MODIFICATION AND ANALYSIS BY ION BEAMS AT MTF STU

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1. Introduction

It is about half a century that the ion accelerators have started to find application in materials science as analytic, testing and production tools. Selecting the most appropriate ion beam parameters (type of ion, energy, current, flux, fluence, beamsize, etc.), the ion–solid interaction can be adjusted, from weak or slightly perturbative (ideal for the nondestructive analysis [1]), to strong (able to modify substantially the material structure and properties).

Particle accelerators have proved to be competitive tool in this scientific/technological field and enabled to evolve the applied branches such as Ion Beam Analysis (IBA) and Ion Beam Modification of Material (IBMM). Even at STU, efforts have been made to engage in this global process from the outset of this development. [2]. However, the result of this endeavor corresponded to real material and other conditions at that time. But thanks to a new Ion Beam Centre (IBC) at MTF STU as a part of the CAMBO Trnava Scientific Park, it is possible to continued using IBA and IBMM in material research at STU at the latest technological level.

A summary of the IBC's first year of operation in Trnava, including selected examples of IBA and IBMM experiments, is summarized.

2. Experimental

The 6 MV Tandetron tandem ion accelerator and the 500 kV ion implanter provide ion beams in a wide range of energies - from 40 kV to tens MeV, beam currents – from sub nA to mA, virtually all elements can be accelerated from hydrogen to bismuth. The accelerator system with the 90° mass separating magnets in the injection section provide isotopically pure beams. Maximum achievable energy is, for example, 12 MeV for proton, 18 MeV for He^{2+} and 54 MeV for Au^{8+} beams.

Pure and carbon-free vacuum ensure oil-free pumping systems, based on scroll and turbomolecular vacuum pumps used on Tandetron and implanter systems. Secure, reliable and efficient operation of accelerators as well of analytical and implantation end stations are covered by their central computer control systems. Since operating parameters, settings and accelerator states are stored in the computer's memory, the repeating of the beam parameters already used beam parameters is considerable simplified.

The implantation end stations at both accelerators are available for the synthesis and modification of materials. It is possible to treat the samples with a diameter of up to 4 inches at Tandetron, up to 8 inches at implanter. Implantation at both accelerators can be performed at a controlled sample temperature ranging from LN2 temperature to 800 °C.

The following analytical methods are available for material analysis:

- RBS - Rutherford Backscattering Spectrometry – typical application is depth profiling of heavy elements at light substrate, depth resolution few nm,
- RBS/C RBS in channeling or blocking mode – provide information about crystalline material, .e.g. the amount and depth distribution of lattice disorder; location of the impurity atoms at the lattice site, the damage / defects of the crystal, etc.,
- ERDA - Elastic Recoil Detection Analysis - is based on the detection of energy and velocity / mass of atoms that are knocked out from the sample by incoming heavy ions. The currently installed ERDA system is an extension of the RBS measurement system and is limited to hydrogen depth concentration profiling.
- PIXE - Particle (Proton) Induced X-ray Emission – the accelerated particles are used to knock electrons out of occupied energy levels and to produce X-ray emission. Specific energy peaks identify the sample elements at the sensitivity of ppm, as the K. L, and M X-rays are a fingerprint of each element [3].
- NRA - Nuclear Reaction Analyses - products of ion beam induced nuclear reactions are detected. The concentration depth profiles of light isotopes (typically e.g. H, D, Li, B, C, O, and F) in complex matrices can be measured with the a sensitivity of 10 ppm.
- PIGE - Particle Induced Gamma ray Emission – is a special case of the NRA when prompt gamma rays are detected, which are emitted from the nuclei's that are excited after the nuclear reaction induced by the charged particle.
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The Ion Beam Center was presented in more detail elsewhere [4].

Tab. 1. *Synopsis of the IBA techniques, their detection capability and the main applications [1].*

Main application		Relevant Techniques	Detection Capability
Material science	**Ion Beam Analysis - IBA**	Rutherford Backscattering Spectrometry (RBS)	Z > 1
Archeometry and Cultural Heritages		Elastic Recoil Detection Analysis (ERDA)	Z < 17 (typically including H)
Earth and Environmental Science		Particle Induced X-ray Emission (PIXE)	Z > 11
Biological Sciences		Nuclear Reaction Analyses (NRA)	Z < 17 (often for C, N, O and isotopes)
Nuclear Safety and Radioprotection		Particle Induced Gamma ray Emission (PIGE)	Z < 17 (Li, B, F, Na, Mg, Al, Si)
Fundamental Nuclear and Atomic Physics		Scanning Transmission Ion Microscopy (STIM)	Sample density
Forensic		Ionoluminescence (IL)	Defects and Sample Structure

3. Ion Beam Modification of Materials

The first year operation summary of the IBMM activity on the facilities is as follows:

500 kV implanter: elements H, B, N, P, Ne, Ar, Cu, Ga, Fe, Co, W, including 57Fe isotope have been accelerated from natural elemental abundance sources in the energy range from 30 keV to 400 keV. The efficiency of the beam transport from the ion source to the sample depends on the ion optics of the system which is ion beam energy dependent. The maximum beam current from the ion source at the High Voltage Terminal was 4 mA (Ar, N), while a maximum 0.5 mA (Co) and at least 1.6 μA (W, Fe) beam currents were achieved at the implantation chamber. Applied implantation doses varied from $1x10^{16}$ at/cm^2 to $8x10^{17}$ at/cm^2 (Co).

6 MV Tandetron: elements H, He, B, C, Si, Cu, Ni, W and Au have been accelerated from natural elemental abundance sources in the energy range from 480 keV (H) to 54 MeV (Au). The maximum beam current of 10 μA (H, Si) and 13 μA (Cu) was at the implantation end-station. During 25 MeV Cu implantation, a 325 Watt beam power achieved. The applied implantation fluences varied from very low $1x10^{6}$ at/cm^2 (54 MeV Au) to $3x10^{16}$ at/cm^2. Implantation of 30 MeV Cu was performed at an elevated temperature of 700 °C.

4. Ion Beam Analysis

RBS: The majority of RBS analyses were performed with the standard RBS He beam energy range from 600 keV to 2 MeV, while for thick layer characterization up to 10 MeV beam energy was used. The thin layers with an equivalent thickness of less than 1 nm to the thickness of 12 μm were characterized. Examples of thin layers of e.g. Au, Al, Co, C on Si substrate, MoS, TiC, TiN, etc., including MoS interdiffusion profiles will be presented.
To increase the sensitivity of measurement of C, N and O, the non-Rutherford back scattering regime was used. The improved sensitivity for same elements by one to two orders of magnitude was achieved, comparing to the standard RBS.
RBS/C: The crystal sample alignment to the specific crystallographic axes and the channeling spectra measurement is demanding task. This requires typically to measure hundreds of RBS spectra. The first step, the automation of the measurement has been done. The automation of the basic evaluation of channeling spectra measurements will be followed.

PIXE: There are activities ongoing to move from quantitative to quantitative PIXE measurements, without using special reference samples. As it is a complex problem, the detailed and comprehensive PIXE X-ray detection system characterization is required. The PIXE setup was modified so that the aerosol filters could be measured.

NRA: Analysis using (p,α) and (p,α) nuclear reactions, e.g. $^{31}P(\alpha,p)^{34}S$, $^{10}B(\alpha,p)^{13}C$, can be performed at the standard RBS/ERDA experimental setup. As an example the boron profile applying $^{11}B(p,\alpha)^{8}Be$ reaction will be presented.
The analysis using nuclear reactions (p,γ) and (p,γ) required an extension of the IBA experimental chamber. The IBA experimental chamber was extended by gamma NRA setup with the LaBr scintillation detector (see Fig. 1). A small single sample chamber for NRA is electrically insulated from other parts of the beamline. This allows energy sweeping by variation of the potential of the NRA chamber without changing the high voltage of the

accelerator. This is particularly advantageous when using resonance nuclear reactions for analysis. But in this case it is not possible to monitor beam dose by measuring the charge at

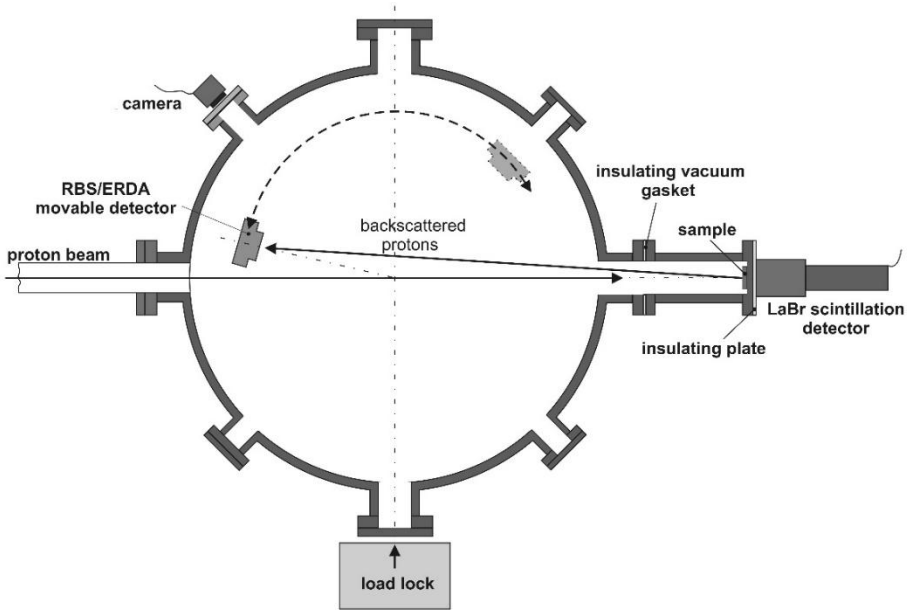

Fig.1: *Extension of IBA chamber for NRA setup. RBS-ERDA movable detector is used for beam fluence monitoring through RBS measurement of analysing beam..*

the sample holder. The movable RBS detector was used collect the backscattered primary beam particles as a dose monitor. This setup was used also for energy calibration of the Tandetron system utilizing selected resonance nuclear reactions.

ERDA: The hydrogen depth profiles have been measure by ERDA, the example of measured spectra will be presented.

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