



## List of Abstracts

### **Ion beam imaging in Brazil: projects, results and perspectives**

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An Oxford microprobe line was acquired in 2007 by the Ion Implantation Laboratory in Brazil, which is the unique existing in the country. The microprobe line was initially applied to proton beam writing in polymers and silicon-based materials. More recently, the microprobe applications are dedicated to imaging of organic and inorganic materials by PIXE and STIM, including food, gunshot residues and biological tissues. The aim of this work is to show the preliminary results obtained by our microprobe setup. For instance, microanalyses of cork stoppers used to seal wine bottles showed that the elements are not distributed homogeneously throughout their surface. Moreover, the region around the lenticels borders presents higher S, K and Ca concentrations and the storage time may have affected the elemental concentrations of some elements, especially K and Cl. Concerning animal tissue, our main goal is the analysis of rat brains submitted to learning sessions. In this case, tissue slice samples are prepared to STIM and PIXE measurements. An important step to biological sample preparation protocol is to guarantee that the images of mass and elemental distribution are the representation of the specific neurophysiological process. In addition, MeV ToF- SIMS equipment was recently acquired and will be in operation this year. Protein analysis will be performed in biological tissues as complementary analyses to PIXE and STIM.



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## **Morphological Study of Microstructured Polymer Foils using STIM with H and He ions**

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Scanning Transmission Ion Microscopy (STIM) provides structural images based on the energy loss of swift ions passing through the sample and therefore it depends on local mass density. That quality makes the technique useful for morphological analysis of microstructures fabricated on homogeneous substrates such as polymer foils.

Proton Beam Writing (PBW) is an important technique for fabrication of various devices with applications on different areas such as microfluidics, tissue engineering substrates and microphotonics, among others. Microstructures obtained by PBW can be an interesting object of study by STIM when fabricated on homogeneous substrates. In this case, STIM can be an important tool for morphological characterization.

In this work we present a study of two different structures obtained by PWB and subsequently submitted to STIM measurements using two different ions, namely protons and alpha particles, in order to evaluate the differences between the analysis by each particle beam considering the energy resolution achieved by them and the effects on the resulting images.



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## **Imaging capabilities of MeV SIMS with 8 MeV Cl beam at Jožef Stefan Institute**

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Particle induced X-ray emission (PIXE) is used at microprobe of Jožef Stefan Institute to measure 2D quantitative elemental maps of biological tissue. To improve chemical and biological understanding of the processes in vivo, supplementary information about chemical bonding and/or molecular distributions could be obtained by heavy-ion induced molecular desorption and a corresponding mass spectroscopy with Time Of Flight (TOF) mass spectrometer. As the method combines the use of heavy focused ions in MeV energy range and TOF Secondary Ion Mass Spectrometry, it is denoted as MeV SIMS [1,2].

At Jožef Stefan Institute, we constructed a linear time of flight (TOF) spectrometer and mount it at our multipurpose nuclear microprobe. A beam of 8 MeV  $^{35}\text{Cl}^{7+}$  could be focused to a diameter of better than  $3 \times 3 \mu\text{m}^2$  and pulsed by electrostatic deflection at the high-energy side of accelerator. Time of flight spectra are currently acquired with a single-hit time-to-digital converter. Pulsed ion beam produces a shower of secondary ions that are ejected from positively biased target and accelerated towards MCP. We start our time measurement simultaneously with the start of the beam pulse and the signal of the first ion hitting MCP is used to stop the time measurement. Standard pulses proportional to the time of flight are produced with time to analog converter (TAC) and fed into analog-to-digital converter to obtain a time histogram.

To enable efficient detection of desorbed fragments with higher molecular masses, which are of particular interest, we recently implemented a state-of art Field Programmable Gate Array (FPGA)-based multi-hit TOF acquisition. New improved high-voltage switching ability with beam pulse duration in the order of 50 ns, a mass resolution of better than 500 is anticipated. We will demonstrate the operation of the system with acquired molecular maps measured on tissue slices produced by a standard shock-freezing and freeze-drying method [3].

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## **Evaluation of ion-induced damage in semiconductor devices using focused ion beams**

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Evaluation of ion-induced damage in semiconductor devices using focused ion beams" and the abstract: "This research is aimed to formulate an experimental protocol able to provide a quantitative characterization of the damage produced by ion beams at low fluences in order to get a largely applicable definition of radiation hardness. Radiation damage induced by MeV-ions in semiconductor rectifying devices has been studied by measuring the progressive reduction of the charge collection efficiency (CCE). The experiment was performed by measuring charge pulse height signals produced by proton beams focused in a selected region of a 4H-SiC epitaxial Schottky diode. The samples were irradiated at different proton energies and the pulse signals were recorded at different applied bias voltages in order to probe different depths of the active regions by considering different energy loss profiles and different extensions of the depletion layer. Assuming that at low fluences the damage is produced only by the creation of vacancy/interstitial pairs, whose density profile can be calculated by the SRIM code, we have interpreted the charge pulse height curves as a function of cumulative ion irradiation on the basis of the Shockley-Ramo- Gunn theory. Finally the fitting algorithm allowed us to estimate the concentration of active trap centers and to provide a quantitative evaluation of the radiation hardness of the material."



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## **IBA applied in the study of Middle Byzantine glass artefacts**

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One hundred and thirty two glass bracelets fragments of different colors and appearances excavated from several Byzantine sites - Issacea, Nufăru, Păcuiul lui Soare, located in Dobrogea, Romania and five mosaic tesserae, all dated to the 10<sup>th</sup>-13<sup>th</sup> centuries A. D. period were measured using external Ion Beam Analysis (IBA) techniques at AGLAE accelerator of the Centre de Recherche et de Restauration des Musées de France (C2RMF), Paris and at the Helmholtz-Zentrum Dresden-Rossendorf (HZDR).

In both labs, the experimental set-up allowed the acquisition of Particle-Induced X-ray Emission (PIXE), Prompt-Induced Gamma-ray Emission (PIGE) and Rutherford Backscattering Spectrometry (RBS) signals induced by the proton-beam bombardment.

The main purpose of this study was the determination of the bulk composition for the investigated glass fragments, in order to obtain clues about the glass-making recipes and raw materials.

One of the archaeological hypotheses to be checked was if the finery items were imports- i.e. products of imperial workshop located in the metropolis or just products of local glass working centers. The experiment intended to verify if the same chemical compositions and working procedures were to be found in the mosaic tesserae and the glass bracelets.

The determined glass compositions shed some light on the employed technology and raw materials and provided information about the pigments providing the color of the glass. Hints about the pigments used to decorate the external surface of some of these archaeological artifacts were also obtained.

The IBA compositional results indicated that at least two kinds of recipes were employed for manufacturing the investigated Byzantine glass bracelets. Thus, most of the samples were found to be soda-lime-silica glass, while some of the analyzed fragments, all from Issacea, turned out to be lead-silica glass, a rather rare kind of recipe for that historical period. All mosaic tesserae turned out to be natron-based glasses.

No connection could be established between the composition of the bracelets and their deterioration status.

As concerning the glass coloring, the blue color was provided from the use of small amounts of cobalt compounds, while the violet resulted from the addition of pyrolusite. The green color was produced by a high content of iron and/or copper, while the opaque red glass featured high concentrations of copper, lead and iron.

For the painted glass bracelets, the most likely candidate for the yellow pigment found on the outer surfaces was lead-tin yellow, while lead-white seems to be responsible for the white paint. However, on a single bracelet fragment, the yellow external decoration turned out to be a gold-based alloy, while for another sample, the white superficial ornamentation was produced by a silver-containing compound.

The reported results represent a pioneering study of glass artifacts unearthed on Romanian territory, further experiments of this kind being envisaged.

The external IBA measurements of the glass items took place in France in the frame and with the financial support of EU FP7 CHARISMA project and in Germany using SPIRIT funding.



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## **Real-Time and RBS channeling investigation of GeSn thin films**

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Alloys of Group IV element, C, Si, Ge and Sn, have received considerable attention as theory predicts unique and intriguing (opto) electronic properties for these alloys. For Ge alloyed with Sn the 13% lattice mismatch is predicted to significantly modify the band structure causing indirect-to-direct transition of the band gap and increased mobility in the strained Ge thin film. Unfortunately the diamond structure of  $\alpha$ -Sn is unstable above 13°C making it difficult to incorporate Sn in the Ge, and the Ge-Sn phase diagram predicts very little immiscibility, in fact limiting thermodynamically stable Sn incorporation to about 1%, which is too low to cause the desired properties. Despite these constraints careful preparation by MBE and CVD has enabled alloy levels of around 10% to be achieved.

Up until now much of the attention has gone into understanding the growth mechanism and properties of the as-grown strained GeSn films. Integration of these strained films into advanced electronic devices will also expose them to thermal treatments and to metallization, which may alter the stability of these metastable films. We have investigated the stability of Sn in GeSn thin films grown on Ge by using real-time RBS during a ramped thermal anneal to monitor their thermal stability during a thermal treatment. Real-time RBS has also been used to monitor the effect of metallization on the stability of Sn in the GeSn layer during their reaction with Ni and Co. RBS / channelling has also been used to establish if any relaxation occurs in the strained metastable films.



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## **Elemental and structural analysis in CPST**

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Introduction of IBA methods applied in Center for Physical Sciences and Technology: two archeological samples from the baptistery that was once held in the church of Žemaičių Kalvarija and one from Pranas Gudynas Restoration Center, where sarcophagus of the singer of God Amon (Ancient Egypt, Thebes, 11th–9th century BC) was conserved, were investigated using PIXE; Niobium films have been deposited by various techniques, including electron-beam evaporation with ion-beam assisted deposition, ion beam sputtering and magnetron sputtering (MS) in CSPT and investigated using RBS spectroscopy.



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## **Surface topography reconstruction by stereo-PIXE**

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## **A possible approach to analyze neurological processes in the brain?**

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Several biological processes could be characterized by the major and trace elements role. Specifically, neurophysiologic functions may be studied in according their elemental concentration modifications. For this purpose, different analytical techniques may be employed. Recently, an ion microprobe system has been installed in Brazil. This facility is located at the Ion Implantation Laboratory of the Physics Institute (Federal University of Rio Grande do Sul). Between another techniques used for elemental analyses of food, technological and biological materials, the microPIXE is a powerful tool to mapping the elemental composition and distribution. Briefly a proton beam interacts with atoms in the sample and characteristics X-rays are emitted and subsequently detected by solid-state detectors. MicroPIXE employs a 1-3  $\mu\text{m}$  beam size and is equipped with a scanning capability. Therefore, it is possible to obtain maps of the elemental distribution in the material and their respective concentration in a micrometric level. The aim of this work is to propose microPIXE as a useful tool to investigate memory formation in animal models. It is know that different types of memory are consolidated in different encephalic structures. Nevertheless, a circuit of integrated encephalic structures such as amygdala, hippocampus and cortex is engaged in memory formation process. Moreover, each structure has a different role in this process. Using microPIXE it is possible to create a map of elemental concentration and distribution of the elements in a brain slice looking for distinct patters among memory types and animal performances. PIXE spectra of brain consist of Mg, Al, P, S, Cl, K, Ca, Fe, Cu and Zn. A previous study demonstrated that the memory acquisition process is characterized by increased synthesis of Ca, Cu, Mg and Zn. The alterations in the element concentrations may indicate their participation in the formation (learning) and accessibility (reminder) of the memory.



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## **Distribution and quantification of trace elements in natural diamonds of Juina, Brazil, using the PIXE and RBS techniques**

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The diamonds from Juína region, Mato Grosso, Brazil, are considered the natural minerals known formed at higher pressures ( $> 670$  km;  $> 22$  GPa). Physical and chemical characteristics of these diamonds provide information on the composition of deep Earth, the Transition Zone and Lower Mantle [1]. Originally, the diamonds studied are irregulars, with size varying from 7 and 10 mm, brown yellowish (samples JU-01F and JU-02F) and colorless (sample JU-03). To determine the distribution and contents of trace elements (N, O, F, Si, Cl and Fe) in these diamonds, PIXE and RBS techniques were used. Images associated with the presence of these elements in the three samples were obtained by PIXE spectra acquired using a microprobe of proton of 3MeV in Surrey IBC[2], with a spot around 20 microns and 3nA of current. Preliminary results show that F, Si, Cl and Fe are punctually distributed, which suggest that these elements form aggregates in the diamond structure. The RBS technique was used to check the absolute contents, to estimate the absolute concentrations was used SIMNRA:  $\approx 320$  ppm for N,  $\approx 2.860$  ppm for O,  $\approx 126$  ppm for F,  $\approx 550$  ppm for Si,  $\approx 440$  ppm for Cl and  $\approx 150$  ppm for Fe. N values are comparable to those obtained for Juína diamonds in other studies [3]. And there are traces of elements like S ( $\approx 420$  ppm) and Rb ( $\approx 120$ ppm), but we need more measures to confirm these contents.

Keywords: PIXE, RBS, diamonds.

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## **Development of Ion Beam Induced Luminescence analysis system at the Zagreb Heavy Ion Microbeam Facility**

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Abstract: We report about the progress in construction of a new Ion Beam-Induced Luminescence (IBIL) system at the heavy ion microprobe facility in Zagreb. In contrast to processes leading to the production of X-rays, in which the inner atomic shells are involved, the luminescence process is related to the transitions of the outermost electrons involved in chemical bonds of atoms. For this reason, IBIL is sensitive to the local chemical environment of compounds and trace substitutes. Therefore, IBIL is very interesting as a complementary technique to PIXE and RBS and can be performed simultaneously. Current experimental set-up is described in detail. Preliminary measurements where IBIL is combined with PIXE using 2 MeV proton beam focused on  $5 \times 5 \mu\text{m}^2$  are reported. IBIL maps were collected simultaneously with PIXE maps of optical fibers cross sections to obtain information on correlation between elemental distribution and luminescence spectra. Additional selection of CVD diamond IL spectra and maps is selected and discussed.



## **Modification of organosilicon thin films induced by ion beam effect during RBS analysis**

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Organosilicon thin films are deposited on silicon substrates using Plasma Enhanced Chemical Vapor Deposition (PECVD). The prepared samples are characterised by Rutherford Backscattering Spectroscopy RBS and Variable angle spectroscopic Ellipsometry (ES). The ion beam irradiation during the RBS analysis seems to induce damage in this kind of materials. This damage is visible to the naked eye as spot of distinguished colour and contrast from the surrounding non exposed surface. The composition and thickness remain invariable even after high ion dose accumulation. This indicates that RBS is still non-destructive techniques in term of composition and thickness. However, The thermal treatment of the prepared samples (intended to induce changes in the optical and electrical properties) decreases the thickness of the deposited films. The ES analysis reveals different behaviour in the formed spots from the surrounding surface. The thickness and refractive index within the spots differ according to the ion beam parameters (incident angle and energy) used for the RBS analysis.



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## **MeV Ion Imaging using combination of IBIL and microPIXE: Preliminary Study**

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## Recombination of hydrogen and deuterium atoms on the metal surfaces

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In the development of the fusion devices like ITER, proper choice of materials, used for inner walls of the reactor is crucial for obtaining the needed performance. In order to obtain the necessary data for modelling the edge plasma and interaction between the plasma and the reactor walls, research on this materials are needed. At the Microanalytical center of the Jožef Stefan Institute we are contributing with the study of recombination mechanisms of hydrogen and deuterium atoms on surfaces of the fusion relevant materials, mostly on tungsten and stainless steel Eurofer.

Our main field of research is the study of vibrational distributions of hydrogen and deuterium molecules, which are formed on the sample surface by different recombination mechanisms. For this purpose we are using the spectrometer for vibrational spectroscopy, which was designed and constructed in our laboratory [1]. Our vibrational spectroscopy relies on the dissociative electron attachment (DEA), where an electron is captured by the hydrogen molecule, forming an intermediate resonant state, which dissociates into hydrogen atom and negative hydrogen ion. Only low energy ions are then collected by the extraction system and detected by the channel electron multiplier. The electron energy is scanned from 0 to 5 eV and because of the dependence of the 4-eV DEA cross-section values and the threshold energy on the vibrational state of the hydrogen molecule, we can distinguish different peaks corresponding to different vibrational states in our spectra[2].

At the moment we are upgrading the system with the differential pumping. The vacuum chamber will be divided into two regions, each with its own turbomolecular pump. On one side (reaction region) there will be a temperature controlled sample surface exposed to hydrogen or deuterium atom beam. On this surface the molecules are formed and enter the spectrometer on the other side (detection region) through a small directed slit. Only molecules from the sample surface will be able to enter the spectrometer, which will simplify the analysis of the obtained results. Besides this upgrade, the resolution of the spectrometer will also have to be improved. Our current resolution is around 400 meV and with new electron gun with the trochoidal electron monochromator we will improve the resolution below 100 meV. With such resolution we will better separate vibrational states and be able to see some higher rotational states.

Although our research is based on the vibrational spectroscopy of the recombined molecules, we are also using the ion beam analytical methods for gaining additional information about the sample surfaces. For this purpose we are using tandem accelerator, located at our laboratory. Used methods are RBS for sample structure specification, ERDA, where we are using  $^7\text{Li}^{2+}$  ions (for H and D), and NRA, where  $^3\text{He}$  ions are used (for D). We are measuring hydrogen and deuterium concentration depth profiles in order to better understand adsorption of hydrogen on the surface, isotope exchange, temperature programmed desorption, etc [3].

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## **Ion channelling: strengths and limitations in the characterization of semiconductor materials**

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1) Ion channelling: strengths and limitations in the characterization of semiconductor materials

2) Ion channelling is a powerful technique to detect defects in crystalline materials and it is therefore a very suitable method for the characterization of semiconductor materials, normally requiring high-quality standards. However, the continuous trend towards Nanotechnology imposes certain limits to conventional ion beam techniques, including channelling. On the one hand, ion channelling is a collective phenomenon generated by the atomic strings of the crystal and, then, it is strongly reduced in low dimensionality structures (very thin films or nanostructures). On the other hand, the angular resolution of ion channelling is often compromised because of intrinsic limitations in epitaxial layers. In this work, we will show the strengths and limitations of ion channelling in semiconductor heterostructures. In many cases, ion channelling is a unique technique to detect growth defects with depth-resolution (e.g., phase separation) being an essential complement for other standard methods such as X-ray diffraction. However, in other cases (e.g., in the determination of strain), the appearance of steering effects in high quality interfaces can preclude an accurate analysis of the structural properties. Despite these latter difficulties, we will show that the accurate strain determination is possible by selecting appropriate experimental conditions and the use of appropriate simulation codes.



## **Forward elastic scattering as a tool to determine the D/H-ratio in geological samples**

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Knowledge of the isotopic composition of hydrogen is of interest in many fields e.g. geology and hydrology, as it yields information about the behavior and mass transport of hydrogen during the evolution of the Earth [1]. It is also of interest in astronomy as it has been discovered that the Deuterium/Hydrogen-ratio in different astronomical bodies varies [2].

A quantitative technique for determining the Deuterium/Hydrogen-ratio in geological samples is currently under development at the Lund Ion Beam Analysis Facility (LIBAF). The technique is based on forward elastic scattering where an incident deuterium ion knocks out a hydrogen nucleus or a deuterium nucleus inside the sample. The scattered deuterium ion and the target nucleus are detected in coincidence using an annular Double Sided Silicon Strip Detector (DSSSD), which provides both position information and energy information of the scattered and the recoiled particle [3]. Using this information it is possible to perform direct measurements of the Deuterium/Hydrogen-ratio [4]. A similar technique has been proven to work for hydrogen analysis in geological samples [5]. In this work we describe the technique and discuss its capabilities. We also present the first results on geological material.

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## MeV SIMS with a capillary microprobe

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MeV Secondary Ion Mass Spectroscopy (SIMS) is a new, emerging technique to analyse a sample surface for molecular information. Secondary ions produced by the bombardment of a surface with ions are analysed with a Time of Flight (ToF) spectrometer, similar to the well-known SIMS method with low energy projectiles. Interestingly, the usage of MeV ion projectiles leads to an increase in secondary ion yields [1, 2] as well as to reduced fragmentation of the secondary particles [1]. Additionally it was proven before [2, 3] that heavier projectiles provide even higher yields of molecular ions. These trends motivate extended research of MeV SIMS with heavier projectiles. A new experiment is currently in the planning phase to investigate this phenomenon with high energy heavy ion and molecular beams. The aim is to build up a unique MeV SIMS setup which will allow for the usage of projectiles as heavy as the C60 fullerene. A capillary microprobe will be included to enable microanalysis and imaging on the sample surface. Foregone studies at ETH Zurich have proven the applicability of a capillary microprobe for heavy ion microbeams, which will ensure a micron size projectile beam spot on the sample independent of projectile mass and energy.

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## **IN SITU ANALYSIS OF THE CARRIER LIFETIME IN SEMICONDUCTORS DURING IMPLANTATION OF MeV IONS**

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Modification of properties of materials and device structures as well as introduction of fast recombination centres using ion implantation is unavoidable accompanied with the creation of harmful defects. Therefore, *in situ* control of defect introduction and evolution in semiconductors during implantation of different ions as well as defect behavior after implantation are important for the development of new materials.

In this work, the carrier lifetime in situ variations during MeV ions irradiation generated by the tandem-type accelerator have been examined in semiconductor samples. In the case of the silicon wafer implantation by protons the nonlinear decrease of the carrier lifetime has been obtained within the ion projectile penetration depth, while the effective carrier decay lifetime in the bulk of a silicon wafer decreases slightly. The separation methodology of the surface and bulk recombination parameters is presented and discussed.

Keywords: ion implantation, in situ carrier lifetime monitoring.



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## **Overview of Ion Beam Analysis Applications at the Lebanese Accelerator**

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## **Microanalysis of Paint Layers in Polychrome Sculptures**

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## **Electronic sputtering resistance of a NbN/Si thin film during Heavy Ion ERD analysis by 26 MeV Cu<sup>7+</sup> ions**

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Heavy Ion ERDA is particularly suited for elemental depth profiling of thin film materials. A likely artefact when using this technique is the loss of thin film atoms due to electronic sputtering during measurement. This presentation reports on the analysis of a NbN/Si thin film produced by rf magnetron sputtering for applications in hard thin film coatings. The measurement was done using 26 MeV <sup>63</sup>Cu<sup>7+</sup> ions. Elemental areal densities at the beginning and at the end of the analysis run were compared to check for atomic erosion. The results do not show any measurable film thickness reduction and the N-Nb stoichiometry is retained up to  $3 \times 10^{14}$  at.cm<sup>-2</sup> (3 hours irradiation time). The only change observed was a reduction in the areal density of the oxygen surface contamination.