Elemental Characterization and Speciation
CEEFI

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The Elemental Characterization and Speciation work line of ITN Ion Beam Laboratory (CEEFI/LFI), carries out R&D work on ion beam based nuclear analytical techniques aiming at elemental composition characterization and instrumental speciation methods. Focusing being on applications to small mass samples (self-supported thin films, micro and nanoparticles) and/or small mass structures (deposited and deep laying thin films, embedded micro and nanostructures).
The main issue being originally, particle induced x-ray emission (PIXE) applied to the characterization of airborne material and its impacts, lead to the installation of an aerosol characterization set-up, which includes a DOAS system (operational since June 2007), a meteorological station (operational since January 2008), on-line in the Portuguese Meteorological Institute Urban Stations Network, and a PM10 and PM2.5 sampling station.

The installation, in 2008, of the High Resolution High Energy PIXE (HRHE-PIXE) set-up at ITN, the world first cryogenic high resolution EDS X-ray microcalorimeter spectrometer (XMS) based PIXE system, lead to very important results showing the advantages of these detectors use for both fundamental research as well as applications.

As a consequence of this a revision of the objectives of the CEEFI main work line was carried out, and the emphasis was shifted towards fundamental, technical and software development for PIXE, as well as frontier applications of the technique.

Within the organics of the Ion Beam Laboratory (LFI), CEEFI is the responsible for the maintenance and improvement of PIXE facilities.

In respect to 2011 developments, focus was put in the quantification of qualitative results developments of 2010 and previous. Besides this, the High Energy component of the HRHE-PIXE system installed in 2008, was promoted and the automation of the HRHE-PIXE positioning system was carried out. Very interesting results were achieved using high beam energy PIXE. In respect to the XMS-PIXE system, comparisons to high resolution data obtained in the Institute Josef Stefan WDS system was initiated but only preliminary results could be achieved so far.

Finally, the quest for quantification lead the team to deepen the relation to a more theoretical work group from the Atomic Physics Centre of the University of Lisbon (CFAUL), which collaboration is looking highly promising and providing the first solid ground results.

Regarding frontier applications, geological samples, nanoparticles, thin films, and chemical or electronic structure environment mapping are presently the main scope of CEEFI work line.

Within this context, during 2011, important results on geological samples analysis, thin films and fundamental developments were published.

Work in the scope of aerosol analysis was reduced to nearly zero in 2011, due to the raise of importance of more fundamental work for the proper quantification of achieved technological developments, but also as a consequence of the present general financial context.

Research Team

Researchers

M.A. REIS, Aux., Group Leader
P.C. CHAVES, Post. Doc. Fellow under the EU SPIRITproject.

Students

A. TABORDA, Ph.D. Student, FCT grant

Technical Personnel

R. PINHEIRO (30%)

Collaborators

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Fundamental Developments and Solid State Effects in PIXE

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Objectives

Elemental and speciation characterization methodologies for small mass samples, are gaining importance world wide and are a niche were ion beam analysis (IBA) may come to play an important role, with special emphasis to high resolution particle induced X-ray emission (HR-PIXE). During 2011 most of the line activities therefore focused on mostly on objective of carrying out fundamental developments related to the identification of solid state environment effects on emitting ion, which reflect upon the X-ray spectra details. This objectives are the continuation of the main objectives of the previous year of 2010, and correspond to the objectives stated in a sub-task of Key task 15 of the 7th FP project "Support of Public and Industrial Research Using Ion Beam Technology (SPIRIT)", Grant agreement No 227012-CP-CSA-Intra (starting date of 2009/03/01), in which the team plays an important role.

As a consequence of this work, technical details lead to the establishing of an associated objective, namely the application of these developments to geological samples.

Results

During 2011, the Ph.D. program of P.C. Chaves was concluded. The know-how acquired during the Ph.D. was used in the frame of the SPIRIT project task, to install the X-Y positioning system of the High Energy High Resolution PIXE system, end-station of the ITN 3MV tandetron, and to carry out experiments for comparison of the ITN energy dispersive X-ray microcalorimeter spectrometer (XMS) spectra form a Si crystal target, and equivalent ones obtained using the wavelength dispersion system of the Institute Josef Stefan, which has much better resolution but much shorter energy window.

In the context of the ongoing Ph.D. programme of Ana Taborda, a new collaboration was initiated with the Federal University of Rio Grande do Sul, Brazil and a three months permanence there allowed the study of Gd2O3 and Tb4O7 nanoparticles, irradiated at different energies. Fig.1 (right) show the unexpected differences in the M spectra of Tb4O7 nanoparticles obtained if these are dispersed or packed in a pellet. In a three months stay at UFRGS, Ana Taborda also carried out Medium Energy Ion Spectrometry - MEIS analysis of these nanoparticle samples.

Still within the scope of this Ph.D. programme, the collaboration in the frame of the CFAUL was increased, starting with *ab initio* calculation of Si spectrum data, to be compared with data from the XMS-PIXE spectrum and from the Ljubljana WDS spectrum of a Si crystal sample.

Results for both collaborations displayed important problems with no immediate answer. In the case of MEIS, un-explained shifts were identified, while for *ab initio* calculations, experimental data shows to be more problematic than expected in the capacity to reproduce (even in simple cases such as that of Si target).

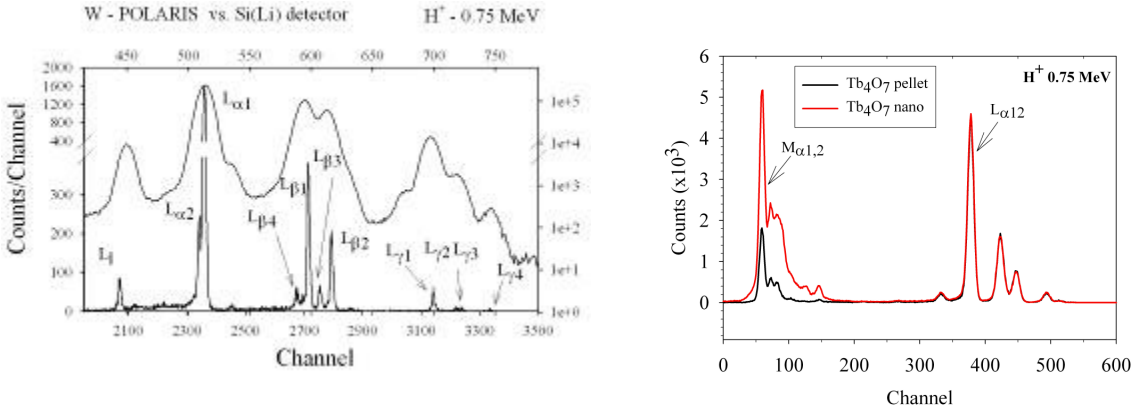

Fig.1 (Left) Comparison of X-ray microcalorimeter spectrometer PIXE system (XMS-PIXE) and Si(Li) spectra of a pure W thick target foil irradiated by a 0.75MeV proton beam provided by the ITN 2.5MV Van de Graaff (Si(Li)) and by the 3.0 MV Tandetron (XMS-PIXE). The clear separation of lines from the XMS-PIXE unit was of major importance for the understanding of results in the scope of P.C: Chaves Ph.D. thesis. (Right) Comparison of the M-shell spectra of Tb4O7 nanoparticles irradiated by a 0.75 MeV protons beam provided by ITN Van de Graaff. and physically disposed in two different ways, namely as a dispersion of nanoparticles in a Nuclepore filter and when packed into a pellet..

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