ION BEAM LABORATORY FOR MATERIALS RESEARCH AT ADVANCED TECHNOLOGIES RESEARCH INSTITUTE (ATRI)

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Abstract

The new Ion Beam Laboratory (IBL) for materials research arises within the Centre of Material Research of Advance Technologies Research Institute (ATRI) of the Faculty of Materials Science and Technology (MTF) in Trnava, Slovak University of Technology (STU) Bratislava. The Centre is expected to be a scientific and experimental base for ion beam and plasma technologies application in the physical and material engineering and nanotechnology research. Its facility includes the ion beam and plasma cutting-edge technologies for synthesis, modification and analysis of surface, subsurface, and thin layers of solids. Description of the main IBL key equipment is given, consisting of the 6 MV Tandetron tandem ion accelerator and 500 kV ion implanter. The whole range of technologies and methods for Ion Beam Analysis (IBA) and for Ion Beam Modification of Materials (IBMM) will be put into trial operation during 2015. These are all outlined in this paper, together with their main experimental characteristics and typical research and industrial applications. Presented IBA analytical methods are considered to be nondestructive. They can be used for example for determination of depth distribution of elemental composition of solid state samples from hydrogen to uranium. The depth of analysis and treatment covers range from the surface up to hundreds of microns. Usability for change in the composition and/or structure of materials is summarized. An example of Ion beams in nuclear material research for Generation IV nuclear fission reactors are presented.

Key words

ion beam analysis, IBA, RBS, channeling, PIXE, ERDA, NRA, ion beam modification of materials, ion implantation, ion mixing

1. INTRODUCTION

The new Centre for Materials Research is currently in the final stage of the construction within the Advance Technologies Research Institute (ATRI) at Faculty of Material Science and Technology (MTF) in Trnava of Slovak University of Technology (STU) in Bratislava. The Centre is supposed to be a scientific research base for the application of ion beam and plasma technologies in the physical and material engineering and nanotechnology. It is equipped with ion beam and plasma high technologies for synthesis, modification and analysis of surface, subsurface and thin layers of solids.

From the beginning of its activities, the intention of the Centre is to start the scientific cooperation at the regional and international levels, and gradually be ranked among international network of research facilities and become a partner of the recognized research centers in the field. We expect that the internationally integrated cutting-edge research will be the basis for applied research and transfer of new technologies to the industry, and, via close cooperation with particular regional industry, will support implementation of innovation in practice. The first step to achieve these goals is that we have received EU funding of the initial phase of the project " Slovak Centre of Excellence in Ion Beam and Plasma Technologies for Materials Engineering and Nanotechnology " - SlovakION within the call of Teaming - Horizon 2020 Widespread 2014 (1).

Centre for Materials Research includes:
- Ion Beam Laboratory,
- Laboratory of Plasma Technology.

However, in this article, we describe only the installation and research core competency of the Ion Beam Laboratory. The Laboratory of Plasma Technology offers equipment for plasma immersion ion implantation and for sputtering - a physical vapor deposition (PVD) as well, for the deposition of thin film layers/coatings. Laboratory of Plasma Technology is described elsewhere.

Instrumentations available on both Ion Beam Analysis and Ion Beam Modification of Materials, their main characteristics and parameters as well as advantages and applicability in research and industry are briefly presented in the following section.

2. ION BEAM LABORATORY

The Ion Beam Laboratory activities will be dedicated mostly to the ion beam based synthesis, modification and analysis of materials. The ion beams available at IBL cover a wide energy range from 20 keV to 50 MeV. The key devices of the Laboratory are two linear accelerators:

- 6 MV Tandetron - tandem ion accelerator;
    - the working range of the accelerating voltage is from 300 kV to 6 MV,
    - almost all elements can be accelerated, i.e. from H- to the Au-, except the inert gases,
    - the maximum achievable energy is selective, depending on the charge state of accelerated ions; for example it is 12 MeV for hydrogen, 18 MeV for helium and about 50 MeV for multiply ionized heavy elements like gold.

- 500 kV ion implanter;
    - the working range of the implanter is from 20 kV to 500 kV,
    - almost all elements can be accelerated, i.e. from H+ to the Bi+.

The depth range of ion treatment is from the sample surface to a depth of hundreds of microns. The transport of ion beams and the experimental work with samples are performed predominantly at high vacuum. In special cases, also external ion beam can be accomplished, for on air experiments on e.g. large objects.

The 6 MeV Tandetron (Fig. 1) is equipped with two end stations, one dedicated to Ion Beam Analysis and the other one to Ion Beam Modification of Materials – mostly high energy ion implantation. The system is ready for extension and installation of additional beam lines.

The 500 kV implanter is primarily designed for ion implantation at the high and low temperature regimes.

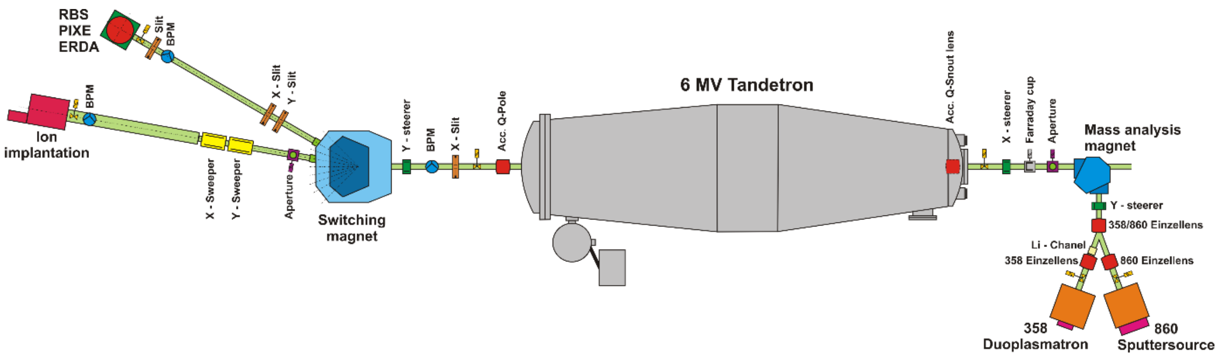

***Fig. 1** System of 6 MV tandem ion accelerator with ion sources (on the right), accelerator pressure vessel (gray), deflecting magnets (blue) and Ion Beam Analysis and ion implantation experimental chambers (see left side)*

<u>IBA – Ion Beam Analysis</u>

IBA analytical methods are considered to be nondestructive. It is an effective tool for investigation of the elemental composition, to some extent also of the structure of the surface layers of materials. The typical diameter of analyzing ion beam during IBA is from 2 mm to 5 mm, a typical beam current is from few to tens of nA.

The interaction of the incident ions with atoms of the sample matter leads to several physical phenomena. Depending on arrangement and geometry of experiment and on what product of interaction is detected, we can utilize different accelerated ion-beam-based analytical methods.

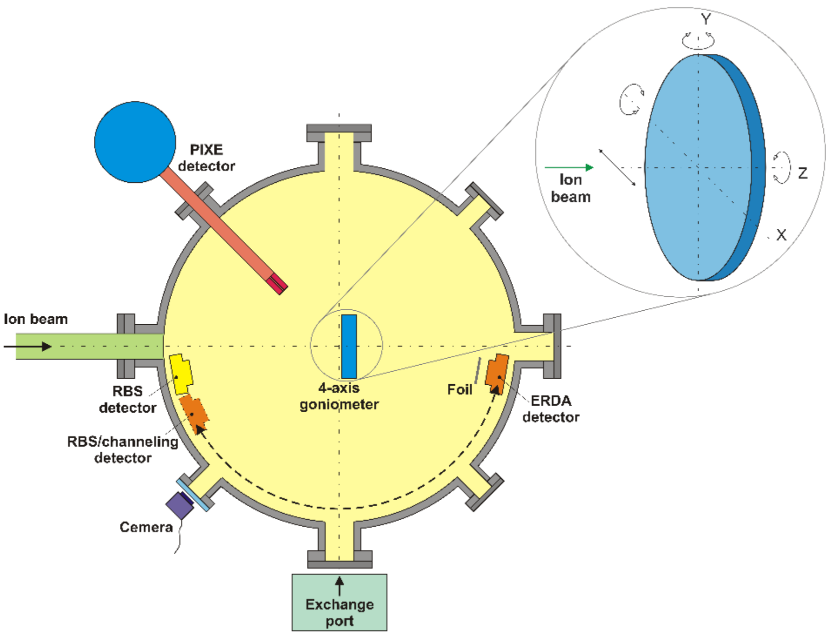

***Fig. 2** IBA experimental chamber equipped with 4-axis goniometer for sample alignment and with detectors for RBS, channeling, ERDA and PIXE analytical measurements*

Consequently, depending on which component of the result of interaction emitted from the sample is detected, (2) we consider the following analytical methods (Fig. 2):

- RBS - Rutherford Backscattering Spectrometry – the energy spectra of backscattered ions are detected,

- RBS/C or /channeling / blocking - the angular dependence of the RBS spectrum with respect to the crystallographic axes and planes of crystalline sample is measured,
- ERDA - Elastic Recoil Detection Analysis - recoil energy and velocity/mass is of our interest,
- PIXE - Particle (Proton) Induced X-ray Emission – detection of the X-ray spectrum,
- PIGE - Particle Induced Gamma ray Emission – detection of gamma radiation,
- NRA - Nuclear Reaction Analyses - detection of products of the induced nuclear reactions.

The typical scope of IBA analysis is from the surface to the depth of micrometer. It is possible to examine thin layers down to nanoscale. The analytical sensitivity of the dopant elements and impurities reaches the level of ppm. More specific are the possibilities of individual IBA methods described below (3), (4).

3. ION BEAM ANALYSIS

Rutherford Backscattering Spectrometry

RBS is elastic scattering of light ions like protons, He, Li, etc. Commonly is the sample bombarded by helium in energy range 1-3 MeV. The energy distribution and yield of the backscattered ions at a given angle is measured. Since the backscattering cross section for each element is known, it is possible to obtain from the RBS spectrum a quantitative compositional depth profile (see Fig. 3).

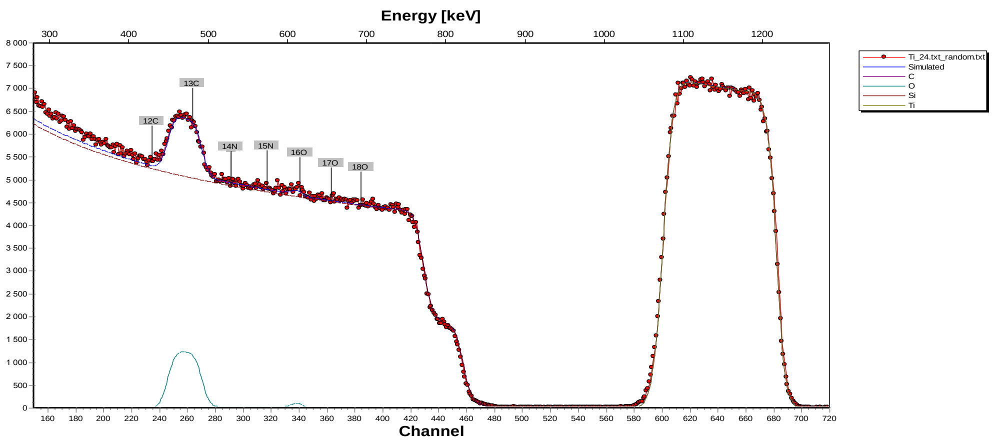

Fig. 3 *RBS energy spectra of 1100 at/cm^2 Ti layer followed by 660 at/cm^2 SiO_2 layer on Si substrate. SiO_2 as well as surface oxygen signals are indicated.*

RBS is highly sensitive to heavy elements in a light substrate. It is a quantitative method without need of using reference standards samples. It is possible to determine film density when thickness is known.
Heavy ions – RBS enables to get the better mass and depth resolution.

The advantages of RBS:

- provides composition and depth information, e.g. concentration depth profiles of the elements of the sample,
- thickness of thin films, depth composition interface, diffusion profile, etc.,

- depth resolution down to few nm,
- the technique is very useful in the analysis of thin films on thicknesses ranging from microns down to a fraction of a monolayer,
- is very sensitive to heavy elements and suitable for analysis of elements heavier than oxygen.

RBS is less sensitive to light elements; however this makes it an ideal complement to PIXE.

Non-Rutherford scattering - improves sensitivity of light elements measurement using resonance cross sections, like 2.4 MeV resonance H^+ or 3.04 MeV He^+ or others, for C, N, O or Si, respectively O measurement. At high energies the cross-sections deviate from Rutherford due to the influence of the nuclear force (5). As there is no general theory for the calculation of non-Rutherford scattering cross-sections, experimental values usually have to be used. The discrepancy between different measurements may be up to 30%, so some care has to be applied when cross-section data are used in data evaluation.

RBS/C channeling / blocking mode:
Ion channeling takes advantage of the material properties of single crystals. When ion beam is properly aligned with the crystalline axis of a single crystal, the backscattering signal drops dramatically.
Ion channeling provides the following information:

- amount and depth distribution of lattice disorder,
- location of impurity atoms in lattice site,
- crystal damage/defect profiling, i.e. determination of the level of amorphisation, of the thickness of amorphous or defective epitaxial surface layers.

ERDA – Elastic Recoil Detection Analysis

It is based on the detection of atoms which are knocked out from the sample by incoming heavy ions. Currently installed ERDA system is extension of RBS measuring system and is limited to hydrogen depth concentration profiles measurements.

After upgrading of the laboratory measurement end station by the Time-of-Flight ERDA (TOF-ERDA), the range of analyzing elements will be extended to heavier ones up to uranium. The advantages of this method are:

- high sensitivity to light elements in heavy substrates,
- parallel depth profile measurement of all detected elements.

PIXE – Particle/proton induced X-ray emission

X-ray emission can be induced by light ions, but in the most common case, the proton beam with energy from 2 to 3 MeV is used. The accelerated particles are used to knock electrons out of occupied energy levels and to produce emission of characteristic X-rays lines. The specific energies peaks identify the elements of the sample, as the K. L, and M X-rays are a fingerprint of each element (4).

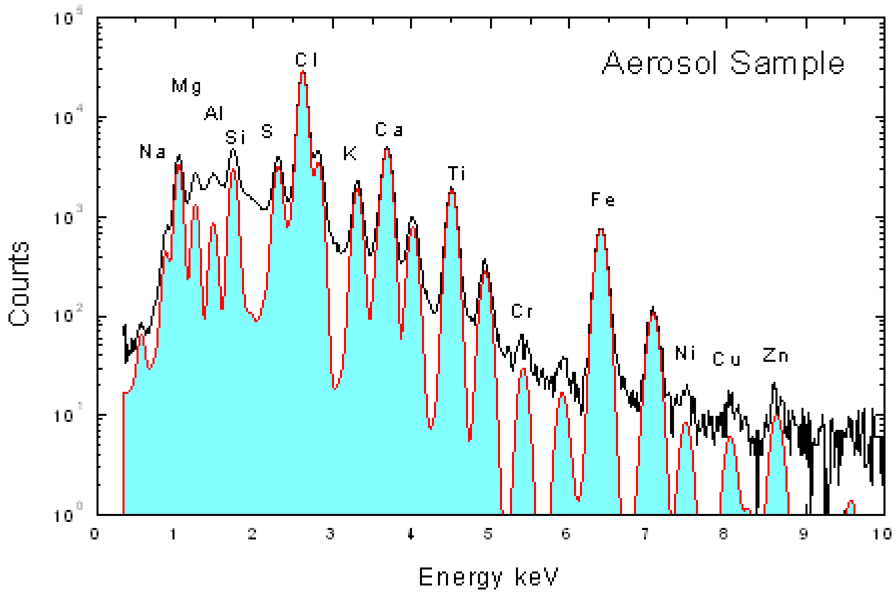

Fig. 4 *Example of PIXE x-ray spectrum - atmospheric aerosols composition measurement*

PIXE is capable to recognize each element heavier than C, it is suitable for trace element analysis with sensitivity to ppm, but gives no depth information, only general information about concentration.

The typical application of PIXE is monitoring of the composition or contamination of materials, as well of atmospheric aerosols (6), liquids, see sediments, analysis of biology samples, but also elemental determination of geological samples for earth science, as well as works of art like paintings and other art objects and archaeological findings, etc.

<u>NRA - Nuclear Reaction Analysis</u>

The NRA measuring end station is not available at IBL from the first stage of operation and has to be additionally supplemented.

NRA uses inelastic scattering and nuclear reactions. The resonant or non-resonant nuclear reaction can be used. NRA utilizes often resonant nuclear reactions induced due to the interaction of the accelerated particle with the specific target atoms of the sample. (7) The emitted radiation is characteristic for given reaction. Commonly the gamma radiation is detected, but depending on the products of the specific nuclear reaction, also emitted nuclear fragments can be detected. The elemental analysis and concentration depth profiles of low-Z atoms can be measured with the sensitivity of 10 ppm. NRA is very suitable for measurements of low levels of B, C, N, and O in thin films.

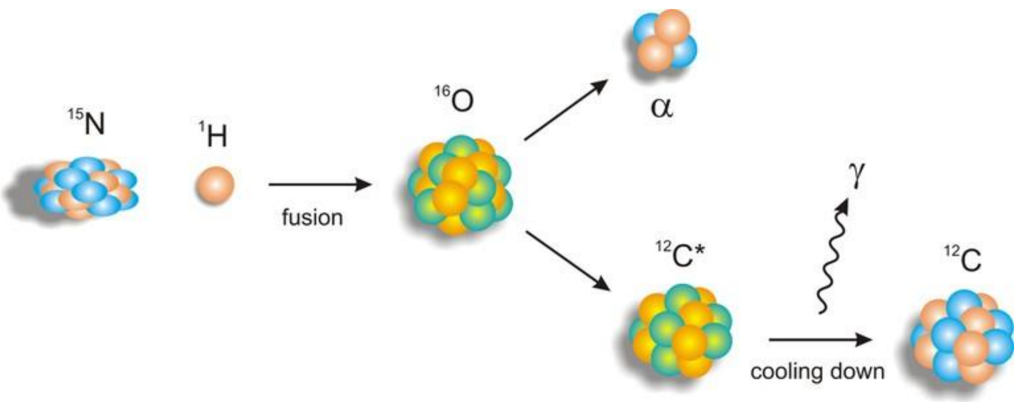

Fig. 5 *Illustration of the nuclear reaction for hydrogen analysis with 15N ions (8)*

Fig. 6 *Principle of hydrogen profiling by NRA with the 15N ions (8)*

For example, the resonant NRA based on the reaction 1H(15N,αγ)12C is the standard method for hydrogen detection with high depth resolution. Only when the energy of the nitrogen ions equals to the resonant energy E_{RES}, nuclear reaction occurs and emits γ-rays. By the progressive increase of 15N energy, one shifts the resonance reaction into a certain depth of the target. In this way, the hydrogen depth profile can be determined (8).

4. ION BEAM MODIFICATION of MATERIALS

In general, the ion beam synthesis and modification of materials can be carried out by:

- ion implantation
- ion beam mixing.

Ion Implantation

Ion implantation is a method of incorporating selected impurity atoms in the form of accelerated ions into a base material (substrate doping), or generating defects in (e.g. semiconductor) material (defect engineering, lifetime engineering). In this way, the surface properties of the base material change, creating new phases and alloys, far away from the thermal equilibrium. By changing the energy of the implanted ions, the implantation depth can be tuned. By the combination of implantation energies and doses, the desired depth concentration profile of the implanted element can be reached.

Ion implantation at IBL will cover the energy range from 20 keV to 50 MeV. The maximum diameter of the implanted substrate is 20 cm for 500 kV implanter and 10 cm for 6 MV Tandetron. The implantation can be carried out at the cooled or heated substrate.

Ion beam mixing
It is stirring of atoms and alloying at the interface of two different materials during irradiation by ions. This method is mainly used for connecting a non-equilibrium, metastable alloys and intermetallic compounds.

Applications of ion implantation and ion mixing.
The main objective is to change the properties of surface and subsurface areas of materials. Ion implantation is one of the key technologies of the semiconductor industry, but it can be used for basic and applied research to modify surface-sensitive properties like adhesion, wear, roughness, hardness, abrasion resistance, corrosion of metal or other materials, biocompatibility, blood-compatibility, etc. Another area of utilization is also targeted modification of electrical, magnetic, optical and other physical or chemical properties of the surface layers. Recent topical application area is the formation of nanostructures, nanoscale porosity and modification of nanostructures properties.

5. ION BEAMS IN NUCLEAR MATERIAL RESEARCH

Steel
The beginning of the nuclear reactor steel material radiation resistance studies by ion beam irradiation started already 50 years ago. The following examples present the continuing progress in ion beam applications for the new materials for nuclear industry development. The new ODS steels are developed as candidate materials for some of the future nuclear fission reactors known as Generation IV, as well as for potential fusion reactors. Ion beams were used for radiation induced swelling and hardening studies (9). For example, Fe–Cr steels as promising candidates for blanket components in fusion reactors and for cladding materials in fission reactors were examined by He irradiation (10). Swelling distribution was investigated by MeV Cr implantation of ferritic/martensitic steels (11). Nanostructure influence by iron ions irradiation was performed, during which nanosized clusters containing atoms of V, Y, O, and N (12) were formed within ODS steel. The Cr ODS steel was irradiated by protons to determine the nanocluster stability at low dose Ferritic–martensitic (F/M) alloys (13).

Silicon carbide
Because of its mechanical stability under high-energy neutron irradiation and high temperature, silicon carbide (SiC) has a great potential as a structural material in advanced nuclear energy systems. For fission reactors, SiC is proposed as the fuel component to retain fission products. Cubic silicon carbide (3C-SiC) is a promising structural and cladding material, and is used in fusion reactors and advanced fission reactors due to its attractive mechanical and thermal properties. The 6 MeV Si irradiation was used to induce microstructural evolution and volume swelling of SiC at 400–1350 ^{o}C (14). Irradiation-induced microstructural change in helium-implanted single crystal and nano-engineered SiC were studied by (15). The following irradiation with 9 MeV Au3+ ions at 700 ^{o}C was performed.

The outline of ATRI Ion Beam Laboratory new experimental capabilities for advanced materials research was given. ATRI offers a chance for increasing the support for industrial innovation enhancement.

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