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Characterisation of PIXE facilities at the 3 MV VDG Accelerator Laboratory of Atomic Energy Centre Dhaka

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Abstract

A new beam line and ion beam analysis (IBA) facility has been designed and constructed at the left beam line of the 3 MV Van de Graaff accelerator facility of BAEC. The goal is to perform standard ion beam techniques such as Proton Induced X-ray Emission (PIXE), Proton Induced Gamma Emission (PIGE), Rutherford Back Scattering (RBS) spectrometry, Nuclear Reaction Analysis (NRA), etc. with a millimeter size beam. A full calibration of the PIXE set-up in this chamber has been done with different thin and thick standards following the H-value method devised by GUPIX, where H is the solid angle plus the correction factor needed for the set-up to produce accurate concentration value. The energy dependant H-values were stored in the GUPIX library, to be used for PIXE analysis. The PIGE and RBS set-up has been also calibrated with known gamma and alpha sources and are ready for experiment.

1. INTRODUCTION

Accelerator Facilities Division of BAEC has been operating a 3 MV Van de Graaff Accelerator, which was established in the Atomic Energy Centre, Dhaka in 1965. There are two beam lines at the end of the Accelerator. The beam line to the right at 25° with respect to the ion beam direction is extensively used for PIXE and PIGE analysis. There is a small experimental chamber with 8 samples holding capacity and an external beam facility. The left beam line at 25° is seldom used for RBS analysis. This beam line with its associated equipments is very old and difficult to use. Thus to achieve better control over the beam and to perform automatic and quality experiment it was decided to modernize the left beam line and its associated facilities. This beam line is designed and constructed to produce a millimetre sized beam, for standard Ion Beam Analysis techniques such as PIXE, PIGE, RBS, NRA, etc. The main objective was to be able to run a large number of samples automatically with minimum user intervention and to perform in situ analysis of the PIXE, PIGE & RBS spectra. For a large number of similar commercial samples this would be very useful. Characterisation of PIXE facilities at VDG Accelerator Laboratory of AECD will be described here.

2. TECHNIQUES AROUND MEV ACCELERATORS

When high-energy charged particles hit a target many processes may occur, e.g. emission of electrons, X-rays, γ-rays and alteration of the nuclear structure of the sample. The ion may scatter in the backward or the forward direction, may scatter particles from the sample, produce luminescence in the sample or simply pass through the sample by loosing part of its energy. The different techniques are named, according to the kind of processes involved. In the following paragraphs PIXE technique commonly used around the VDG Accelerator Laboratory of Atomic Energy Centre Dhaka is briefly discussed.

2.1 PIXE

Particle Induced X-ray Emission spectroscopy is one of the most common and widely used analytical techniques at MeV energy accelerators. Sven Johansson and co-workers at the Department of Nuclear Physics, Lund University first developed this technique in the early

1970s [1]. When charged particles with sufficient energy hit a sample, a vacancy in the inner shells of an atom may be created. The probability of creating a vacancy is higher when the velocity of the incoming ions matches the velocity of the inner shell electrons. For MeV ions this probability (cross-section) for ejecting inner shell electrons is quite high (of the order of barns)[1]. Such a vacancy can be filled in a number of ways and one of the processes may emit X-rays with energy characteristic of that particular atomic number. In the PIXE-technique these characteristic X-rays are detected using semiconductor detectors. An energy dispersive analysis of the detector signals can reveal the identity of different elements present in the sample and, more importantly, by measuring the charge, i.e. the number of incoming particles, the concentrations of the elements can be accurately quantified. PIXE is a truly multi-elemental technique and can identify elements from Na (this lower limit is set by inefficiency of the X-ray detectors below Na X-ray) and up through the rest of the periodic table. Applications of this technique are wide-ranging and diverse [1]. In the Accelerator Facilities Laboratory of Atomic Energy Centre Dhaka, PIXE is routinely being used for the analysis of biological, geological and environmental samples.

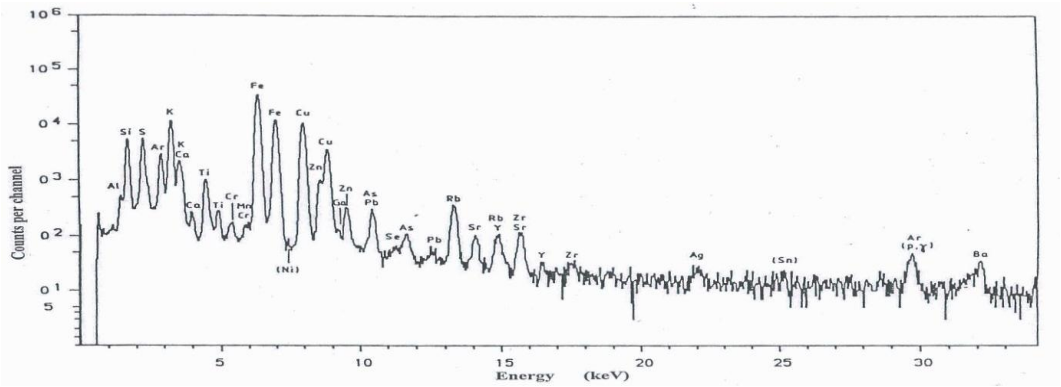

Figure: 1 PIXE spectrum of a soil sample.

2.2 Calibration of the set-up for PIXE analysis

The main goal in constructing this beam line was to perform conventional PIXE, PIGE & RBS analysis on large number of samples. To analyse complex PIXE spectra, the GUPIX software [6] with DAN32 [6] interface software has been used. GUPIX uses all the updated and best databases available (cross-sections, fluorescence and Coster-Kronig probabilities, stopping powers and attenuation coefficients) and has many useful options, such as matrix iteration, layer thickness iteration, the possibility to add invisible elements to the fit, batch-analysis of spectra, the possibility to include an energy-dependent calibration factor H (which is the product of the detector solid angle and any correction factor needed for the whole set-up), and to include energy-dependent, low energy tailing parameter descriptions of the X-ray detector in the database. These measures, if taken, greatly improve the accuracy of the quantification of the PIXE analysis. For the PIXE calibration process we followed mainly the H value method described in ref. [7], which is based upon the equation:

$$Y(Z,M) = Y_I(Z,M) \cdot Q \cdot C_Z \cdot T(Z) \cdot \varepsilon_Z \cdot H \qquad \text{.....(1)}$$

where,

Y(Z,M) is the measured X-ray yield computed by the fitting program.

Y_I(Z,M) is the theoretical X-ray yield per unit beam charge, per unit solid angle and per unit concentration computed from the GUPIX database, which includes the matrix correction and secondary fluorescence for the thick targets.

Q is the measured beam charge or some value proportional to the charge.

C_z is the concentration of the element quoted by the manufacturer or measured by some other method.

T (Z) is the fractional transmission of X-rays through absorbers.

ε_z is the intrinsic detector efficiency.

H is the product of the detector solid angle and any correction factor for the charge measurement.

In our case H also includes the effects of possible inaccuracy in the description of the detector solid angle, the thickness of the detector crystal, the thickness of the absorber layer, etc. It also includes any imperfection in the various databases used in GUPIX [5]. The energy-dependent H-values and low energy tailing parameters are detector-specific [7]. To evaluate the H-values a set of thin MicroMatter standards [8] has been irradiated. First by using the actual solid angle as the H value in the GUPIX the concentration values were calculated then the absorber thickness were adjusted to get the closest concentration value to the manufacturer quoted value, especially for those elements whose X-rays were sensitive to the absorbers of that particular thickness. Then H values were calculated by comparing the calculated concentration values to the manufacturer quoted concentration values multiplied by the detector solid angle [7]. The H values were then plotted as function of X-ray energy, which is a scattered curve as function of X-ray energy and fitted by a suitable trend line to obtain a equation. Then this equation is used to generate a smooth set of H values as function of X-ray energy, which is stored inside the GUPIX library for further PIXE analysis of unknown samples.

2.3 Charge measurement

Accurate charge measurement is very important for the absolute quantification of the sample [1]. The most important factors for charge measurements are: i) loss of secondary electrons, ii) loss of scattered particles, iii) charge build-up in insulating samples. Figure 2 shows the layout of the charge measurement set-up in the new IBA chamber at the left beamline. For thin samples, all the beam passes through the sample and the charge measurement is done in the Faraday cup (on a graphite disc to minimise γ-ray background) at the end of the beamline after the chamber. For thick samples, charge is

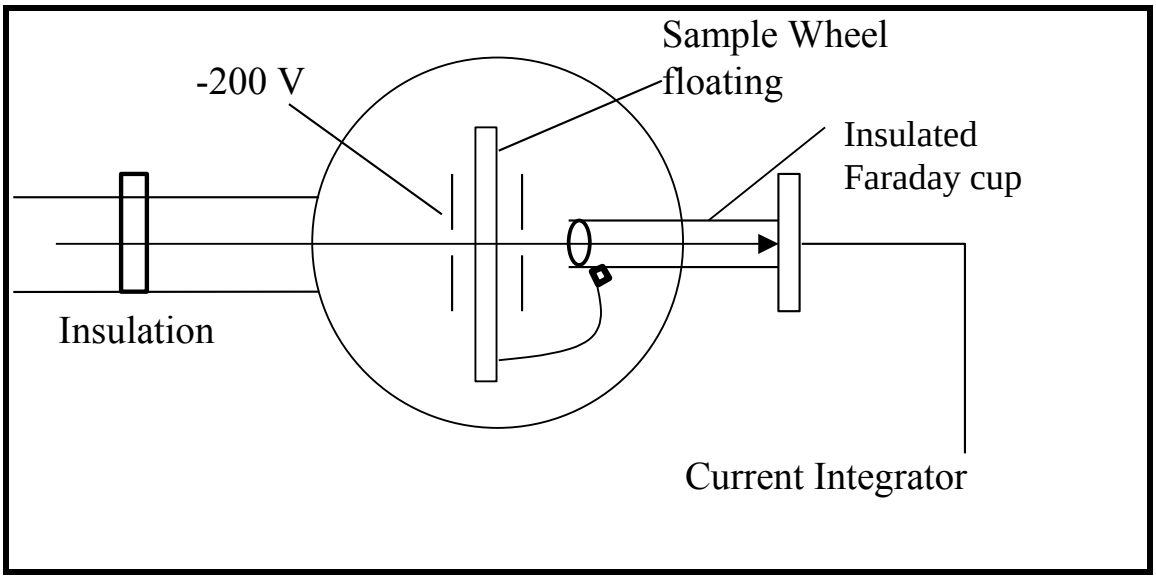

Figure: 2 Lay-out of the charge measurement system at the PIXE chamber.

collected from the sample holder wheel, which is electrically insulated from the chamber, via a flexi-cable connected to the faraday cup. The secondary electrons emitted from the front surface of the sample while bombarded by the beam are suppressed by a negative voltage of 200 V applied on a pair of circular metal ring close to the sample. Charge collections for the thin samples are rather straightforward and are working satisfactorily, which we see from the thin standard calibrations.

For charge build up in insulating samples there are several ways to handle it for example, to place an electron gun inside the chamber to spray electrons on the sample, to introduce a gas in the chamber to give a discharge path to the particles and, to coat the sample with a thin conducting film (normally carbon), to mix a conducting material (normally graphite) with the sample to give a discharge path to the particles, etc. In our facility we follow the last approach since it is better suited for our need.

3. ABSORBERS

It is important to use some absorber material [1, 4] in front of the X-ray detector for at least two reasons, firstly, to save the X-ray detector from radiation damage from the scattered high energy particles from the sample, secondly, to decrease the count rate in the X-ray detector, which is very important in order to decrease pulse pile-ups and the dead time in the data acquisition system. Normally, it is the low Z elements present in the sample that produce most of the counts compared to the tracer levels of high Z elements in the sample. In order to balance the counts of both low and high X-ray energy, it is very useful to have the so-called 'funny' filter, which consists of a suitable absorbing material (which can absorb almost all of the low energy X-rays and will absorb only a little or none of the high energy X-rays) with a small hole in it (which will let some fraction of the low energy X-rays to pass). Mylar of appropriate thickness is widely used as absorbing material, because it does not produce any unwanted secondary X-rays or γ-rays.

The Si(Li) X-ray detector has a built in Be window of thickness 25 micrometer. To put additional absorbers a special absorber holder has been designed, which is installed on the top nose of the X-ray detector. The following absorbers are now made and can be mounted one at a time in the holder.

170 μm Mylar

340 μm Mylar

50 μm Kapton

160 μm Mylar with a hole of 1 mm dia.

11.2 μm Aluminium with a hole of 1 mm dia.

4. REGULAR CHECKING OF THE SYSTEM USING STANDARDS

A routine check of the whole system for PIXE analysis is formulated from the old PIXE set-up, i.e. irradiating a special, thick Al standard at the beginning of each set of runs. The procedure is as follows: the beam current must be adjusted so that the PIXE count rate is about 1500c/s. The thick Al standard is irradiated and the ratio of Al to Fe, the Al counts/nC and the Fe counts/nC, is evaluated and compared with nominal values, to be sure that both the beam energy and charge collection is in order. A logbook is maintained for this purpose at the Accelerator Laboratory. In addition, we also often check the calibration of the system with the MicroMatter thin standards.

5. AUTOMATIC SAMPLE RUN AND IN-SITU ANALYSIS

One of our main objectives with the development of the new broad-beam facility is to be able to run samples automatically and perform in-situ analysis of the PIXE spectra. For this purpose some C++ programming sub-routines would be written inside the interfacing software. In one sub-routine the user can programme the run sequence starting from any position. The system will then acquire data until a pre-set time or pre-set charge is reached and then the sample will be moved to the next position and so on, up to the pre-defined number of samples. In another sub-routine, the acquired data are saved in a special GUPIX file format with some necessary header information, in a pre-defined folder. The DAN32 windows interface software for GUPIX has the script option which can be run continuously connected to that folder, so that, as soon as a GUPIX file is written, it starts to work on it and prints out the results in a spreadsheet in an

additive mode. All the necessary information to run GUPIX for a PIXE analysis is written in each file-header and in the DAN32 database. The user can get a spreadsheet with the PIXE results, while the experiment is still running.

6. APPLICATIONS

After successful completion of the beam line many PIXE samples will be analysed using different beam sizes and currents. The majority of these are aerosol samples collected by the Aerosol Group at the Chemistry Division of AECD, which has a long tradition of aerosol-related, atmospheric research going back more than 10 years. The group engages in both fundamental and applied research that will provide a sound scientific basis for policy-making. The following aerosol characteristics and processes (and their relevant negative effects), are studied by the group, for which the PIXE set-up of the left beam-line has been used extensively:

Aerosol emissions from various anthropogenic sources such as traffic and biomass combustion (human health);

Aerosols in the upper troposphere (climate change);

Long-range transport of acidifying, toxic and health-threatening compounds (acidification, human health);

Source-receptor relationships at various spatial and temporal scales (acidification, human health);

The new beam line and ion beam analysis facility will be extensively used for routine PIXE analysis of the following samples:

Aerosol samples collected on polystyrene filter material.

Biological samples like human blood, milk, hair, nail, etc.

Fish, shrimps, etc.

Environmental samples like water residue, lake sediment, soil, etc.

Plant samples like chewing sticks.

Material analysis with PIXE and RBS like solar cell material, thin films, etc.

Another possible plan is the PIXE analysis of a large number of geogas samples. Geogas bubbles are formed in water-filled fractures of the bedrock and move upward, and carrying matters with them to the surface. These geogas samples can be collected on thin polystyrene membranes. The concentration of trace elements is very low, and therefore PIXE is an ideal technique due to its high sensitivity and its ability to analyse many element at once. The measured elemental composition can give valuable geological information, for instance, concerning ore deposits.

CONCLUSION

The PIXE setup is incorporated with new scattering chamber of the left beam port and the characterization has been done as a part of ion beam analysis facility development process of the Accelerator Laboratory of AECD, as demand for better analytical results are growing in many fields of research. The PIXE set-up has been calibrated and is now ready to analyse any kind of samples. The support from the authority for this work is greatly acknowledged.

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