultraviolet-visible spectroscopy to both original artifacts and laboratory produced samples. From the data the correlation between synthesis conditions, nanostructure, and optical properties can be obtained for this class of materials. More information can be found in a dedicated article of this activity report.
List of CMAM publications focused on CH (2002-2013)


**Acknowledgements**

The list of publications bears in it as well a list of nice colleagues, from other institutions, with whom it has been a pleasure collaborating and from whom we have learned quite a lot about the fantastic adventure that is studying and preserving our cultural heritage. To all of them go our warmest thanks. We would also like to specially thank Olga Enguita and Carolina Gutiérrez Neira, former colleagues, who have left the CMAM but whose contribution to the CH research field is still well visible in the Centre.
Surface Science Studies at CMAM: Perspectives

José Emilio Prieto

The surface science research line at CMAM is devoted to 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. Nanoscale materials are characterized by at least one of the spacial dimensions approaching the nanometer scale. With decreasing dimensions, the properties of surfaces and interfaces become increasingly dominant and require a detailed characterization in order to understand the properties of the system and to be able to modify them in a controlled way. They find application in many different fields, such as optoelectronics, biomaterials and magnetic materials. As examples of the latter, one can mention: the discovery of the giant magnetoresistance effect, associated to the oscillatory magnetic coupling of very thin magnetic layers separated by nonmagnetic spacer layers [1,2]; the strong dependence of magnetic ordering on structural parameters, as in the case of Fe chains on the Ir(100) surface [3]; the richness of the magnetic behavior of the ultrathin epitaxial films of Fe on Cu(100) in connection with the different structural transformations [4].

The experimental setup of the UHV-surfaces beamline at CMAM [5] contains a set of facilities for the growth of thin epitaxial films and sample characterization using several experimental techniques. Films can be grown by Molecular Beam Epitaxy (MBE) and 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 (ERDA), etc.], as well as the characterization of the samples with surface-sensitive techniques [low-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/channelling 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 about 15 m and focused to a spot smaller than 1 mm on the sample by means of the 0-degree and the UHV beamlines. A quite unique experimental facility will be the new LEIS-ToF system for surface structure determinations. Here the sample is bombarded by a chopped beam of noble gas ions (He, Ar, Ne...) with energies typically in the range 2-6 keV. Time-of-Flight (ToF) spectra of scattered and recoiled particles are recorded. The characteristics of the ion-solid interaction at low energies characteristic of the LEIS regime, such as the strength of the interaction, the screening of the nuclear charge by the electrons, and the large width of the shadow cones, convey this technique a high surface sensitivity, in particular to the surface structure through the effects of shadowing and blocking. Azimuthal or polar scans of the intensity of scattered or recoiled particles can be obtained from the spectra by rotating the sample. Efficient detection over an extended range of scattering or recoiling angles allows for surface structure determination using ion fluences of less than 10^{12} ions/cm^2. On the other hand, ion beams can also be used to modify the properties of materials in a controlled way. 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, comparisons of 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 [6], 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 LEED but with increased sensitivity for light elements. On the other hand, the sensitivity of LEED to the first few monolayers is very convenient for the analysis of ultrathin films. A transfer systems under UHV conditions is planned, which will allow samples to be transferred to other experimental facilities.

References

The activity of the Experimental Nuclear Physics group of the IEM–CSIC related to the Nuclear Physics Line is primarily focused on characterizing excited states near particle threshold for their relevance in Nuclear Astrophysics. At CMAM we can populate these states in low energy reactions. These studies are complementary to our main activity where we study the structure of the atomic nucleus. This research is performed at the major European Facilities; in beta decay at ISOLDE-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.

At CMAM we have, by using the reaction $^{10}$B($^{3}$He, pααα), explored excited states in $^{12}$C in order to obtain a better understanding of the triple alpha process as well as to study α-clustering in light nuclei. Improved Limit on Direct alpha Decay of the Hoyle State [1]. We have also studied the properties of $^{12}$C resonances determined from the $^{10}$B($^{3}$He,p) and $^{11}$B($^{3}$He,d) reactions studied in complete kinematics [2], the breakup of $^{12}$C resonances into three alpha particles [3], the observation of gamma-delayed three-alpha breakup of the 15.11 and 12.71 MeV states in $^{12}$C [4].

Further, we have performed measurements in order to determine the cross section of the $^{3}$He+$^{4}$He→$^{7}$Be reaction as a function of energy. The technique used in the experiment is the activation method, which consists on the detection of the delayed 478 keV γ-ray from the first excited state in $^{7}$Li after the $^{7}$Be electron capture decay. New measurement of the $^{3}$He-(α,γ) $^{7}$Be cross section at medium energies [5].

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; CEPA (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 (<30MeV) and high energy protons (<300 MeV) at the future European Nuclear physics facility FAIR (Facility for Antiproton and Ion Research), Darmstadt, Germany. LaBr$_3$(Ce): LaCl$_3$(Ce) Phoswich with Pulse Shape Analysis for High Energy Gamma-ray and Proton Identification [6].

In the continuation we will enter into more detail of the recently finished project of S34 and a test experiment paving for new projects in 2014.

**Determination Of The Cross Section For The Reaction $^{4}$He($^{3}$He,γ)$^{7}$Be**

*Project FPA2009-07387, (doctorando JAE-predoc. M. Carmona-Gallardo)*

One of our goals is to measure the cross section of the $^{4}$He($^{3}$He,γ)$^{7}$Be nuclear reaction. The astrophysical relevance of this reaction is two-fold. On one hand it plays a determining role in the prediction of the solar neutrino flux. Among all nuclear inputs parameters of the Standard Solar Model, the cross section of this reaction is the major source of uncertainty. On the other hand this reaction is important in the Big-Bang nucleosynthesis. Specifically, it is determining to resolve the abundance of the primordial $^{7}$Li in the universe.

We have studied the cross section of this reaction at medium energies using two different experimental techniques. The first experiment was performed at the Centro de Microanálisis
de Materiales (CMAM) in Madrid with the Activation Technique, where the $^7$Be recoils were deposited in copper catchers. The reaction cross section is estimated subsequently by detecting the delayed $\gamma$ from the ions in the catcher [5]. The results resolve the discrepancies between the previous measurements and corroborate the first ab-initio calculations by T. Neff [7].

In the second experiment we, in collaboration with Univ. of York and TRIUMF, used the $^7$Be Direct Detection Technique. In this case, the cross section is obtained from the direct counting of the $^7$Be recoils. The experiment was carried out using the DRAGON spectrometer at TRIUMF. DRAGON is a mass separator with magnetic and electric dipoles that allows separating the recoils of interest ($^7$Be) from the unreacted beam ($^4$He) before being detected in a Double Sided Si-strip Detector (DSSD) placed at the focal plane of the separator. These studies of the cross section for the nuclear reaction of $^4$He($^3$He,$\gamma$)$^7$Be is the thesis topic of Mariano Carmona Gallardo, to be defended during the spring of 2014.

**Characterization Of Nuclear States And Relevant Reactions In Stellar Nucleo Synthesis Using $^{19}$F ($p,\alpha\gamma$)$^{16}$O To Populate A-Unbound States In $^{16}$O**

*Project EUROGENESIS EUROCORES-EUI-2009-04162 and to be continued during 2014 within the project MINECO: FPA2012-32443*

We have performed a feasibility study for using the nuclear reaction $^{19}$F($p,\alpha\gamma$)$^{16}$O to determine the reduced $\alpha$-widths of the states, relevant for the direct radiative capture reaction $^{12}$C($\alpha, \gamma$)$^{16}$O. A mono energetic proton beam in the energy- range between 800 and 3000 keV impinged upon a 120 g/cm$^2$ MgF$_2$ target (with a 10 $\mu$g/cm$^2$ $^{12}$C backing). An array of highly segmented $\Delta$E-E Silicon detectors was used to measure the energy and angle of the outgoing particles. This experimental setup covered forward angles in the range of 37 to 64 degrees, and from 115 to 156 degrees in the backward direction, which allowed for the detection of particles emitted back-to-back in coincidence. Therefore, the focus of the analysis is to identify two $\alpha$ particles plus (ideally) a $^{12}$C recoil in coincidence. Of special interest are the alphas that populate the 2+ and 1- sub-threshold states in $^{16}$O located at 6917 keV and 7116 keV respectively, which are considered to play a key role in the radiative capture process. Also, it is important to identify and eliminate random coincidences.

The $^{12}$C($\alpha, \gamma$)$^{16}$O reaction is of great importance for nuclear astrophysics due to its major role in determining the Carbon to Oxygen ratio during Helium burning in stars. Also, it influences the nucleosynthesis of all elements beyond $A = 16$ in later burning stages. A high number of studies have been dedicated to the investigation of the $^{12}$C($\alpha, \gamma$)$^{16}$O reaction, but its very low cross section (around 10-17 barn) at 300 keV, the relevant energy for He burning, has limited the knowledge of its rate. The use of the inverse reaction $^{19}$F($p,\alpha\gamma$)$^{16}$O for the indirect study of $^{12}$C($\alpha, \gamma$)$^{16}$O, will allow for the determination of the reduced $\alpha$-widths of the sub-threshold states in. The analysis of the data is in progress. The results will be used to optimize the experimental set-up for an improved experiment during 2014.

**References:**

Modification and analysis of structural and functional materials for future energy production sources

F. Agulló, D. Bachiller Perea, A. Climent Font, B. Gómez Ferrer, David Jiménez-Rey and A. Muñoz Martín

The use of particle accelerator technologies is revolutionizing much of the scientific research. In particular, ion beam facilities applied to the research and development of new technological materials are continuously increasing. Ion beams are used for some advanced characterization techniques. These techniques are currently known as Ion Beam Analysis (IBA). They are routinely used in nuclear physics, microelectronics, space & aeronautic or energy research. The irradiation by high energy ion beams also permits the formation of new materials with properties that would be impossible to obtain by means of the usual synthesizing and manufacturing processes. These Ion Beam Modification of Materials techniques, IBMM techniques, are continuously developed. They open the door to new technological applications. Furthermore, studies of accelerated damage under irradiation that emulates hazard environment are necessary to understand the response of materials exposed to severe environmental conditions and the applicability of functional and structural materials. The above mentioned capabilities of high energy ion beams for analysis and modification of materials are used in the research on materials science.

Since 2004, some of the CMAM research activities are focused in materials for future energy production sources, an unavoidable task nowadays. These activities began with early studies on fused silica (adsorption on insulator materials enhanced by D implantation). They have evolved from studies on materials for fusion and fission reactors to the most recent studies on photovoltaic materials.

Functional and structural materials for nuclear reactors are exposed to a hostile environment (high fluxes of high energy neutrons, charged particles and photons). This irradiation produces defects in the structure of the materials -via atomic displacement phenomena and ionizing processes-. In addition, the nuclear reactions induced by the neutrons generate the accumulation of He and H impurities. Both defects and impurities usually modify the physical properties of the materials, and therefore, affect their reliability.

The use of ion beam accelerators is considered an efficient and useful tool to experimentally study the effects of particle irradiation in metallic alloys and functional materials, even when the involved processes are different to those of neutron irradiation. The CMAM has been involved in several studies in this respect; it is worth mentioning:

- Radiation damage and hydrogen, helium and tritium migration in ceramic breeder blanket materials used in fusion reactors (Li₃TiₓO₉, Li₃SiₓO₉).
- Defect generation, impurities diffusion, mechanisms of atomic transport and evolution of the mechanical properties of silica under irradiation and/or thermal gradient processes.
- Microstructural stability, degradation mechanisms and diffusion and aggregation processes induced by radiation on Fe, Fe-Cr alloys, EUROFER and EU-ODS EUROFER steels.
- Properties of scintillators to be used for wide range ion detectors in fusion devices.
Recently, some of the CMAM research activities have evolved towards the investigation on materials of technological interest for solar energy. New materials that boost the efficiency of current solar cells are needed before the large scale use of solar energy. Real-time studies using energetic ion beams begin to be used for the characterization of photovoltaic materials at CMAM, besides the traditional techniques as RBS or ERDA. The CMAM is actually involved in the following studies on materials for third generation solar cells:

- Growth and characterization of luminescent materials as spectral converters for enhancing solar cells efficiency (ZnO and TiO$_2$ doped with rare earths)
- Growth and characterization of nanostructured antireflection materials (silicon nitride based materials).

Some of the research activities carried out at CMAM in materials for energy production has been carried out in collaboration with several national and international research groups:

Fusion National Lab (LNF-CIEMAT): R. Vila and B. Gómez-Ferrer (studies on low-temperature resistivity measurements on irradiated Fe-Cr samples), M. González and E. Carella (studies on lithium ceramic for breeder blanked), P. Fernández and M. Roldan (studies on Eurofer and ODS-Eu steels properties), P. Martín and R. Saavedra (studies on SiO$_2$ defects creations under irradiations), I. García and F. Sánchez (studies on irradiation of materials under B), B. Zurro (plasma fusion diagnostics TJ-II stellarator).

Joint Accelerators for Nano-science and Nuclear Simulation (JANNUS): L. Beck and S. Miro (diffusion in combination with IL with triple beam, u-Raman)

Department of Physics of the Indian Institute of Technology, Delhi: S. Ghosh and R. Bonmali (amorphous hydrogenated silicon nitride)

Department of Applied Physics, Universidad Autónoma de Madrid: R. Pérez Casero (ZnO$_x$ doped with rare earths)

Department of Materials Physics, Universidad Autónoma de Madrid: C. Sánchez and X. Ares (H storage, thermoelectric and photovoltaic energy conversion)
Microscale engineering of silicon surfaces for cell adhesion and migration control

Aurelio Climent Font

The definition of surface patterns at the microscale with contrast on determined physical and/or chemical properties is a powerful tool for analyzing cellular communication factors involved in the process of adhesion, migration or expansion which can have notable influence on therapeutic applications such as tissue engineering. Human mesenchymal stem cells (HMSC) are increasingly used in therapeutic applications for bone, cartilage or fat transplantation and repair and their adhesion to a substrate and migration activity can be affected by external factors as diverse as the presence of oxygen, substrate stiffness and geometry, and surface chemistry.

A research group at University Autonoma of Madrid to study the behavior of cells in micro-patterned surfaces was formed in 2008 as a collaboration from researchers of CMAM, the Department of Applied Physics and the Department of Molecular Biology. Our approach to create one and two dimensional (1D; stripes and 2D; squares) microscale patterns with some kind of physical and/or chemical contrast was based on the increase in resistivity that single crystal silicon substrates may experience when exposed to ion beams [1]. The surface micropatterns were fabricated in three steps. First, p-type Si samples cut out of <100> single crystal wafers with resistivity of 0,01 μcm and were back-side coated with aluminum to form an ohmic contact. In a second step the polished front of the samples were covered with Cu micro-masks (Gilder, Lincolnshire, UK), and irradiated with MeV Si ion beams generated with the 5 MV Cockcroft-Walton CMAM accelerator. In a third step, the Cu mask was removed and the sample was galvanostatically etched in HF:ethanol electrolytes under illumination following a standard nano porous silicon (nanoPS) fabrication process. The patterns with well defined areas of different conductivity, the area of lower conductivity being the area exposed to the ion beam, were converted by the etching process into patterns with different surface characteristics as the nanoPS grows selectively in the non-irradiated areas while non-etched Si remains in irradiated regions. A pictorial description of the overall process is shown in Fig. 1. Typical beam energies used in the irradiation process where 20 MeV, and irradiation fluences as low as 5·10^{12} ions /cm² where enough the conveniently change the resistivity of the Si substrate to inhibit the nanoPS formation.

Figure 1. Procedure followed to define a 1D or 2D microscale patterns on the silicon surface (see text for details).
Fig. 2a shows a perspective scanning-electron microscopy image from a cross-section performed in micropatterns showing the etched region where nanoPS has been formed (region protected by the grid of the micro-mask during irradiation) and the squares corresponding to irradiated regions where the formation of porous silicon has been inhibited. Fig. 2b shows the red photoemission characteristic of nanoPS, and Fig. 2c and Fig. 2d show the x-ray photoelectron spectroscopy signal for Si on the irradiated region (almost oxygen free) and the region with nanoPS. Notice the inset in these last two figures indicating the different wettability of the two regions; hydrophobic for the region without nanoPS, and hydrophilic for the region with nanoPS. These two different regions also show different surface roughness, being higher for nanoPS (1.1 nm rms roughness measured over 2 x 2 μm² versus 0.2 for irradiated Si).

Human bone marrow samples from healthy donors provided by the Hospital Universitario de la Princesa (Madrid, Spain). HMSC were prepared and cultivated on 1D and 2D nanoPS patterned surfaces in an attempt to study the cell adhesion and migration [2]. The results obtained from 1D nanoPS patterned in three different sizes consisting of 100 μm-wide Si stripes and 25 μm-wide nanoPS stripes, 50 μm-wide Si stripes and 25 μm-wide nanoPS stripes, and 35 μm-wide Si stripes and 25 μm-wide nanoPS stripes, suggested that the surface of nanoPS behaves as an antifouling platform probably due to its surface roughness and particular chemistry.

Experiments on 2D patterns consisting of Si squares 100 x 100 μm and 15 μm-wide nanoPS as the one shown in Fig. 2. The experimental results show that the HMSC respond to a particular 2D structure as shown in Fig. 3. It can be observed that the nuclei of the cells tend to migrate
to the intersections of nanoPS while the position of the cytoskeleton does not seem to follow an evident trend. The bar diagram adjacent to the image of the cell culture shows an statistical study of the position of the nuclei distinguishing intersections, stripes and squares either in absolutes values or normalized to the available surface for each position.

This experimental behavior has been reproduced by a simple computer simulation [2] taking into account that the cells do not tend overlap and that they will migrate tending to occupy in preference the region free of nonPS as was deduced from the 1D micropatterned substrate experiments.

Acknowledgements

This work was financially supported by the Spanish ministry MICINN under research prproject MAT2008-06858-C02-01/NAN. We also acknowledge grants from Fundación Domingo Martínez, and Comunidad de Madrid (Spain) under project Microseres. The help and disposition of the technical team of CMAM during the accelerator irradiations is also gratefully acknowledged.

References

The Potential of CMAM for Biological Research Studies

Mª Dolores Ynsa

Although in this moment biological applications at CMAM are not as numerous and familiar as other applications in other fields, such as material science, photonics and arqueometry, there are a wide range of possibilities to apply it.

Ion beam techniques can be used both to modify the materials and to obtain structural and elemental information about the studied samples with ion beam analysis. In both cases there are many biological applications.

The CMAM activity in the biological field is firstly based on lot of equipment to prepare the biological samples, which consist of: cryotome, freeze dryer, digestion pump and microwave oven, optical microscope, and diamond saw/polishing machine for mineralized tissue. The most important tool is the internal microbeam line, placed at +30°, where it is possible to work with a micrometric or sub-micrometric beam. In this line the beam can scan the target in a controlled way to obtain distribution maps. CMAM apparatus, designed and built in Melbourne, uses two sets of diaphragms (object and collimator) and five magnetic quadrupole lenses to focus the ion beam. At the same time it is provided with a magnetic deflector to perform the beam scan. The samples are positioned inside a vacuum chamber where they can be moved along the x, y, z axes. The reaction chamber is provided with an optical microscope to control, at any time, the target area that is irradiated or analyzed, as well as several particle detectors and an X-ray detector.

The beam line is particularly suited to perform irradiations, under controlled dois, of very small areas and produce micrometric or submicrometric structures. Furthermore it is possible to apply routinely the PIXE and RBS techniques to obtain elemental distribution maps and the STIM technique on thin samples to study the density variations. Although at the present moment the facility is not totally operative, we can already work with a proton beam with a beam size of about 1 µm x 1 µm and a current higher than 1 nA (Fig. 1) and with different heavy ions such as boron, nitrogen and silicon with a beam size around 5 µm x 5 µm (Fig 2).

On the other hand, the great capacity of ion beam to modify materials also has two important application fields in biomedicine: irradiation cells and the manufacture of different devices with biological applications. For both applications, beam size and its control are decisive. At the present moment CMAM is not ready to do irradiation cell experiments and no project is in mind, however the external microbeam line could be modified to do them. In contrast, the manufacture of devices using an ion beam is one of the priorities of CMAM. Up to now, the first tests have been carried out using an irradiation program developed by CMAM and Apply Physics Department of Universidad Autónoma de Madrid. Most of these microdevices will be manufactured for biological purposes.
Figure 1. Copper map of the Cu grid (2000 mesh, wire diameter 5 µm). Beam current higher than 1 nA, protons energy 2 MeV.

Figure 2. Copper maps of the Cu grid (1000 mesh, wire diameter 6 µm)
# SUMMARY OF SCIENTIFIC ACTIVITIES

## Research projects

- Projects funded by MICINN, CDTI: 8
- Projects funded by CAM: 2
- Projects funded by other sources: 4

## Scientific publications

- Papers in SCI journals: 50
- Papers in no-SCI journals: 9

## Theses

- PhD Theses: 1
- MSc Theses: 1

## Scientific contributions to conferences

- Invited talks: 5
- Oral contributions: 19
- Posters: 9

## Seminars

- Seminars at CMAM: 21
- Seminars of CMAM members: 5

## Visits

- Visits to the CMAM: 21
- Visits of CMAM members: 2

## Collaborations

- International collaborations: 20
- National collaborations: 22

## Courses and training

- Courses and training: 8
1. **Surface functionalisation of material for high added value applications.**
   - Financed by: Ministerio de Educación y Ciencia (Programa CONSOLIDER).
   - Duration: January 2009 - December 2013.
   - Main researcher: J.M. Albella (ICMM-CSIC)

2. **Nanociencia Molecular CDS2007-00010 (Program Consolider-Ingenio 2010).**
   - Financed by: Ministerio de Educación y Ciencia.
   - Duration: October 2007 - September 2012.
   - Main researcher: Eugenio Coronado Miralles (U. Valencia)

3. **Fundamentos y aplicaciones de moléculas, nanopartículas y nanoestructuras S2009/MAT-1726 (NANOBIO MAGNET).**
   - Financed by: Comunidad Autónoma de Madrid.
   - Duration: January 2010 - December 2013.
   - Main researcher: Rodolfo Miranda Soriano (UAM-IMDEA Nanociencia)

4. **Marcadores crono-tecnológicos para una contextualización de la metalurgia prehispánica HAR2011-12809-E.**
   - Financed by: Ministerio de Ciencia e Innovación.
   - Duration: October 2011 - October 2012.
   - Main researcher: Alicia Perea Caveda (CHHS-CSIC)

5. **Síntesis de Materiales para Dispositivos Fotovoltaicos de Nueva generación.**
   - Financed by: Universidad Autónoma de Madrid y Banco de Santander Proyectos de Cooperación Interuniversitaria UAM-Santander con América Latina.
   - Duration: July 2013 - December 2014.
   - Main researcher: David Martín y Marero (UAM-CMAM)

6. **Síntesis y Caracterización de Nanomateriales para Aplicaciones en tecnología de Sensores y Biomedicina.**
   - Financed by: Universidad Autónoma de Madrid y Banco de Santander Proyectos de Cooperación Interuniversitaria UAM-Santander con América Latina.
   - Duration: June 2011 - May 2012.
   - Main researcher: Juan José Saenz Gutiérrez (UAM)

7. **Propiedades estructurales y electrónicas de estados emergentes de la materia: aislantes de Mott y aislantes topológicos (FIS2011-23230).**
   - Financed by: Ministerio de Economía y Competitividad.
   - Duration: January 2011 - June 2012.
   - Main researcher: Enrique García Michel (UAM)

8. **Observación directa de las propiedades individuales y colectivas de vórtices en superconductores mediante espectroscopia túnel de barrido. VORTEX MATTERS. FIS2011-23488.**
   - Financed by: Ministerio de Ciencia e Innovación.
   - Duration: January 2012 - December 2014.
   - Main researcher: H. Suderow (UAM-INC)

9. **Fabricación de dispositivos fotónicos basados en silicio nanoestructurado mediante la escritura con haces de protones.**
   - Financed by: Universidad Autónoma de Madrid y Banco de Santander. Proyectos de Cooperación Interuniversitaria UAM-Santander con Asia.
   - Duration: July 2013 - December 2014.
   - Main researcher: Vicente Torres Costa (UAM)

10. **Third Generation Solar Cells.**
    - Financed by: Universidad Autónoma de Madrid y Banco de Santander. Proyectos de Cooperación Interuniversitaria UAM-Santander con Asia.
    - Duration: July 2013 - December 2014.
    - Main researcher: Rafael Pérez Casero (UAM-CMAM)

11. **Desarrollo de programa de actividades +D multidisciplinares de la instalación Científico-técnica singular del centro de tecnologías para la fusión, Technofusión. S2009/ENE-1679.**
    - Financed by: Comunidad de Madrid: Programas de actividades de I + D entre grupos de investigación.
    - Duration: January 2010 - December 2013.
    - Main researcher: Angel Ibarra (CIEMAT)

12. **Micro y nanoestruturación de materiales fotónicos inducida mediante irradiación ionica y luz laser MAT2011-28379-C03-02.**
    - Financed by: M. Ciencia e Innovacion.
    - Duration: January 2012 - December 2014.
    - Main researcher: José Olivares (CSIC-CMAM)

    - Financed by: MICINN.
    - Duration: January 2012 - December 2013.
    - Main researcher: José Olivares (CSIC-CMAM)

14. **Taller internacional sobre la modificación y análisis de materiales para las futuras fuentes energéticas. Accion Complementaria MAT2011-13437-E.**
    - Financed by: MICINN.
    - Duration: January - December 2012.
    - Main researcher: Alessandro Zucchiatti (UAM-CMAM).


17. T. Pradell, RS. Pavlow, P.C. Gutiérrez, A. Climent-Font, J. Molera, Composition, nanostruc-


CONFERENCES

Invited


2. M.A. Ramos, Boson peak and low-temperature properties of isomeric butanol glasses. 7th International Discussion Meeting on Relaxations in Complex Systems, Barcelona (Spain), 21-26 July 2013.


4. A. Zucchiatti. Materials research at CMAM. Ion Beams 12 International Conference, Legnaro (Italy), 6-8 June 2012.


9. M. A. Ramos. Ion-beam induced magnetism in pure carbon materials. 8th International Workshop on Magnetism and Superconductivity at the nanoscale, Comarruga (Spain), 1-5 July 2012.


Oral Contributions


Poster Contributions


30. A. Climent-Font, E. Punzón-Quijorna, V. Torres-Costa, RJ. Martin-Palma, M. Manso-Silvan, Ion Beam damage in c-Si by MeV Si ions irradiation. 21th Intern. Conf on Ion Beam Analysis, Seattle (USA), 23-28 June 2013.


32. E.Z. Fratczak, J.E. Prieto, M.E. Moneta. Study of α,ε and γ-phases of iron nitride thin films. 17th International Conference on Radiation Effects in Insulators (REI-17), Helsinki (Finland), 30 June-5 July 2013.


SEMINARS OF CMAM MEMBERS


SEMINARS AT CMAM


4. Thuto Makgato, University of the Witwatersrand, School of physics, Johannesburg, South Africa. Ion-solid interactions in diamond and graphite. 6 March 2012.


7. José Olivares Villegas, CMAM, Madrid, Spain. In-situ optical reflectance characterization of ion-beam irradiation damage on the crystalline (quartz) and amorphous (silica) phases of SiO₂. 11 October 2012.


10. Sara Azimi, National University of Singapore, Singapore. Three-dimensional silicon micro and nano machining. 8 March 2013.

11. Frédéric Garrido, Centre de Spectrométrie Nucléaire et de Spectrométrie de Masse, CNRS-IN2P3-Univresité Paris-Sud, Paris, France. Study of materials properties with ion beam channeling. 12 March 2013 (10th anniversary celebration).


15. Aurelio Climent Font, Universidad Autónoma de Madrid, CMAM, Madrid, Spain. Deviations from Rutherford elastic scattering cross sections for Cu and Zn with He ions. 6 June 2013.


18. Milko Jaksic, Ruder Bošković Institute, Zagreb, Croatia. Heavy ion microbeam, a sensitive probe for materials characterization and an efficient tool for materials modification. 17 July 2013.


Ph.D. THESIS

Daño por excitación electrónica en SiO₂ mediante irradiación con iones pesados de alta energía

Defended by Javier Manzano Santamaría at the faculty of Sciences of the Universidad Autónoma de Madrid, on 8th November. Supervisors: José Olivares and Ángel Ibarra.

This thesis has performed a systematic study on amorphous and crystalline SiO₂ trying to understand the damage processes originated in the electronic excitation induced by high energy heavy ions. The aim has been to study the creation of point defects in both materials and structural changes induced by radiation.

M.Sc. THESIS

Gamma-ray production cross section determination with application to material analysis: natLi, 19F


PIGE is an analytical technique which exploits the interaction of charged particles between 1 and 10 MeV with nuclei located near the surface of the sample to determine the composition and structure of the surface regions of solids, (0 - 50 µm) measuring the characteristic gamma rays that are emitted. The problem of this technique, with better resolution (beyond the surface) than other techniques of ion beam analysis, is the reliability of the analytical results due to insufficient knowledge of the evolution of the γ-ray production cross section with energy, which is a key part as also is the specific energy loss in the material. The ultimate goal of this work is to obtain information about the cross sections of a series of nuclear reactions with the intention of clearing the differences between previous works and incorporate the results into a IBA techniques database for the whole international scientific community. The work developed during the M.Sc. Thesis has focused in Lithium and Fluorine gamma reactions, firstly, performing an assessment of all the existing data and later, measuring with high energy precision gamma yields for the same elements.
### VISITS TO CMAM

<table>
<thead>
<tr>
<th>Name</th>
<th>Institution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Prof. Piero Corvisiero</td>
<td>Dept. of Physics, University of Genova and INFN, Genoa, Italy</td>
</tr>
<tr>
<td>Prof. Douglas D. Osheroff</td>
<td>Dept. of Physics and Applied Physics, University of Stanford, USA</td>
</tr>
<tr>
<td>Prof. Blas Cabrera</td>
<td>Dept. of Physics, University of Stanford, USA</td>
</tr>
<tr>
<td>Prof Jürgen Fassbender</td>
<td>Institute of Ion Beam Physics and Materials Research Helmholtz-Zentrum Dresden-Rossendorf, Dresden, Germany</td>
</tr>
<tr>
<td>Dr. Giancarlo Rizza</td>
<td>Laboratoire des Solides Irradiés, Ecole Polytechnique, Paris, France</td>
</tr>
<tr>
<td>Dr. Milko Jaksic</td>
<td>Ruder Bošković Institute, Zagreb, Croatia</td>
</tr>
<tr>
<td>Dr. Natko Skukan</td>
<td>Ruder Bošković Institute, Zagreb, Croatia</td>
</tr>
<tr>
<td>Dr. Gastón García</td>
<td>ALBA-CELLS, Barcelona, Spain</td>
</tr>
<tr>
<td>Dr. Caterina Biscari</td>
<td>ALBA-CELLS, Barcelona, Spain</td>
</tr>
<tr>
<td>Dr. Wilfried Schildkamp</td>
<td>ALBA-CELLS, Barcelona, Spain</td>
</tr>
<tr>
<td>Prof. Peter Townsend</td>
<td>University of Sussex, Brighton, UK</td>
</tr>
<tr>
<td>Dr. José Luis Ruvalcaba Sil</td>
<td>Dept of Experimental Physics, Universidad Nacional Autónoma de México, México City, México</td>
</tr>
<tr>
<td>Prof. Elias Sideras Haddad</td>
<td>Materials Physics Research Institute, School of Physics University of the Witwatersrand, Johannesburg, South Africa</td>
</tr>
<tr>
<td>Dra. Carmen Vela</td>
<td>Secretaria de Estado de Investigación, Desarrollo e Innovación</td>
</tr>
<tr>
<td>Dra. Elisa Durán</td>
<td>Directora general adjunta de Fundación “la Caixa”</td>
</tr>
<tr>
<td>Dr. Jorge Sainz González</td>
<td>Director general de Política Universitaria del Ministerio de Educación, Cultura y Deporte</td>
</tr>
<tr>
<td>Dr. José Ignacio Fernández Vera</td>
<td>Director General de la Fundación Española para la Ciencia y la Tecnología</td>
</tr>
<tr>
<td>Dr. Chris G. Ryan</td>
<td>CSIRO, Exploration and mining, Clayton, Australia</td>
</tr>
<tr>
<td>Kurdistan scientific group</td>
<td>A group of Kurdistan scientists invited by the Fundación Parque Científico de Madrid</td>
</tr>
<tr>
<td>South Korea scientific group</td>
<td>A group of South Korea scientists invited by the Fundación Parque Científico de Madrid</td>
</tr>
<tr>
<td>Czech Republic representatives</td>
<td>Delegates of the Czech Republic Ministry of Industry and Commerce, invited by the Fundación Parque Científico de Madrid</td>
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### VISITS OF CMAM MEMBERS TO OTHER INSTITUTIONS

<table>
<thead>
<tr>
<th>Name</th>
<th>Institution</th>
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<tbody>
<tr>
<td>Dr. Alessandro Zucchiatti</td>
<td>Centre de Recherche et Restauration des Musées de France, Paris, France</td>
</tr>
<tr>
<td>Dr. José Olivares Villegas</td>
<td>Institute of natural Sciences, Ural Federal UniversityEkaterinburg, Russia</td>
</tr>
</tbody>
</table>
COLLABORATIONS

1. ALBA – CELLS, Cerdanyola del Vallès, Barcelona, Spain.
2. Asociación Euratom Ciemat, Madrid, Spain.
3. Centre de Recherche sur les Ions, les Matériaux et la Photonique, Caen, France.
6. Centro Nacional de Aceleradores, Sevilla, Spain.
7. Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas (CIEMAT), Madrid, Spain.
8. Commissariat à l’énergie atomique et aux énergies alternatives (CEA), Saclay, France.
9. Departamento de Física Aplicada, Universidad Autónoma de Madrid, Madrid, Spain.
10. Departamento de Física de la Materia Condensada, Universidad Autónoma de Madrid, Madrid, Spain.
11. Departamento de Física de Materiales, Universidad Autónoma de Madrid, Madrid, Spain.
15. Inst. de Ciencia de Materiales de Madrid, Consejo Superior de Investigaciones Científicas, Madrid, Spain.
17. Inst. de Óptica, Consejo Superior de Investigaciones Científicas, Madrid, Spain.
22. Istituto Nazionale di Fisica Nucleare, Genova, Italy.
23. Laboratorio Beni Culturali (LABEC), INFN Firenze, Italy.
25. Museo de America, Madrid, Spain.
26. Nanotech Laboratory, Indian Institute of Technology, Dehli, India.
27. National University of Singapore, Centre for Ion Beam Applications, Singapore, Singapore.
28. Ruder Bošković Institute, Zagreb, Croatia.
29. Universidad Carlos III de Madrid, Leganés, Spain.
30. Universidad Complutense de Madrid, Depto. de Física Atómica, Molecular y Nuclear, Madrid, Spain.
31. Universidad de Genova, Dipartimento di Fisica, Genova, Italy.
32. Universidad de la Havana, La Havana, Cuba.
33. Universidad de Leipzig, Leipzig, Germany.
34. Universidade de Lisboa, Lisboa, Portugal.
35. Universidad de Sevilla, Sevilla, Spain.
36. Universidad Federal de los Urales, Ekaterinburg, Russia.
37. Universidad Nacional Autónoma de Mexico, Mexico DF, Mexico.
38. Université Paris Sud, Orsay, France.
40. Universidade Tecnica de Lisboa, Lisboa, Portugal.
41. Université Paul Sabatier, Toulouse, France.
42. University of the Witwatersrand, School of physics, Johannesburg, South Africa.

**COURSES AND TRAINING**


2. **Training stages for the Erasmus students of the Physical Measurements Department. IUT A-Toulouse 3 University, France (in collaboration with dept. of Applied Physics, UAM).** Students: Guilhem Rivals, Gaetan Riviers. Tutor: Maria Dolores Ynsa, Aurelio Climent Font.

3. **Stage of Erasmus students from the Berlin University, Germany.** Students: Stephanie Marquardt. Tutor: Aurelio Climent Font.

4. **Stage of Ph.D. students from the University of the Witwatersrand, Johannesburg, South Africa.** Students: Thuto Nelson Makgato, Nimhal Daya. Tutors: Miguel Ángel Ramos, Alessandro Zucchiatti.

5. **Stage of Erasmus students from the University of Guadalajara, México.** Student: Sandra Camarena de la Mora. Tutor: Alessandro Zucchiatti.

6. **Interuniversity Master in Nuclear Physics.** Lecturers: Aurelio Climent Font, Ángel Muñoz Martín.

7. **Master in Synchrotron radiation and particle accelerators.** Lecturer: Ángel Muñoz Martín.

8. **High specialization Master’s degree in plastics and rubber.** Lecturer: Ángel Muñoz Martín.
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 5MV using the Cockroft-Walton power supply system (previously, terminal voltages were never higher than 3MV with this system and the power supply itself was perpendicular to the acceleration stage). It has a remarkable stability and low ripple (less than 50V at 5MV). 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. 1a shows the demand of ions during the period 2012-2013. New irradiation campaigns, the commissioning of the internal microbeam and the participation of CMAM in an international coordinated research project for the development of PIGE have led Hydrogen to be the most demanded ion.

Fig. 1b shows the distribution of time for activities involving the accelerator. At CMAM, several beamline have been commissioned during 2012-2013 and some other improved, resulting in 17% of total time (24% of beamtime). More than a half of total time (76% of beamtime) has been used for scientific experiments, both under free and competitive access and under contract.
A higher than usual time has been needed for maintenance during 2012 and 2013. Fig. 2 shows the evolution of maintenance and beamtime in hours for the last six years. In 2012, extra maintenance time was required for dismounting, cleaning and alignment of the accelerator injector. While these operations are performed, it is not possible to provide any ion beam as the injector block is shared by the two sources and, therefore, none can be operated.

In May 2013, for the first time since its installation in 2002, the accelerator was not able to reach 5 MV of terminal voltage. Several external diagnosis were done and eventually an opening of the tank was programmed for the end of the year. It was also the first time in 10 years the tank had to be opened for maintenance. Inspection of internal components and, in particular testing of rectifiers stacks, confirmed an expected damage of few hundreds of diodes (out of the 2400 used for the Cockcroft-Walton high voltage generator). A first repair, done using some spare diodes, allowed us to work safely at 4.3 MV. A new opening of the accelerator tank is scheduled for 2014 to finish the substitution of damaged rectifiers.

Since 2012, operation and maintenance of the accelerator and its related facilities are audited according to the ISO 9001:2008 standard. The standard guarantees the following of dedicated protocols for each individual task and a continuous improvement of the operation. Fig. 3 shows the evolution of failures in supply of ion beams normalize to the total number of beamtime hours. Since 2010 there has been a continuous reduction in the number of failures that we expect to continue in the following years.
BEAMLINES

Four beamlines were available by the end of 2011, namely Standard Beamline, External Microbeam, Time of Flight and Nuclear Physics Beamline. Two years later, in 2013, another beamline is fully operational, implantation beamline, and a second one, an internal microbeam is almost ready. Besides, two new dedicated experimental stations have been installed as a continuation of the ToF beamline: the first one for measuring secondary emitted electrons under ion bombardment of in situ grown thin films. The second set up is devoted for in situ electrical measurements of irradiated samples at very low temperatures (liquid Helium). Detailed information about the most important improvements carried out in the beamlines and experimental stations can be found in the following reports. Updated information about the beam lines at CMAM can be found in the web site.

Fig. 4 shows statistics about the use of different beamlines, both for experiments and commissioning.

![Beamlines](#)

*Figure 4. Use of beamtime, including experiments and commissioning.*
The time of flight beamline at CMAM was designed to perform Elastic Recoil Detection Analysis (ERDA) experiments with simultaneous particle energy and Time of Flight (ToF) detection. ERDA-ToF method is of particular interest in determining the depth profile of light elements from surface to several micrometers in depth.

ToF beamline is at the $10^\circ$ 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 $40^\circ$ from the beam, collects the particles, measuring in coincidence both energy and time of flight for each 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 42 cm the nominal value. When a particle crosses a time station, a fast signal is generated. This signal is used to feed Fast Preamplifiers and Time 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 elapsed between the production of the signals at the two detectors (time of flight). At the end of the telescope, a solid state detector for charged particles measures the particle energy. Both, time and energy signals, are recorded in list mode. Real time or offline software treatment allows determining which events occurred in coincidence, making possible mass determination for each detected particle. For heavy particles, energy spectra obtained from time spectra are with much higher resolution than the ones obtained directly from energy detector.

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

Commissioning of beamline showed low efficiency of time detectors for light atoms such as Hydrogen and Helium. Therefore, an effort in increasing efficiency was necessary. A significant improvement was done depositing a lithium fluoride (LiF) layer of 30 nm on each side of the carbon foils. The result is an increment of proton detection efficiency up to 80% (Fig. 1).

![Figure 1. Hydrogen detection efficiency (red: energy data, blue: time data in coincidence, green: detection efficiency)](image_url)
Besides, some other improvement arise from the experience obtained during commissioning, namely:

- The beamline Faraday cup was placed between the last pair of slits and the transmission Faraday cup (TFC). The consequent advantage is a better characterization of the TFC transmission factor, resulting in a more precise charge value during an experiment.
- An end-switch has been mounted on the azimuthal movement of the goniometer, and the goniometer has been re-aligned.
- Data analysis code, based on an external software (POTKU from University of Jyväskylä), is being tested to be adopted as routine software to obtain elemental energy spectra and the corresponding elemental depth profiles (Fig. 2).

During the 2012-2013, two papers using data measured at ToF beamline have been published:

Ion beam modification of materials (IBMM) techniques are the basis of a well-established research field. The implantation of ions in a wide variety of materials has found a wide range of applications, such as semiconductor devices fabrication, study of the radiation effect [1, 2], in view of e.g. industrial and space applications. If we work with ion beams in the range 10-500 keV, we can carry out implantations in materials with depths of several hundreds of atomic layers and modify their surface properties. On the other hand, if we use ion energies lower than 10 keV, we can perform ion beam sputtering and plating, ion plasma deposition, and thin layers coating. In higher energy accelerators, such as the one at CMAM which covers, depending on the ion type, a range from 500 keV to 50 MeV, we can work with high electronic stopping power and modify materials using low fluences with penetrations up to several microns. Furthermore, an implantation beamline allows obtaining high displacements per atom (d.p.a.), and high implantation depths, making it an ideal instrument with which carry out radiation damage studies. An implantation beamline may also allow carrying out in situ and on line analysis in the irradiated samples by any of the so-called Ion Beam Analysis (IBA) techniques, optical methods and others. One of the research lines of CMAM and a relevant one in quite a few accelerators around the world is the study of materials involved in the development of new energy sources, such as nuclear fusion [3]. A hypothetical future commercial fusion reactor must be manufactured with materials capable of brook harsh conditions such as strong irradiation damage with high displacements per atom (d.p.a. close to 50-100), high operating temperatures (above 600 °C), and large productions of He and H (from 1 to several hundreds of appm/dpa), or other ions that affect the radiation damage [4-6]. Testing these materials under conditions similar to those that they will stand in the installation, or not too far, within the performances available from actual accelerators, is fundamental to understand their alteration mechanisms and guarantee the proper operation as a reactor component. Conducting experiments on the evolution of the irradiated materials requires keeping under precise control the sample temperature within a wide range, and defining precisely the size of the irradiation area and thus the fluence, which are key parameters in the modification of the material properties. Many ion accelerators have installations capable of carrying out homogeneous implantations on samples with relatively reduced areas [7,8], but it is not common for these installations to have the ability to control the sample temperature within a wide range and on large areas [9]. To extend the potential of the implantation beamline of the CMAM we have installed and tested a new cryostat furnace and modified the beam sweep and monitoring system to allow implantation of ions within a wide range of masses and charge states, in areas up to 10 x 10 cm² on target, with precise control of the sample temperature and the swept area and fluence.
Figure 1. (Upper) Panoramic view of the CMAM implantation and irradiation beamline and (Lower) a schematic diagram with its main elements.
Setup

The implantation CMAM beamline is located at 20° (left) with respect to the Tandem accelerator axis, after the second switching magnet. It has a length of 6 m. Fig. 1 shows a panoramic view of the entire line and a schematic diagram with its main elements, namely the switching magnet, the Faraday cups (FCs), the vacuum valves, the sweep system, and the pumping flanges. The irradiation chamber (Fig. 2) is electrically isolated, and it is designed for ultra-high vacuum (UHV), provided with ConFlat flanges (CF), and compatible with load-lock loading for up to 20-cm-wafers. Its location at 20° and the terminal voltage control done by a GVM (Generating Volt Meter) system, allows transmitting ions from H to Pb, at maximum terminal voltage, in their most abundant charge state, i.e in the highest possible current conditions. The irradiation fluence is controlled by measuring the beam current in two different ways. The system, provided in origin by High Voltage Engineering Europa (HVE), consists either of a single FC, located before the vacuum valve, or a combination of four FCs (4FCs), located after the vacuum valve, immediately before the irradiation chamber (Fig. 1 and 2).

In origin, the four FC’s occupied the vertexes of a fixed 5, 7 or 9 cm-side square inscribed inside one of three circular disks centred on a flange (Fig. 3a). This was a rigid and lengthy to change configuration, which has been replaced by a new system (Figs. 3b and 3c) that allows the independent movement of each of the FCs, and defines square irradiation areas, still centred on the flange, with continuously variable size up to 10 x 10 cm². Figs. 3b and 3c show the set-ups for big (from 10x10 to 7x7 cm²) and small (down from 7x7 cm²) samples,
respectively. The system, designed and fabricated by CMAM, is much more flexible and reduces to minimum the time needed to adjust the beamline to the irradiated samples size while maintaining the highest possible beam flux on target.

Irradiations can be performed under a homogeneous (quasi-static) beam, with precise control of the ion fluence, by using the electrostatic beam sweeper of HVE (Fig. 4) which allows controlling precisely both the position and the dimension of the beam as result of X/Y-offset (oX, oY) and X/Y-amplitude (AX, AY) inputs. The offset deflection is up to 9 mm for both axis being the vertical (Y-axis) and horizontal (X-axis) deflection rates 2 kHz and 31 Hz, respectively. This design allows carrying out different scans, prior configuration of the X and Y steps, with an output resolution independent of the energy/charge (E/q) ratio. Fig. 4b show a typical scan pattern, which can be worked out, for example, in just 64 ms over a 20 x 20 cm² area. Fig. 4c is an internal view of the beam sweeper.
The cryostat/furnace was designed at the CMAM and fabricated by TRINOS Vacuum. The sample holder temperature is controlled by a Watlow power supply, and a Watlow EZ-ZONE software, that allows modifying the heating and cooling ramps and, consequently, programming different heating/cooling recipes in order to vary the sample temperature, ideally from LN\(_2\) (-196 °C) to 600 °C, during the irradiations. The sample can be oriented from 0° to ±90°, with respect to the beam axis, for transfer and on line/in situ characterization, respectively (Fig. 5a). The temperature is measured by two thermocouples located in one of the sides (TC2), and in the middle of the sample holder (TC1), respectively (Fig. 5b). TC1 is the control thermocouple, so that its signal is used for the furnace power supply feedback, TC2 is used to evaluate the thermal gradient distribution on the holder during the experiments. The sample holder is made of copper to favour a uniform temperature gradient. Additionally, the beamline is provided with an InfraTec VarioCAM thermographic camera, and a InfraTec IRBIS software, to study the thermal distribution on the sample during the irradiations.
The irradiation chamber has additional ports, with different geometries with respect to the beam, to host multiple optical characterization tools, such as in situ laser interaction and excitation equipments, spectrometers, ellipsometers as well as particle detectors to perform IBA techniques such as ERDA and RBS. These instruments are not strictly bound to the irradiation chamber. They are of general use in the laboratory but can be easily installed, when required. An extra chamber is also foreseen along the line to install a beam degrader which will produce a “white” beam with continuously variable beam energy and good stability.

**Commissioning Results: Beam Transport**

The CMAM magnetic configuration allows transmitting heavy ions through the implantation beamline at maximum current within the range 2-50 MeV, depending on the ion type. Fig. 6 shows the maximum beam deflection angle versus the mass of ions, with a state of charge of 4, which have been directed towards the beamline by the switching magnet, using magnetic fields of $\approx 0.75$ and $\approx 0.80$ T.m, respectively. STD, IMP and ToF acronyms correspond to the standard, implantation and time-of-flight CMAM beamlines, respectively. As can be seen, the deflection angle decreases with the ion mass being the values (continuous line) lower than those obtained in accelerators similar to CMAM (dashed lines). Assuming a charge state 4 (the most abundant after stripping in many instances), and a magnetic field for the switching magnet of $\approx 0.80$ T.m, we can deflect at $20^\circ$ up to 200-amu ions. Table 1 shows some of the ions accelerated at the CMAM together with their charge states, their corresponding energies and the maximum beam currents achieved on target in the implantation chamber. The tests showed that the separation of the different charge states of the ions is excellent, and ions as heavy as the gold dimer, Au$_2$, can be accelerated, which is not generally achieved in other installations.

![Graphical representation of the maximum deflection angle versus mass of ions, accelerated at the CMAM Tandem accelerator, and switched by the magnet at the beginning of the implantation beamline, using magnetic fields of 0.75 and 0.8 T.M, respectively. The dashed lines correspond to characterizations carried out in accelerators analogous to CMAM.](image)

<table>
<thead>
<tr>
<th>Beam</th>
<th>Charge state</th>
<th>Max. Beam Current (nA) [charge state]</th>
<th>Energy (MeV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^1$H</td>
<td>1</td>
<td>3000 [+1]</td>
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<tr>
<td>$^4$He</td>
<td>1-2</td>
<td>500 [+2]</td>
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<td>$^{12}$C</td>
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<td>$^{19}$F</td>
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<td>$^{28}$Si</td>
<td>1-10</td>
<td>1480 [+3]</td>
<td>4-22</td>
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<td>$^{35,37}$Cl</td>
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<tr>
<td>$^{56,62}$Fe</td>
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<td>$^{79,81}$Br</td>
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<td>12.3 [+3]</td>
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</tr>
<tr>
<td>$^{197}$Au</td>
<td>1-13</td>
<td>700 [+3]</td>
<td>2-28</td>
</tr>
<tr>
<td>$^{397}$Au$_2$</td>
<td>2-4</td>
<td>27.7 [+4]</td>
<td>3-12</td>
</tr>
</tbody>
</table>

Table 1: Some of the ions transmitted through the CMAM implantation line together with their charge states, their maximum beam current on target and their energy.
The cryostat/furnace was checked by varying the sample holder temperature from -180 °C up to 600 °C, which results fully exploitable for experiments. The measurements were performed under a LN2 flow using a heating/cooling ramp of 5 °C/min. The temperature was stabilized during 10 min for intervals of 100 and 50 °C during the high and low temperature testing, respectively. Fig. 7 shows the time trend of the temperature measured by the two thermocouples located at the sample holder, as well as the set-point temperature (SP) marked by the PID system. The system submits perfectly the ramps selected by the software. The graphs show that the temperature read by the two TCs is practically the same after the stabilization time and coincident with the set-point. The maximum difference in the thermocouples signals is ≈ 7 °C, which takes place when the furnace is maintained at 600 °C. This difference corresponds to the TC2 with respect to both the TC1 and the SP, whose values match. This deviation is due to heat dissipation processes that run from inside to outside the furnace, an expected behaviour for this kind of devices. With the aim of studying the thermal distribution on the sample holder during the heating/cooling, we installed the thermographic camera in front of the ZnSe optical window provided in the irradiation chamber (Fig. 2). Fig. 8 shows the thermal maps corresponding to the furnace sample holder after keeping the furnace at 200, 300, 400 and 500 °C, respectively, during 10 minutes. Table 2 summarizes the SP, the temperatures measured by the two TCs, and the temperature measured by the thermographic camera in the P1, P2, P3, P4 y P5 points marked on the thermo-images, for the four set working temperatures. Although the thermo-images show shadows from the flanges, walls, windows, and other elements of the irradiation chamber, which mask slightly the real values, the temperature is practically homogeneous throughout the holder surface. These results demonstrate that the implantation beamline is ready to perform irradiations/implantations under strict control of the temperature sample within a temperature range from -180 to 600 °C.

<table>
<thead>
<tr>
<th>P</th>
<th>200 °C</th>
<th>300 °C</th>
<th>400 °C</th>
<th>500 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>TC1 / TC2</td>
<td>200.5 / 200.0</td>
<td>300.1 / 301.9</td>
<td>400.3 / 403.9</td>
<td>500.0 / 504.8</td>
</tr>
<tr>
<td>P1 / P2</td>
<td>191.2 / 204.3</td>
<td>295.4 / 312.6</td>
<td>398.7 / 421.4</td>
<td>514.1 / 524.2</td>
</tr>
<tr>
<td>P3 / P4</td>
<td>198.5 / 202.3</td>
<td>299.6 / 313.3</td>
<td>400.6 / 419.8</td>
<td>501.3 / 512.6</td>
</tr>
<tr>
<td>P5</td>
<td>189.0</td>
<td>301.2</td>
<td>402.3</td>
<td>499.8</td>
</tr>
</tbody>
</table>

Table 2: Temperatures displayed by SP, TC1, TC2, and the five measure points corresponding to the thermal-images showed in Fig. 6.
Comissioning results: beam sweep system

The best focalization of the beam on target is just below 2 mm in diameter. The beam sweeper can, in principle, cover an area up to \(30 \times 30 \text{ cm}^2\) but to achieve a convenient compromise between particle flux and measuring time it is normally limited to cover the collimated area and the 4FC’s system (maximum \(10 \times 10 \text{ cm}^2\)). The accelerator HVE telemetry software provides a current measurement, corresponding to the single FC or the sum of the 4FCs, respectively from which we can calculate the irradiation fluence based on the area of irradiation set for the sweep system, and limited eventually by the new 4FCs system. The performances of the 4FC’s system have been thoroughly checked and it is possible to perform homogeneous scans varying the irradiation area dimensions from \(10 \times 10 \text{ cm}^2\) to \(5 \times 5 \text{ mm}^2\). In the example of Fig. 9 we observe the light produced in a circular (10 cm diameter) quartz scintillator, irradiated with a swept 2 MeV-hydrogen beam. The irradiations were performed, in the case shown, by using the HVE 4FC system (Fig. 3a) on a 10cm-diameter disk. The quartz was installed on the sample holder with a copper grid in front to avoid charge accumulation. Fig. 9 shows the light corresponding to (a) the hydrogen focused beam without scanning, (b) the beam sweeping the sample with a scan up to \(4 \times 4 \text{ cm}^2\), and (c) the beam sweeping the sample with a scan of \(30 \times 30 \text{ cm}^2\). In the latter case (Fig. 9c), the scan exceeds the irradiation area bounded by the 4FCs system and we can indeed observe the shadows of the disc with the 4 FCs located on it.

First application: waveguides fabrication by ion implantation

A waveguide is a physical structure which consists on three layers of materials, with different refractive index, extended in the direction parallel to their interfaces (Fig. 10a) which has the ability to confine electromagnetic waves and guide them in one direction.

![Figure 10. (a) Schematic drawing of a waveguide and (b) quartz waveguide fabricated at the CMAM implantation beamline by irradiation with a 20-MeV-F\(^+\) beam.](image)

The refractive index of the middle layer must be larger than those of the surrounding layers. The ideal waveguides must be infinite in the direction parallel to the interfaces but, in practice, their size is much larger than the depth of the layer. One way to produce waveguides is implanting ions from a particle accelerator, technique that allows the strict control of several parameters such as ion nature, depth of ion penetration, fluence, and so produces homogeneous and reproducible specimens. In previous CMAM activities, LiNbO\(_3\) waveguides have been fabricated by implantation using Kr and Xe beams [10].
At the upgraded CMAM implantation beamline we fabricated a quartz waveguide by ion implantation, using a F\(^{4+}\) beam. When an energetic ion beam impinges on a material the ions penetrate a certain distance which depends on their initial kinetic energy. Along their path, their energy is transferred mainly through two processes: ionization and structural damage. The amount and localization of these processes is function of the ion, the nature of the irradiated material, and the ion energy. Most of the structural damage takes place near the end of the ion path and the consequence is the formation of a disordered region with lower density, with respect to the original material, and, consequently, lower refraction index that constitutes an optical barrier. The depth at which the barrier appears can be modified by adjusting the ion energy.

Firstly, the IL of the two SiO\(_2\) phases, namely amorphous silica and crystalline quartz, was compared in order to study the structural defects generated by means of the ion irradiation. Irradiations were performed with 10 MeV Cl and 15 MeV Br ion beams. It is well established that every ion impact generates a nanometric amorphous track, even when the electronic stopping power is above a critical threshold value (=4keV/nm [11]), so the damage is cumulative and, even below the threshold, crystalline samples can be partially amorphized. High-purity synthetic quartz samples, with dimensions of 6.0 x 1.0 cm\(^2\), were fixed on a fused silica disk with a diameter of 10 cm (Fig. 11). Both samples were irradiated using an irradiation area of 6 x 6 cm\(^2\), and currents in the range of 10-15 nA to avoid the sample overheating. The IL emission was transmitted through a silica window port, placed at 45\(^\circ\) with respect to the beam and collected and focused with a silica lens into a silica optical fibre located outside of the irradiation chamber. Finally the light was guided to a QE6500 spectrometer, configured for measuring spectra in the range 200-850 nm. Fig. 11 shows representative instants of the irradiations, as well as their corresponding IL spectra. The spectra have been decomposed into two main emission bands, attributed to the non-bridging oxygen hole centres (NBOHC, red band, 1.9 eV) and the emission from self-trapped excitons (STE, blue band, 2.7 eV) [12], respectively. For silica, the blue band is dominant over the red one but, in the case of quartz, the red dominates at low fluences and the blue does it at high fluences (Figs. 11a and 11b). At the end of the irradiation process, the spectra become nearly identical for both phases (Figs. 11c and 11d).
The blue band is assigned to the recombination of the STE corresponding to the strained bonds that are initially present in silica. In the case of the quartz, blue band is developed during the irradiation. Red band is assigned to the NBOHC generated during the recombination of the STE. The silica kinetic behaviour shows a reduction of the yield emission attributed to the compaction of the Si-O-Si lattice. The quartz kinetic behaviour indicates a competition between recombination of NBOHC and STE sites.

Secondly, and from the results presented above, 0.5 x 6.0 cm² quartz rectangular samples were fixed on a fused silica disk and implanted with a 20 MeV F⁴⁺ ion beam from the Tandem accelerator, swept over the whole surface to produce waveguides. Fig. 10b shows the operation of a quartz waveguide fabricated in the CMAM implantation beamline by irradiation with a F⁴⁺ beam with a current of 2.200 mA using a beam scan of 6x6 cm².

**References**

Elastic properties of B-C-N films grown by N$_2$-reactive sputtering from boron carbide targets

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Boron-based compounds have been extensively investigated as hard coatings and as semiconductors for high-temperature applications [1]. They also present a wide diversity of functional properties ranging from wide band-gap insulators with very good thermoelectric properties at high temperatures [2] to high-temperature superconductivity [3]. Boron carbide is specially interesting due to its exceptional mechanical properties that include high hardness (even at high temperatures), low friction coefficient, high wear capability and chemical stability. In the last years ternary systems based upon B-C-N have been subject of extensive studies aiming to obtain a combination of specific properties of boron carbide, cubic and hexagonal boron nitride. On the other hand, the large absorption cross section for thermal neutrons of $^{10}$B isotope (20% present in natural boron) allows the application of boron carbide thin films in solid-state neutron detectors.

Unfortunately, boron carbide and boron carbonitride thin films present delamination [4] when the film thickness lies about 0.5 µm. The mismatch of mechanical properties between the hard film and the substrate together with the existence of huge internal stresses are at the basis of the bad adhesion of the film to the substrate.

In this work [5] we have analyzed BCN films, deposited by reactive RF-magnetron sputtering on Si(100) substrates, by a combination of techniques. Elastic Recoil Detection Analysis with Time-of-Flight (ERDA-ToF) has been used to characterize the composition and High-Resolution Brillouin Spectroscopy (HRBS) has been applied to measure the velocity of surface acoustic waves (vSAW) which is directly related to the tensor of elastic constants and thus to the mechanical properties of the film material.

Figure 1. Left: Energy vs. time of flight scatter plot of ERDA data obtained for a 87 nm-thick BCN film prepared by reactive sputtering with 60% N$_2$-content on a Si(100) substrate. Right: Intensity of branches corresponding to each element.
The structural and compositional characterization carried out by ERDA-ToF, Raman and FTIR shows that nitrogen incorporation into boron carbide is not effective and results in phase segregation, producing amorphous carbon and hexagonal-structured boron nitride compounds. The formation of a boron oxide layer on the sample surface is also reported, as previously observed. Elastic properties in nitrided boron carbide thin films were studied by means of high resolution Brillouin spectroscopy. The combination of experimental data with numerical simulations renders a film composition that agrees with the picture obtained from atomic and chemical characterization techniques. As a result, the elastic behaviour is dominated by amorphous carbon compound and is influenced by hexagonal boron nitride and surface boron oxide. This explains the fact that surface acoustic waves velocities measured in nitrided boron carbide thin films are lower than in previously studied boron carbide thin films. The corresponding Young’s modulus is much lower than that of B$_{5.6}$C but clearly larger than typical values for polycrystalline metals. Thus, B-C-N films can be advantageously used as buffer layers between hard coating layers and metallic substrates in order to provide mechanical stability under temperature oscillations.

References

Growth and characterization of epitaxial iron-nitride thin films

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The Fe-N phase diagram has been studied in great detail. Nevertheless, there is still a debate on the most efficient way of obtaining thin films of some of the desired iron nitrides phases. We have presented the results of our study on the preparation and characterization of thin films of Fe-N compounds [1] focussing on the low and high nitrogen-content phases and transformations between them due to irradiation, temperature and time exposure. Iron-nitride thin films are very promising for magnetic applications [2]. Films of the FeN alpha (α”-Fe16N2, epsilon (ε-FeN, with 2 ≤ x ≤ 3) and gamma (FeNγ, with y > 0.5) phases were deposited by Molecular Beam Epitaxy (MBE). We aimed at the production of the α”-phase in its purest possible form; we grew the non-magnetic ε-phase and worked also on iron mononitride FeN, which is known to exist in different phases. There is a debate on the exact crystal structure and the coexistence of these phases. On the basis of room-temperature Conversion Electron Mössbauer Spectroscopy (CEMS) and Rutherford Backscattering Spectroscopy (RBS) experiments, we addressed the synthesis of the α”-, ε- and γ”-phases of iron nitrides by applying several variants of the deposition method and exploring different combinations of growth parameters in order to obtain the possible purest phases in a controlled way. N-assisted MBE and/or postnitriding can be applied successfully to produce many of the different iron nitride phases. The metastable character of iron nitrides makes growth parameters, such as deposition temperature, pressures of nitrogen and hydrogen in the atomic source, deposition rates, etc. key factors in determining the phases which are formed.

Figure 1. RBS spectrum for an ε-FeN thin film measured with He ions at 3.705 MeV.

Figure 2. Room-temperature CEMS spectrum for a film of the γ”-FeN phase.
The highest fraction (24%) of $\alpha''$-Fe$_{16}$N$_2$ was obtained by postnitriding an epitaxial Fe layer at 200°C. By postnitriding $^{57}$Fe with a mixture of N an H at a temperature of 200°C we obtained the nearly pure paramagnetic $\varepsilon$-Fe$_{21}$N phase. A representative RBS spectrum is shown in Fig. 1. At low deposition temperatures (below 150°C) and high nitrogen pressures, N-assisted MBE growth was applied to produce the phases present at the high N-content side of the phase diagram. We find that $\gamma''$-Fe$_y$N (with $y > 0.5$) is formed. Although the Mössbauer fits show the existence of two phases, characterized by a singlet and a doublet component, as shown in Fig. 2, we attribute the singlet to the pure $\gamma''$-FeN phase and the doublet to a $\gamma''$-FeN phase containing vacancies.

References
CMAM-JANNUS collaboration to improve on the study of fusion materials

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On February 2013, CMAM and the Joint Accelerators for Nano-science and Nuclear Simulation platform (JANNuS) started a collaboration for developing Ion Beam Induced Luminescence (IBIL) as a technique for in-situ studying fusion materials during single, dual and triple ion beam irradiation.

Amorphous silica (a-SiO\textsubscript{2}) was chosen as first candidate for the application of the technique as it is a fundamental material in fusion technology: optical viewports, plasma diagnostics, and safety and control systems are based on fused silica. In particular, KU1 fused silica is one of the main candidates for these purposes. In fusion reactors, degradation of optical and structural properties of a-SiO\textsubscript{2} due to neutron irradiation will be an important issue. The induced damage presents a considerable number of differential features not yet sufficiently understood.

IBIL is a very sensitive technique for the analysis of impurities and defect centers, such as those introduced by irradiation. Therefore, in-situ ionoluminescence during ion beam irradiations can be used to investigate the microscopic processes accompanying the generation of damage, the formation of color centers and its kinetic evolution with the irradiation fluence. We have widely used the IBIL technique at CMAM, so what we did was to transport our IBIL system to JANNuS and to install it on the triple beam irradiation chamber. We carried out some tests to check if it worked and if we could acquire good spectra with this new setup. In Figs. 1a and 1b we can see how the optical fiber was placed in the middle of the three beams and normal to the sample surface. We tried to reduce the distance from the fiber to the sample as much as possible in order to increase the solid angle and obtain better spectra.
During the stay at JANNuS in 2013, we could also use a Raman spectroscopy system to measure some Raman spectra of fused silica before and after irradiation with single and dual beam. Some differences could be appreciated as it is shown in Figs. 4 and 5.

We also studied the helium diffusion in fused silica. We made a $^3$He implantation at JANNuS, the energy of the beam was 3MeV, the dose $5.5 \cdot 10^{16}$ ion/cm$^2$, and we used liquid nitrogen to cool the sample at -176 °C during implantation. The implantation depth was calculated with SRIM code and it was 12.5μm. After the implantation, the sample was kept at room temperature for 8 hours. Then we irradiated with a deuterium beam at 1.3 MeV to detect the $^3$He through the nuclear reaction $^3$He(d,p)$^4$He (which has a resonance at 440 keV). We used a TiN sample, also implanted with $^3$He at the same conditions, as a reference. Three different positions in the sample were analyzed with this technique and we could appreciate that there was not $^3$He remaining in the sample, as it is shown in Figs. 6 and 7.
Fe-ion irradiation to simulate neutronic damage on structural reference ODS EUROFER and EUROFER97 steels for fusion applications

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High energy fusion neutrons produce atomic displacement damage and nuclear transmutations atoms (H, He) within the irradiated materials. From the point of view of structural materials, these microstructural changes will induce severe mechanical properties degradation, leading to strong hardening and/or embrittlement effects. In the fusion radiation environment neutron damages up to 150 dpa and ~10appmHe/dpa and ~45appmh/dpa are expected.

Within the Fusion Materials Community one of the major scientific challenges related with the structural steels is to obtain a deep knowledge of the interactions between irradiation damages (dpa) and gas atoms (He and H) and their effects on the microstructural and mechanical properties. Nowadays, ion accelerators are being used to investigate the irradiation effects on structural materials at accelerated damage rates up to high doses with the benefit that this method do not active the material. Self-ion irradiation (Fe-ions) is useful to provide accelerated damage rates studies under carefully controlled conditions.

The aim of this work is to determine the mechanical properties change due to irradiation level in comparison to un-irradiated material and to establish the correlation between the microstructure and mechanical properties.

The materials investigated in this study were the reduced activation ferritic/martensitic steels EUROFER97 and EU-ODS-EUROFER. Both alloys have the identical chemical composition (wt. %): 0.11C, 8.7Cr, 1W, 0.10Ta, 0.19V, 0.44Mn, 0.004S, balance Fe, except that the EU-ODS EUROFER contains 0.3% of Y₂O₃ particles. The steels have been studied in the normalized (980 °C 27') plus tempered (760 °C/90'/air-cooled) condition for EUROFER97 and normalized (1150 °C/60') plus tempered (750 °C/120'/air cooled) for EU-ODS-EUROFER.

Ion irradiation with Fe-ions was performed on a 5 MV terminal in a Tandetron accelerator at room temperature (Fig. 1). The temperature of the sample was continuously monitored with a thermo-graphic camera and thermocouples placed between the specimens and the holder.

Figure 1: Accelerator specimen holder

The specimens were irradiated at different damage dose from 0.05 to 30 dpa, using the SRIM code to calculate the damage profile. The irradiated samples are being evaluated by nano-
indentation technique using the CSM method, which permit to determine the hardness and the elastic modulus at the same time. Fig. 2 show a scheme of the procedure followed to perform the nano-indentation tests. Matrices with 36 indentation tests are being made for each state, in order to have good statistics.

The preliminary analyses have showed hardness increase in both alloys after irradiation, being more pronounced for EUROFER97 steel (Fig. 3). In addition, the results seem to indicate that the EU-ODSEUROFER is more resistant to self-ion implantation than EUROFER. This behavior is attributable to the addition of Y2O3 particles. Further tests are in progress.
Fused silica damage created by high energy Si, O and He ion irradiation

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Optically polished samples of silica with different OH and impurity content, that are candidate materials for optical components in fusion devices [1] due to their radiation hardness, have been studied: two high purity synthetic silica, KU1 (high OH~820 ppm) and KS-4V (low OH < 1 ppm), and a commercial silica Infrasil 301 (from Heraeus named I301, with low OH < 8 ppm) with higher impurity content (Al~20 ppm). The samples of ~1 mm thickness were covered by a copper mask during implantation, to avoid electric arcs and to define an irradiation area of 5x5 mm\textsuperscript{2}.

Irradiations were carried out with 24.37 MeV Si\textsuperscript{4+}, 13.5 MeV O\textsuperscript{4+} and 2.455 MeV He\textsuperscript{+} in a standard scattering vacuum chamber (up to 10\textsuperscript{-5} Pa). The ion energies were chosen to produce implantation at about 9 µm and the damage was produced mainly by electronic excitation. Depth profiles of energy loss of ions, in both electronic and nuclear processes, were estimated from calculations using SRIM 2008. A beam current of 45 nA, for the different ion masses, has been used in a homogeneous distributed irradiation area of 5x5 mm\textsuperscript{2}, and to discard possible overheating effects of the samples by ion irradiation 1 nA was also used. The maximum temperature of sample during bombardment was ~ 40°C. Fluence was determined by direct current integration from the target using electron suppression. The fluences were from 5\cdot10^{12} to 1.6\cdot10^{15} ions/cm\textsuperscript{2}.

After ion implantation the samples were characterized at CIEMAT by optical absorption, luminescence and microscopy. Increasing the ion fluence up to 1.6\cdot10^{15} ions/cm\textsuperscript{2} we have observed a saturation of the absorption spectra in the Si\textsuperscript{4+} and O\textsuperscript{4+} implanted samples, which indicates defect density saturation. Fig. 1 shows an example of the optical density induced in KU1 silica implanted with 13.5 MeV O\textsuperscript{4+} ions at different fluences from 5\cdot10^{12} to 1.6\cdot10^{15} ions/cm\textsuperscript{2}.

Figure 1. Optical density measured in KU1 silica (implanted minus unimplanted) Implanted with 13.5 MeV O\textsuperscript{4+} ions at different fluences from 5\cdot10^{12} to 1.6\cdot10^{15} ions/cm\textsuperscript{2}

Figure 2. Optical density spectra of KS-4V irradiated with different ions: 24.37 MeV Si\textsuperscript{4+}, 13.5 MeV O\textsuperscript{4+} and 2.455 MeV He\textsuperscript{+} at the same fluence (1.6\cdot10^{15} ions/cm\textsuperscript{2})
The absorption spectra produced with different ions, at the same fluence (1.6 \cdot 10^{15} \text{ ions/cm}^2) and projected range (~9\mu m) are compared in Fig. 2. As it can be seen, the number of created defects is higher in the case of irradiation with Si^{4+} ions and lower in He^{+} implanted samples. The number of defects increases with total energy deposited (i.e., defect density increase with implanted ion mass).

It is worth mentioning that in the three types of silica implanted with 13.5 MeV O^{4+} and 24.37 MeV Si^{4+}, visible surface cracks were detected in the implanted area at fluences just below the fluence required to reach absorption saturation. An example of cracks produced can be seen in Fig. 3 where implanted and unimplanted regions are shown for comparison in the case of KU1 silica (O^{4+} implanted at a fluence of 5 \cdot 10^{13} \text{ ions/cm}^2). The fluences at which the cracks were produced (after ion beam off), correspond to irradiation energy densities estimated between 1023 eV/cm^3 and 1024 eV/cm^3.

During ion irradiation the ionoluminescence (IL) spectrum was measured to study the evolution of different defects. In Fig. 4 is shown the IL spectra of the three types of silica at low fluence (~1.5 \cdot 10^{12} \text{ ions/cm}^2), as can be seen the three grades show different emission spectra depends on the OH and impurity content. The degradation induced by irradiation depended on the OH and impurities concentration. The 1.9 eV luminescent band is directly associated to native OH defects and OH defect created by irradiation. At higher fluences (~1.6 \cdot 10^{15} \text{ ions/cm}^2), the IL associated to the native OH defects are totally removed by irradiation, therefore only the defects created by irradiation (named STE or/and NBOH) contributed to the 1.9 eV emission. At high dose, the three silica types show IL spectra similar qualitatively.

Such IL spectrum has different intensity emission of the luminescent bands (associated to defects) depending of the ion stopping range, due to the different damage applied to the lattice.

References
In the Early Middle Ages, in the 9th century AD, Islamic potters in Iraq succeeded to decorate glazed ceramics with vivid lasting colors showing iridescent effects. These have been revealed to be caused by metallic nanoparticles of silver and/or copper buried in the first layers of the glaze that the potters were able to diffuse through empirical carefully controlled heat treatments [1, 2]. It is known that similar kind of decoration occurs in nature in materials in minerals and living creatures. Sparkling opals, consisting of submicron size silica spheres arranged in a face centered cubic structure or Morpho butterfly’s blue shining color wings consisting of regular rows of thin plates so-called scales, are examples. We can refer to the brilliancy of these colors resulting from reflection and diffraction in the nano-structured material as structural colors, in contrast to pigmentary colors that result from absorption of light by pigments [3]. In the case of the metallic Ag and Cu nanoparticles, the reflection is produced by an additive coherent effect of the surface plasmons in the nanoparticles excited by the incoming light.

In 2006 a collaboration between a group from the Universitat Politècnica de Catalunya, leaded by Trinitat PRADELL and the Centro de MIcro-Análisis de Materiales (CMAM) at Universidad Autónoma Madrid leaded by Aurelio CLIMENT-FONT, was set to study luster ceramics combining elastic backscattering spectrometry (EBS), transmission electron microscopy (TEM) and energy dispersive X-Ray analysis (EDX) [4]. This collaboration has been quite fruitful involving as well other research groups from different institutions like Universitat de Vic (Judit MOLERA), Synchrotron Radiation Source Daresbury Laboratory (Andrew D. SMITH), and Research Laboratory for Archaeology and the History of Art in Oxford (Michael S. TITE).

Recently we have applied new analytical tools to our study of luster ceramics using a 3070 keV He beam at CMAM to simultaneously produce EBS and ion induced luminescence (IL) spectra on archaeological Islamic shards of the IX to XVII century provided by the Ashmolean Museum in Oxford. The analysis of these experimental results have been combined with EDX and TEM analysis made on the same samples at Universitat Politècnica de Catalunya [5].

EBS is a useful technique to study the in depth distribution and the amount of the metallic nanoparticles of Ag and Cu in a glaze mainly composed of SiO$_2$ with other minor components including Na$_2$O, MgO, CaO, and, eventually SnO$_2$ and PbO. Fig. 1 shows one of the shards studied containing SnO and PbO, P693 (Iran-Safavide XVI XVII century), mounted in the sample holder with the EBS spectra (3070 keV He beam backscattered through an angle of 170.5°) of two regions in the sample, without luster (white) and with luster decoration (brown). In this case the glaze contains Pb. It is clearly seen as a continuous signal starting at the highest energy in the spectrum (around channel 1050) as Pb is the heaviest element in the sample. Its high Z value makes Pb extremely sensitive for EBS. In fact the average atomic concentration of Pb present in the glaze obtained from this EBS spectrum is 1.65 ± 0.05 %. The sharp peak near channel 350 is the elastic resonance of $^4$He with $^{16}$O occurring
at an energy of 3035 keV. The EBS spectrum of the luster decorated zone readily shows the presence of Cu and Ag in a narrow region below the surface, as it is indicated in the depth concentration profile (atomic fraction) also shown in Fig. 1. Also shown in this figure is the IL spectra simultaneously detected with the EBS at the beginning of the acquisition with an integration time of 30 seconds.

The two IL spectra (luster region; L and no luster region; B) are notoriously different. To remark for the moment being that for the glaze containing lead oxide three main emission bands are seen, at around 450 nm (2.8 eV), 540 nm (2.3 eV) and 680 nm (1.8 eV), and for the luster region the band at 450 nm is somewhat filtered and a new band around 600 nm (2.1 eV) is emerging. The provenance and evolution of all these bands will be discussed later in this report. The IL light spots were also recorded with an optical camera.

Samples P702C and D (also Iran-Safavide XVI XVII century) is an example of glaze not containing Pb. The lack of Pb in the glaze is evident from the EBS spectra of Fig. 2. The luster in this case is based on Cu nanoparticles. Fig. 3 shows the TEM images of the two shards, where the Cu nanoparticles are depicted in the TEM images as a band of dark points (notice the scale of 100 nm for sample P702C and 200 nm for sample P702D). The Cu atomic fraction profiles deduced from the EBS spectra is also shown in this figure. The accordance of TEM and EBS results determining the depth distribution of the Cu nanoparticles is quite good.

According to TEM images, in sample P702C the nanoparticles are bigger, more shallow and concentrated in a narrower region than in sample P720D. The same results concerning the distribution in depth are deduced from the EBS spectra. Fig. 3 also shows the IL spectra comparing the results for P702C (Luster,L, and glaze, B) and the difference in IL from the luster of 702C and 702D.

Considering the IL spectra, contrary to what happened in the luster samples with Pb in the glaze shown in Fig. 1, it is now seen that for the glaze without lead the IL spectra taken in the glaze (B) and in the luster (L) are more similar in shape, although not in intensity, showing a main band around 540 nm. The IL spectrum from the zone with luster is less intense, in accordance to the luminosity of the IL spot shown in Fig. 2, and has an incipient band around 600 nm. The IL spectra from the luster region in P702D shows a relative increase in the band at 600 nm as compared to sample P702C, again the scales for each spectrum are different. The effect of the Cu luster layer would be either to filter the IL produced below the luster layer and enhance a band around 600 nm, or selectively filter more efficiently the part of the spectrum centered at about 540 nm. According to the difference in scale of the intensity of the IL spectra, the luster with smaller particles would have a more efficient filtering action.

The two cases reviewed here illustrated in Figs. 1 to 3 resume the main features observed in the initial IL of a set of luster ceramic samples with a glaze containing Pb, and a luster layer with Cu and Ag nanoparticles, and another set with a glaze free of Pb and with luster nanoparticles of Cu and a negligible amount of Ag. Under the irradiation of 3070 keV He, the glaze containing Pb shows, as previously mentioned, a IL spectrum with 3 main emission bands at 450 nm (2.8 eV), 540 nm (2.3 eV) and 680 nm (1.8 eV), while the glaze without Pb only shows a major emission band at 540 nm. This band at 540 nm is attributed to SiO₂, while the presence of Pb enhances IL emission bands around 450 nm and 680 nm. Besides, the presence of Cu enhances an IL emission band at around 600 nm (2.1 eV). Silver nanoparticles from the luster layer would absorb, via surface plasmon
excitation, the emission band at 450 nm. In conclusion the detection of IL simultaneously with EBS measurements may add extra valuable information for the characterization of the extraordinary decoration technique of luster.

Figure 1. Luster shard P693 (Iran-Safavide XVI XVII century), mounted in the sample holder with the EBS spectra (He 3070 keV) of two regions in the sample, without luster (white) and with luster decoration (brown). Also shown the corresponding IL spectra (L for luster and B for no luster) and the atomic fraction for Ag and Cu obtained from the EBS spectra.

Figure 2. Luster shards P702C and D (Iran-Safavide XVI XVII century), mounted in the sample holder with the EBS spectra (He 3070 keV) of two regions in the sample P702C, without luster (white) and with luster decoration (brown) and P702D (only brown luster). Also shown is the IL luminescent spots taken during the EBS acquisition at dark.
Figure 3 TEM images of the luster shards with corresponding IL spectra and concentration profiles.

References
Resistivity Measurements of iron rich alloys at cryogenic temperatures

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In order to identify suitable structural materials for fusion reactors and establish boundary conditions for such materials in operation, it is crucial to fully understand the radiation effects, which can be done using a physically-based modelling approach. This can be achieved undertaking computational simulations to reproduce well-designed experiments carried out under well controlled irradiation conditions on model alloys. This method allows validating parts of our models can provide additional information on the atomistic mechanisms responsible for defect evolution. This approach is non-straightforward and still strong efforts need to be made to develop the models and to obtain new experimental data. Electrical resistivity measurement technique on ion-irradiated samples at cryogenic temperature with follow-up capability during thermal recovery will help establishing the configuration and dynamics of defects induced by ion irradiation. This task is of high importance in Materials European programme as it is part of the of EFDA work-programme milestones since 2008.

CIEMAT and CMAM have developed an experimental set-up (Fig. 1) able to perform such kind of experiments of resistivity measurements at low temperatures on “in-situ” irradiated samples. Objective of these measurements is getting better understanding of creation and migration mechanisms of defects and impurities in reduced activation ferritic/martensitic (RAFM) base alloys. This kind of experiments has been rarely done using ions as accelerated incident particle. Since 2008, Fusion Materials unit of CIEMAT concurrently with a group from DEMOKRITOS laboratory in Athens (Greece) have been implementing this technique under an EFDA project. Currently each group is focusing efforts on obtaining different (but complementary) experimental data related to specific model alloys provided by EFDA [1].

The standard experimental procedure for Resistivity Recovery (RR) experiments has been explained in detail in previous CMAM reports [2]. The change of slope of the recovery curve provides the RR spectra curves whose peaks give information related to the kinetics of the defects and the micro-structural evolution as a function of the annealing temperature. In pure Fe the resistivity of the material should increase with the radiation and decrease as the annealing temperature rises up to its original (unirradiated) value, nevertheless such behaviour might be changed in Fe-Cr specimens. The radiation enhance diffusion (RED) will lead short-range order (SRO) changes in Cr distribution [3] and thus changes in resistivity. The SRO will superpose with the defect recovery making RR data of concentrated alloys more difficult to interpret.
First RR results

The resistivity recovery (RR) curve has first been measured on a Fe-5%Cr model alloy irradiated with 5 MeV protons [4]. The procedure to obtain the RR derivative curve is outlined and experimental errors are identified and quantified in ref. [4]. Special care has been taken to use a sample with very low impurity content and low dislocation density ($1.2 \cdot 10^8 \text{ cm}^{-2}$) (see Table 1 for details).

<table>
<thead>
<tr>
<th>Composition &amp; Microstructure</th>
<th>Fe-5%Cr</th>
</tr>
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<tbody>
<tr>
<td>C</td>
<td>3</td>
</tr>
<tr>
<td>O</td>
<td>6</td>
</tr>
<tr>
<td>S</td>
<td>3</td>
</tr>
<tr>
<td>N</td>
<td>3</td>
</tr>
<tr>
<td>P</td>
<td>&lt; 5</td>
</tr>
<tr>
<td>Cr</td>
<td>5.40 wt%</td>
</tr>
<tr>
<td>Annealing Temperature</td>
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</tr>
<tr>
<td>Mean Grain Size</td>
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</tr>
<tr>
<td>Dislocation Density</td>
<td>1.2 \cdot 10^8 \text{ cm}^{-2}</td>
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</table>

Table 1. Detailed microstructure of studied material: impurity content (ppm), grain size and dislocation density.

Thus, the recovery spectrum of the Fe-5%Cr alloy (Fig. 2) is not affected by the presence of impurities and only due to the presence of Cr and irradiation defects, which will be mainly Frenkel Pairs (FPs) given that the mean energy of the Primary Knock-on Atoms (PKA) is close to 0.35 KeV. The results obtained for the Fe-5% Cr under 5 MeV proton irradiation are found to be in overall agreement with previous experimental measurements performed under electron irradiation although some differences appear due to the different spatial distribution of the created defects and the higher temperature resolution of annealing steps.
The RR spectrum obtained reveals the appearance of the structure of stages I and II and also a partial suppression of the stage III peak with respect to previous results obtained after electron irradiation. The stage III suppression is explained as a superposition of vacancy recombination effects and short-range ordering (SRO) effects which are apparently dependent on the spatial distribution of defects created during irradiation. Moreover, recombination phenomena are observed beyond stage III up to 500 K.

Figure 2. Isochronal recovery $\Delta p(T)/\Delta p_0$ and its derivative, $-d(\Delta p(T)/\Delta p_0)/dT$ for Fe-5%Cr alloy.

Work in progress

The so-called SRO effects hide (or overlap) the defect recombination effects that computing simulations pretend to reproduce. This is because the rearrangement of Cr atoms is possible (and hence changes in the residual resistivity) while the migration of irradiation defects occurs (30-500K). Thus there is a need to “eliminate” these SRO effects in RR experiments. The materials studied in this research are Fe-Cr model alloy of 5, 10 and 14% Cr. Such selection of Cr concentrations is because it has been observed a change in their SRO around 10% of Cr concentration. At concentrations of Cr below 10 at. % there is repulsive SRO, i.e., a tendency of Cr solute atoms to distance from one another forming an ordered compound. At higher concentrations this behaviour is inversed and short-range clustering prevails, i.e., there is a tendency for the formation of Cr pairs and small clusters [5]. These effects are related to the phase stability and radiation resistance of iron rich alloys.

We are developing a new irradiation technique which might provide RR results where resistivity contributions of SRO and defect recombination can be disentangle. And thus, we will be able to provide new reliable data to be used for validation of modelling.

Moreover, in order to check our assumptions on the new method, we are also performing Mössbauer experiments on 30µm samples. Analysis of the data is in progress and more experiments are planned for the next year in order to complete the study of three alloys: Fe-5Cr, Fe-10Cr and Fe-14Cr.

References

Dissemination Activity
Accelerator for the youngest: the tutorial experience of the “Campus Científico de Verano”

As a university centre the CMAM is fully committed to teaching and training at any level. Thanks to the initiative of the FECYT (Spanish foundation for Science and Technology), the economic support of the “La Caixa” foundation and the coordination of the UCCUAM (Unity for Scientific Culture of the Universidad Autónoma de Madrid) the CMAM has had the pleasure of hosting one of the activities of the Summer Science Camp (SSC or CCV). The program aims at promoting the interest of 15-17 years old students for science, technology and innovation, offering them, through a national selection, about 1800 places to participate in scientific projects designed by university professors and developed in research departments of 16 campuses of International or Regional Excellence. A fantastic opportunity for the students to experience the research work in a multicultural university, which will help them defining the future projection of their studies but also enjoy cultural scientific complementary activities and entertainment on campus.

The CMAM has participated in 2011, 2012 and 2013 hosting in total 84 students: in groups of 7 for each of the four weeks of the SSC. The idea behind of our project was to make the students feel part of the organization and involve them in an experimental activity centered on our accelerator. This was designed in such a way as to be, though in its simplicity, the archetype of a professional research activity: set the problem and review the scientific knowledge on the topic, design and prepare the experiment, run the experiment at the accelerator, analyze the data and comment the results where the fundamental steps. Not an easy task, which we carried on devoting the utmost attention to the programming and organization of each activity so to assure at the same time the easiest students work and the least interference with the CMAM regular research activity.

Involving young students in research has been indeed a rewarding experience for the CMAM members who have animated the program: David Jiménez Rey, Diana Bachiller Perea, Arantza Maira Vidal, Begoña Gómez Ferrer. Taking advantage of the experience these highly selected students had in the high school, we could jointly develop a unique educational experience in the laboratory. It took indeed very little time to see them fully busy in the activity, eager to raise questions, willing to take the responsibility of tasks, concerned about teamwork and, most important of all, conscious of the difficulties of any research activity and of the satisfaction that a well done work gives at the end. The questionnaires that we distributed the beginning and end of the week indicated quite clearly that the expectations of the students were met or surpassed and that their motivation to chose a scientific career was in many cases even stronger than before.

The 2013 SSC was inaugurated, on July 3, by the Secretary of State for Research Development and Innovation from the Ministry of Economy and Competitiveness, Carmen Vela, with a visit to CMAM, where our group of students was participating in the activity “Exploring matter with an Ion Accelerator”. During the visit the Secretary of State has been accompanied by the general director of University Policy from the Ministry of Education, Culture and Sports, Jorge Sainz Gonzalez, the general director of the Spanish Foundation for Science and Technology (FECYT), José Ignacio Fernández Vera, the deputy general director of “Fundación la Caixa”, Elisa Duran, and the Vice Rector for Science Policy and Research Infrastructures of the UAM, Rafael Garesse, who highlighted the importance of the SSC for the University and for the future of research.
Figure 1: Four moments of the intense students activity during the Summer Science Campus, supervised by the CMAM members

Figure 2: Authorities and students posing for the official photo at the end of the inauguration ceremony of the 2013 Summer Science Campus
The International Workshop on Modification and Analysis of Materials for Future Energy Sources, Energy 2012, took place in the campus of the Universidad Autónoma de Madrid in 17-20 September 2012. The WS aimed at reviewing and debating, between scientists and industrial companies, the challenges of energy production in relation to the highly technological materials needed by future energy sources such as nuclear fusion, fourth generation fission, high efficiency photovoltaic and fuel cells. In particular the workshops aimed at discussing how ion beams can contribute, through both modification and analysis processes, to the definition of the properties and suitability of materials and the development of future sources of energy production worldwide. It was proposed by the CMAM and financed by the Spanish Ministry for Economy and Competitivity (MINECO), the government of the UAM and several generous sponsors. The organizing committee had as a honorary president, the deputy-rector Rafael Garesse, was chaired jointly by David Jiménez Rey and Alessandro Zucchiatti and counted with the dedication of Aurelio Climent-Font, Beatriz Renes Olalla, Ángel Muñoz Martín, Jorge Álvarez Echenique, Ana Granados Simón, Diana Bachiller Perea and Begoña Gómez-Ferrer.

The UAM develops extensive scientific research especially along a few outstanding research lines that enjoy international reputation and constitute the backbone of the program “Campus of International Excellence UAM-CSIC”. The workshop followed the mainstream of one of such research lines, namely that on “Nanoscience and Advanced Materials”, a natural consequence of being the CMAM among the UAM support infrastructures of this field with research programs focused in the application of the ion beam techniques to materials for energy production. It is worth remembering that UAM is involved through the CMAM and the Department of Applied Physics in the technological infrastructure named “TechnoFusión” and that Spain and the Madrid Community have excellent infrastructures in the field of energy research and production with strong connections established between research organizations and the Spanish industry.

The international workshop was as a good opportunity for sharing ideas, techniques and results on advanced materials for energy technologies, offering a forum for debate at the crucial moment when we have to select and plan the production activity in view of the implementation of feasibility projects (such as ITER, IFMIF, etc.).

The workshop, organized into 8 plenary talks, 23 oral contributions and a poster session, covered the following topics:

- General interest topics: energy policy, energy demand, international events, use of resources and sustainability.
- Fusion systems and applications: inertial fusion and magnetic confinement, hybrids, waste burners.
- Fission systems and applications: evolution and concepts of fourth generation reactors, including high-temperature and subcritical reactors, waste management.
- Small scale production systems (Solar, Fuel Cells, Wind) and applications: energy efficiency, materials performance, advanced energy cycles.
- Ion beam modification and analysis of materials.
We received 48 delegates, coming from 12 different countries. The workshop was inaugurated by a panel composed of: Prof. J.R. Dorronsoro Ibero, UAM Deputy Rector for Innovation; Prof. A. Martínez Cebrían, UAM Deputy Rector for International Relations, Transfer and Technology; Dr. P. Alonso Miguel, Director of the Technological Cooperation Area of the Madrid Community; and Dr. A. Zucchiatti, Director of CMAM, UAM. The closing ceremony was chaired by Prof. José Maria Sanz Martínez, Rector of the Universidad Autónoma.

The eighth plenary talks were meant at summarizing the most important achievements and guiding the discussion on the key topics of the workshop. They covered:

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<tr>
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<td>CEA, DEN, Gif sur Yvette, France</td>
<td><em>The triple beam facility at CEA Saclay (France): a new platform for materials irradiation, implantation and ion beam analysis</em></td>
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<tr>
<td>A. Ibarra</td>
<td>National Laboratory for magnetic fusion CIEMAT, Madrid, Spain</td>
<td><em>Workshop summary</em></td>
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Workshop overview

The presentations and their discussion demonstrated a thriving research activity around energy and the materials that are needed to exploit each of the available and future sources. The outline of our work was that nuclear energy is undergoing a slow transition that will be based in the medium range on the evolution of fission reactors, for which the technological background is already well established. Meanwhile, the experience of ITER (magnetic confinement fusion) and NIF (inertial confinement fusion) and the constant quest for structural and functional materials, apt to support the extreme working conditions of either inertial fusion or magnetic confinement, are expected to open the way to a realistic production on nuclear fusion energy in the long range. In the short term we expect good and viable results from distributed sources like, in first place, the fuel cells and the photovoltaic devices, that show a constant and significant progress and from the improvement in energetic efficiency that the materials study can guarantee in all electronic devices.

Amongst the tools that the scientific community has to tackle the severe technological problems connected with future energy sources, the ion accelerators can offer today an extended analytical support even at micrometric scale. They represent as well, in their actual configuration, a first step in the study of material behavior under extreme irradiation conditions, being capable inter alia of simulating with multiple beams the effects of neutron irradiation. However, further efforts are needed to implement the dynamic analytical capability of ion beams and to achieve, with the high luminosity machines under construction (IFMIF), close to real environmental conditions.

The workshop would not have been possible without the support received in first place by the Spanish Ministry for Economy and Competitivity (MINECO) and the government of the UAM. The support of the UAM government has been constant since the very beginning of our initiative and the financial and infrastructure support that the concerned deputy-rectors have made available has been decisive for the organization of the event. Without the generous donations of the sponsors our work would have been very difficult (maybe impossible) and several of the attendants would have lost the opportunity to participate.

The Foundation Ramón Areces has allowed us to put together a rich scientific program that includes eight keynote invited speakers among which two; Carlos Alejaldre and Leonard C. Feldman, have given their talk in the framework of the “Ramon Areces Foundation Conferences”.

We are particularly grateful to the International Atomic Energy Agency. It is thanks to their collaboration that we have been able to extend the participation to those who needed financial support to travel to Madrid, especially students. We have been very happy to welcome Dr. Andrej Zeman, of the Department of Nuclear Sciences and Applications who has accepted to give an overview of the many Agency activities in support to R&D on materials for energy.

The Madrid Science Park (Fundación Parque Científico de Madrid), as an institutional partner, has once again offered his kind collaboration. The publication of the Workshop Proceedings in Energy Procedia volume 41c (2013), with guest editors Aurelio Climent and Manuel Perlado, has been made possible by their financial support.

We would also like to acknowledge the support of the technical sponsors, HVEE and Trinos Vacuum, long time partners of the CMAM, the work of the Foro Nuclear and Sociedad Nuclear Española, who have helped us in spreading the information to a wider platform.
of potential participants and the constant attention that we receive from the Laboratories Network of the Madrid Community.

We are indebted to all members of the Program Advisory Committee, chaired by Prof. Fernando Agulló Lopéz, for their efforts and dedication in reviewing the submitted abstracts and structuring the scientific program.

The group of participants to ENERGY 2012

Carmen García Rosales talking about tungsten based alloys
Leonard C. Feldman and “finger” vectors

The closing ceremony chaired by Prof. José María Sanz Martínez, rector of UAM
Quality Management
ISO 9001
TWO YEARS OF THE CMAM
QUALITY MANAGEMENT SYSTEM

The CMAM runs its main activity, the “Delivery of Beamtime to the Accelerator Users”, under a Quality Management System (QMS) according the ISO 9001:2008 standard. It was certified by the company SGS on February 23rd 2012. The implantation of a QMS was motivated by our strong will to be recognized as a laboratory that deserves the utmost confidence because of the quality of service provided to users and was seen both as a way of improving the work organization of the Centre and as an instrument to increase the competitiveness of the CMAM and its users.

The bases of our QMS

Of the eight quality management principles, which were obviously followed in implementing the QMS we emphasized the following four:

a) Process approach

The work done to identify and manage interrelated and interacting processes has lead to a very simple QMS, based on only 10 processes (divided into operative, support and management ones) and 47 documents and registers in total and it is practically a zero paper QMS. The core of the system are the three operative processes under which we run the delivery of ion beams:

1. Beamtime Allocation at the CMAM ion Accelerator

This is supported by the users portal where we receive, process and trace all the beamtime applications. The access is easy, the procedure of proposals submission has been maintained as simple as possible and is conveniently guided and the portal has proved to be a very efficient and friendly instrument of interaction with our users.

2. Supply of ion beams to Accelerator users

Once the beamtime is allocated, the work program that establishes the tasks of operators and supervisors is prepared and made available internally. Its execution is controlled by registers that are filled, as demanded by the involved processes, and technical instructions. Eventual changes of the working plan are as well registered and fully traceable. The registers that are connected with the accelerator use and must be collected daily from different instruments scattered within the lab, are entered in a fully digital form from a tablet while the data connected with the accelerator control program are registered directly from the accelerator computer.

3. Monitoring of Beam Supply activities

Besides the users feedback (which we receive continuously through enquiries, complaints and suggestions) the head of the technical division revises every three months the statistics of beam delivery, the occurrence of failures, the ordinary and extraordinary maintenance actions and addresses an activity summary to the CMAM director for his consideration in the annual review.
b) Involvement of people

People are the essence of our centre at all levels. Their full involvement in the implementation of the QMS has been the key of the positive outcome of our work. Thanks to the competence of our staff, we have developed in house the most relevant digital tools (beamtime applications, purchases, databases) and built a very robust QMS that was certified with only a minor nonconformity and, on the 1st follow-up audit on February 2013, had zero non conformities. The abilities of our staff are really beneficial to the CMAM; their continuous training and involvement in decision making and in quality improvement is one of the targets of the QMS and is supported by a solid training program and by an internal organization fully open to representativeness and individual contributions.

c) Focus on users

Making effective the CMAM quality policy, implies understanding the current and future user needs as well as meeting and anticipating their requirements. The interaction with the users is fundamental. The series of tools that we have implemented for this purpose has been quite appreciated and routinely accessed by our users and has been one of the pillars of the QMS continuous improvement.

d) Continuous improvement

The aim of continuous improvement of any QMS is to increase the probability of enhancing the satisfaction of customer and other interested parties. As a general principle we decided to:

- Promote teamwork, participation and commitment of the staff to achieve the objectives established by the organization.
- Provide staff training to ensure awareness and understanding of quality and its impact on users.
- Establish mutually beneficial relationships with our customers and key suppliers, continuously improve processes and products (“beams”) as a permanent objective.
- Establish regular review of objectives, indicators and audits and make decisions based on facts and data.
- Effectively communicate our quality policy within the organization and users.

How it worked

Two years after its implantation some general considerations can be made, about the efficacy of the system and the perception the users have of it. The outcome of the internal and certification audits and the content of the periodic reports of the quality manager and the heads of the technical and administration divisions point to a reliable and regularly working system. The staff more involved in the activities covered by the QMS have positively incorporated the quality control in their working habits and the CMAM takes advantage from a work organization aimed at achieving the general objectives marked by the quality policy of the Centre. The communication with the users and providers has been definitely positive and has been one of the pillars of the QMS continuous improvement. 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 our efforts. We are grateful to all those who have, are and will cooperate with us in the forthcoming years to allow us improve further and further.
Figure 1. The users satisfaction index by period for the years 2012 and 2013. Four topics are evaluated by the user: The beamtime request, the quality of the beam delivery, the quality of the beam itself and the general satisfaction after performing an experiment.

Figure 2. The users feedback by period for the years 2012 and 2013. Complaints and suggestions are considered. The sudden change between 2012 and 2013 indicated that the users collaboration has greatly improved after a year of application of our QMS.
Figure 3. The number of non-conformities open per month in the years 2012 and 2013.