A new beam line for characteristic X-ray experiments at the Pelletron accelerator, Instituto de Física, UNAM

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The design of a new beam line at the Pelletron accelerator laboratory (Instituto de Física, UNAM), for characteristic X-ray experiments induced by ion impact is presented. The description of the beam line components, scattering chamber, detectors, and associated electronics is included. Uses of the device include measurements of X-ray production cross sections by ion impact, other inner-shell processes, and ion beam analysis. Examples of results obtained in the new beam line are given, as well as future improvements aimed to perform more complex experiments.

Keywords: X-ray spectrometry; ion beam analysis; inner-shell processes

Se presenta el diseño de una nueva línea de haz dentro del laboratorio del acelerador Peletrón (Instituto de Física, UNAM), para experimentos de rayos X característicos inducidos por impacto de iones. Se incluye la descripción de las componentes de la línea de haz, la cámara de dispersión, los detectores empleados y su electrónica asociada. Los usos de la línea incluyen mediciones de secciones eficaces de producción de rayos X por impacto de iones, otros procesos de capas internas y análisis con haces iónicos. Se dan así mismo ejemplos de resultados obtenidos en la nueva línea de haz, al igual que futuras mejoras para la realización de experimentos más complejos.

Descriptores: Espectrometría de rayos X; análisis con haces iónicos; procesos de capas internas

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1. Introduction

The development of materials research, and in particular the surface of those materials, has lead in the latter decades to the implementation of analytical techniques powerful enough to properly characterize them. Among the aforementioned methods, those based on the interaction of ion beams with the materials have shown an increasing popularity, due to their high sensitivity, multielemental capabilities, and very often, non-destructiveness. Additionally, several of the techniques can be applied simultaneously, so it is possible to obtain information on contents of almost every element in a sample. The most widely spread methods are Rutherford backscattering spectrometry (RBS), particle induced X-ray emission (PIXE), nuclear reaction analysis (NRA), elastic recoil detection analysis (ERDA), and proton elastic scattering analysis (PESA) [1]. The main instrument for the application of these techniques is a particle accelerator.

The application of these methods is not limited to materials science, but it has also been extended to areas like art, archaeology, medicine, biology, geology, agriculture, and environmental sciences. In many of these fields, it is important to obtain information from a very large number of samples, due to the high variability in natural systems. This imposes a need for fast and reliable analyses, conditions fulfilled by the techniques based on particle accelerators.

The arrival of the National Electrostatics Corporation (NEC) 9SDH-2 Pelletron accelerator to the Instituto de Física, UNAM (IFUNAM), in 1995, not only opened the door to wider applications of the traditional ion beam techniques, which were already routinely used in the 5.5 MV and 0.7 MV Van de Graaff accelerators [2], but to other studies, like modification of surfaces by heavy ion bombardment, radiation detector response to heavy ions, or atomic processes induced by the interaction of ions with atoms. The latter phenomena, in the beam energy range provided by the Pelletron accelerator, is usually reflected in the emission of characteristic X-rays from the irradiated atom, as normally inner-shells are affected by the presence of the incoming ion.

The original setup of the Pelletron laboratory did not include a beam line suitable for X-ray experiments, and the possibility of initiating studies on X-ray emission induced by heavy ion impact demanded the construction of a new beam line dedicated to this kind of experiments.

Moreover, the sensitivity and reliability of the traditional techniques, such as PIXE, pushes in the same direction, because they can also be used for analysis in the already mentioned scientific fields, and even in commercial analyses.

The present work, thus, presents a description of the new beam line associated to the Pelletron accelerator, dedicated to X-ray experiments. The line design, scattering chamber, detectors, and electronics, are discussed, as well as a few representative results. Also, future developments are proposed.

2. Beam-line design

The Pelletron accelerator has the possibility of directing the beam towards either one of six directions, namely $\pm 15°$, $\pm 30°$, and $\pm 45°$, if the original beam direction is considered

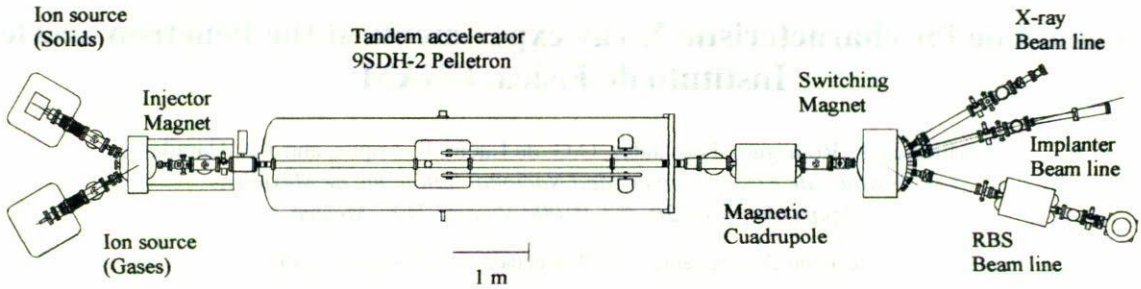


FIGURE 1. Top view of the Pelletron accelerator laboratory. The beam line for X-ray experiments is shown at the top right corner, following the $+30°$ direction of the switching magnet.

as the $0°$ line. This can be done by means of the switching magnet. Thus, the beam line for the X-ray experiments was installed in the $+30°$ direction, as shown in Fig. 1, which shows a general view of the laboratory. The other two existing beam lines are dedicated to ion implantation ($+15°$) and a multi-purpose chamber ($-15°$), mostly used for ion backscattering spectrometry analysis, including channeling in single crystals [1, 3]. As the particle accelerator must work in an ultra-high vacuum, the material selected for the beam line is mainly stainless steel. The vacuum seals are normally chosen as Conflat®, in order to maintain the high vacuum, although in several instances Viton® seals are used, for convenience in connections.

A detailed diagram of the beam line is displayed in Fig. 2. There, it is possible to see that the main components of the line are the following:

a) Electro-pneumatic gate valve with $4\frac{1}{2}$" Conflat® seals (MDC Vacuum, model LGV- 2500-P), isolating the line from the rest of the accelerator;

b) Welded bellow, connecting the valve with the line itself;

c) Stainless steel tubing segment, with a lateral nipple used to obtain a vacuum or venting the line in case maintenance procedures require so, through a roughing valve;

d) Tee, for the insertion of vacuum measuring devices, which are a thermocouple tube (for pressures between 10^{-1} torr and 10^{-3} torr) and a Bayard-Alpert nude gauge (for pressures lower than 10^{-4} torr); the measuring system was manufactured by Granville-Phillips (Boulder, CO, USA);

e) A double slit assembly (NEC model 2EA039041), to collimate the ion beam, improving the charge state and energy resolution of the ion beam;

f) Beam profile monitor, or BPM, (NEC model 2EA008242) to check the beam symmetry and cross section;

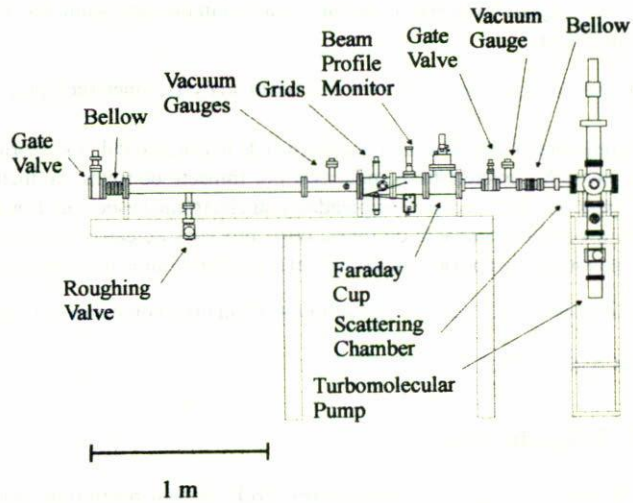


FIGURE 2. Diagram of the beam line, showing the major components. The dotted line represents the iron base of the beam line.

g) Faraday cup (NEC model 2EA059590), to measure beam current entering the line; the cup is capable of measuring beams with a power of up to 50 W;

h) Electro-pneumatic gate valve, using $2\frac{3}{4}$" Conflat® seals (MDC Vacuum model LGV- 1500-P), to separate vacuum in the beam line and the scattering chamber;

i) Tee with nude Bayard-Alpert vacuum gauge, to measure pressure at the chamber;

j) Welded bellow, to provide independent movement of the scattering chamber with regard to the line, so proper alignment can be carried out;

k) Scattering chamber, which is described below in more detail.

The whole line is supported on an iron base, with an independent support for the scattering chamber. Moreover, electric power is provided through a triphasic line, split into its three components, to energize the vacuum pumps, valves, and required nuclear electronics.

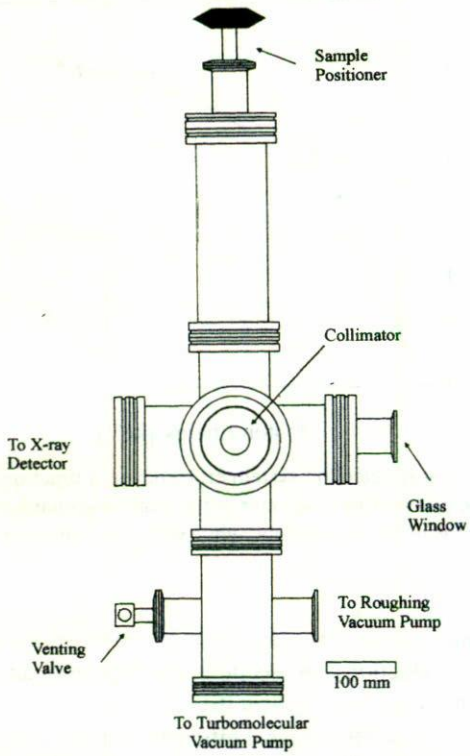


FIGURE 3. Front view of the scattering chamber, as "viewed" by the incoming ion beam, which enters through the collimator at the center of the chamber.

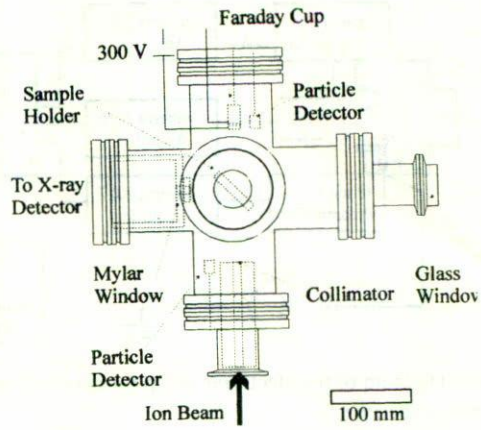


FIGURE 4. Top view of the scattering chamber, showing the ion beam incoming direction and, in dotted lines, the various components inside the chamber.

3. Scattering chamber

The scattering chamber is based on a six-way stainless-steel cross, with ISO100 vacuum flanges at each end (Figs. 3 and 4). This kind of connectors was chosen due to the frequent need of installing different components in the chamber, or opening for sample changing.

Figure 3 displays a lateral view of the chamber, to show the sample positioner, on the top of the chamber, and a turbomolecular pump on the bottom (Alcatel model 5101-TMP), separated from the chamber with a manual gate valve (MDC Vacuum model LGV-4500). The pump has a pumping speed of 100 L/s for N_2. The turbomolecular pump is supported by an Alcatel model CE-2005 mechanical pump (6.5 m^3/h). An assembly of valves and tees is used to obtain a by-pass for fast vacuum roughing after changing samples. Typical operating pressures at the chamber are 10^{-5} torr to 10^{-6} torr.

Figure 4 presents a top view of the chamber. The beam collimator is located in the chamber mouth closest to the line, where also a feedthrough for a particle detector is installed; at the opposite end, there is a Faraday cup for measuring beam current, including electrical feedthroughs for connecting a secondary electron suppressor in the Faraday cup, and a second particle detector. All these electrical feedthroughs use BNC type connectors in the exterior side. The Faraday cup uses a 300 V dry cell for secondary electron suppression. On the lateral mouths, there is a glass window, to observe sample position inside the chamber, and a window for the X-ray detector. This window is based on an aluminum cylinder inserted in the chamber, to increase the solid angle of the X-ray detector towards the sample. In this cylinder, the chamber is isolated from atmosphere with a 12 μm thick Mylar® film. This thickness is very convenient, as X-rays with energies as low as 1 keV can be detected with no great difficulty. However, other kind of filters can be easily installed to reduce the X-ray yield on the detector, whenever it is necessary.

Particle detectors are located both in the forward and backward directions (using the beam incident direction as reference), to perform ion backscattering (RBS) and forward scattering (ERDA or PESA) analyses. The detectors are located at angles of 155° and 25°, for RBS and ERDA-PESA, respectively.

The sample positioner can be of various types. When a large number of samples is to be analyzed, a linear movement feedthrough is used, supporting an aluminum frame that contains the samples (with a maximum size of about 50 mm × 50 mm). Thus, vertical displacement of the frame allows positioning of the sample for beam irradiation. To have the largest possible solid angle subtended by the X-ray detector, the beam incident angle with respect to sample normal is 45°, while the X-ray detector is placed at 90° from the beam direction. Additionally, a high resolution linear movement feedthrough can also be used, together with a very fine collimator (as small as 50 μm), to obtain one-dimensional mappings of composition in samples such as sedimentary rocks, sea shells, or tree rings.

4. Detectors and electronics

Radiation is measured using different kinds of detectors. X-rays, for example, can be measured either using lithium-drifted silicon detectors, also known as Si(Li), or high purity

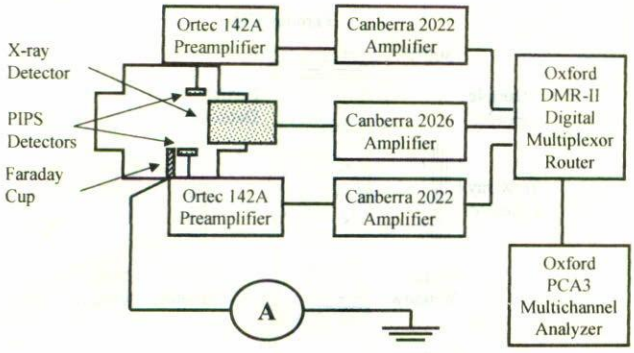


FIGURE 5. Diagram of the electronic setup for data collection during a typical experiment.

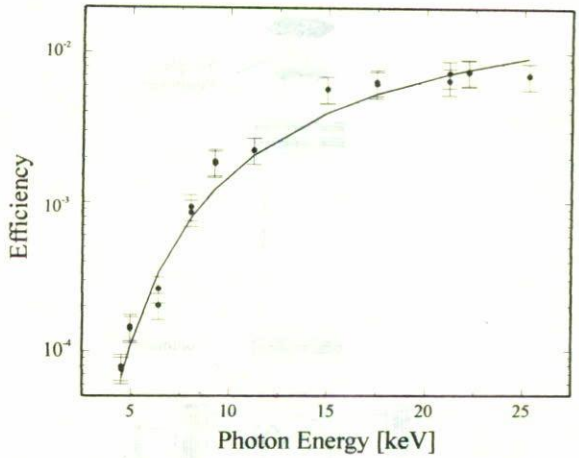


FIGURE 6. X-ray detection system efficiency as a function of photon energy, using a LEGe detector in the scattering chamber. Black circles are the experimental data, while the curve represents a fit to those data.

germanium detectors (HPGe or LEGe, for low energy germanium). The choice depends on the efficiency required for different X-ray energies [4]. Thus, measurement of X-rays from elements such as Mo, Ag, or Sn, is better achieved with a LEGe detector, while a Si(Li) is more sensitive to X-rays from Al or Si. On the other hand, ion detection is carried out using surface barrier or planar implanted passivated silicon (PIPS) detectors. The second kind, of more recent development, has been observed to be better than the former, as it shows a smaller resolution, its surface can be cleaned, and has a lower cost. In every other respect, both kinds are roughly equivalent. Care should be taken, though, to choose the proper depletion depth, considering different ranges of the ions inside the detectors, depending on their masses and energies.

The electronics setup is sketched in Fig. 5. The preamplifier is always attached to its corresponding X-ray detector. Preamplifiers for particle detectors are Ortec model 142-A, while the amplifiers are Canberra model 2026 for the LEGe detector, and Canberra model 2022 for the particle detectors. Depending on the number of parameters to be measured simultaneously, it may be necessary to use an Oxford DMR-II digital multiplexor-router, which allows processing of up to eight signals. In the particular case of the chamber, two to three signals are normally used. The DMR-II is connected to an Oxford PCA card, inserted into a personal computer. The PCA3 computer code, a multichannel analyzer emulator, is used to collect the data, which are saved on disks for future analysis using appropriate programs.

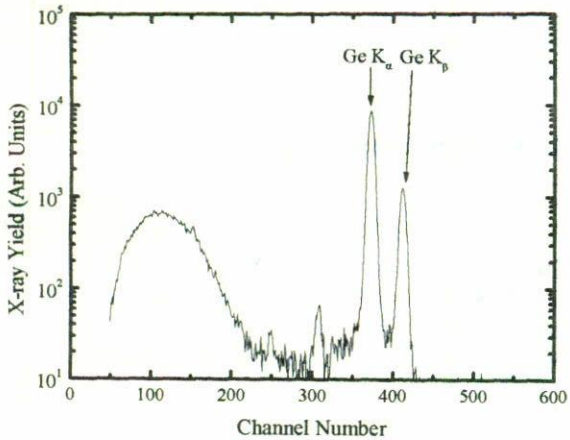
5. Experimental results

The first step in the characterization of the chamber for normal operation is the measurement of the X-ray detector efficiency [5]. This procedure is commonly done using thin film standards (MicroMatter Co., Deer Harbor, WA, USA) of pure metals or compounds. These films (with a thickness around 50 μg/cm^2) are deposited onto 3.5 μm Mylar® substrates. X-ray and backscattered ions detection are carried out simultaneously, with a proton incident beam (typical energies in the range 2.0 MeV to 2.5 MeV). The K X-ray lines of elements with atomic number between 13 and 35 (Al to Br) are used, as the X-ray production cross sections for these lines and proton energies are rather well known [6].

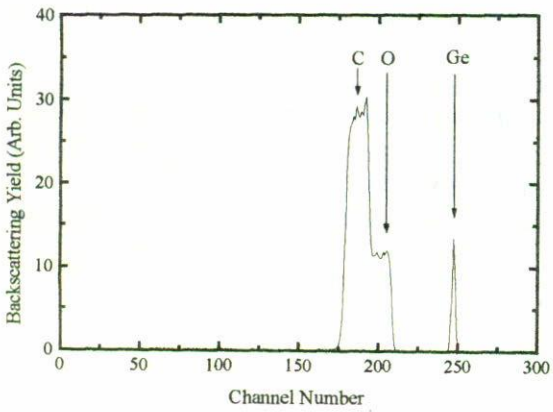
Figure 6 presents a typical efficiency curve. The detector is less efficient for X-rays from light elements, because they are absorbed in the windows along their path towards the detector. The detection of the backscattered ions is necessary to have an absolute measurement of the total charge incident on the sample. Alternatively, it is possible to detect protons scattered on the Mylar® substrate to integrate the current, or use the Faraday cup with that purpose, although it has been found that absolute current measurements in the latter case are less exact than the other two.

A typical application of the line is the study of thin samples, that is, those having a thickness smaller than the range of the ions in the sample [6]. In this case, as was the situation with the thin film standards, it is possible to detect three signals at the same time, namely, the characteristic X-rays, backscattered and forward-scattered ions. The advantage of doing this is the determination of contents of almost all the elements of the periodic table which may be present in the sample. Examples of spectra produced by the irradiation of Ge film deposited on Mylar® are given in Fig. 7. The X-ray spectrum taken with a LEGe detector (Fig. 7a) includes the Ge K_α and K_β lines, together with the lines from other impurities (Fe) and background radiation. Figure 7b is the backscattering spectrum, with signals from Ge (the peak at the right), and wider peaks from C and O present in the substrate. Finally, Fig. 7c represents the forward scattering spectrum (PESA). The left peak is due to H from Mylar®, while the right one corresponds to C, O, and Ge from the film; all these elements are represented by a single peak due to the poor energy resolution of the detector.

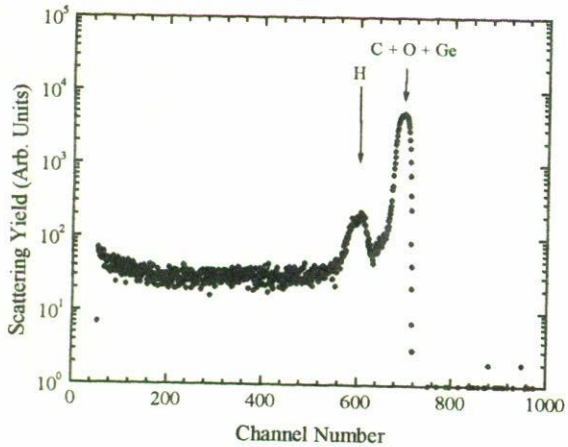
Other uses cover the measurement of X-ray production cross sections by heavy ion impact. Although the phenomenon has been known for a long time, theoretical models



(a)



(b)



(c)

FIGURE 7. Spectra obtained after irradiation of a 50 μg/cm^2 Ge film deposited onto a 3.5 μm Mylar substrate. (a) PIXE; (b) Ion backscattering; (c) proton elastic scattering.

are still unsatisfactory. For instance, the dependence of the cross section as a function of the target and projectile atomic numbers are not reliably known, especially because experimental results are scarce. Thus, studies of the afore mentioned cross sections are under progress, using C, N, O, and F ions, impinging on different films deposited onto pyrolithic carbon substrates, to produce either K or L X-rays. An example is shown in Fig. 8, where K X-ray production cross sections by C^{4+} impact on In are compared with theoretical predictions from the MECPSSR model [7] and the molecular orbital calculations by Montenegro and Sigaud [8].

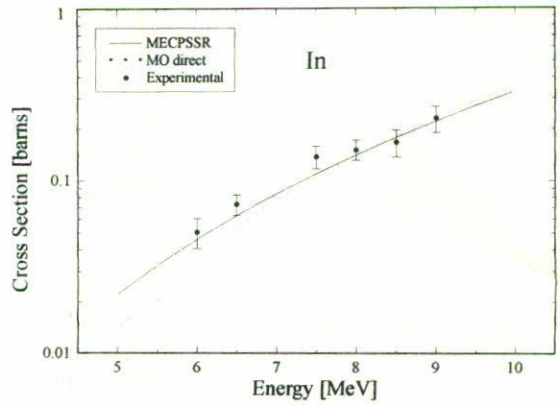


FIGURE 8. K X-ray production cross section of In by C^{4+} ions impact, as a function of ion incident energy. The black circles represent the experimental data, while the curves show theoretical predictions [7, 8].

6. Future developments

Among the improvements to the beam line that are planned in a short to medium term, it is possible to mention:

a) A more versatile scattering chamber, where more than three detectors can be installed, including a HPGe or scintillation detector for gamma ray studies [1], and even an electron detector, to study other more complicated inner-shell processes. Examples of these processes are the Coster-Kronig transitions, fluorescence yields, or the radiative Auger effect [6]. Also, a high resolution crystal spectrometer is desired.

b) Mounting of an external beam setup, so a proton beam can be extracted to the atmosphere (air or helium), allowing analysis of samples that can not be introduced in a vacuum chamber; examples are artistic or archaeological artifacts.

c) Automatization of the sample changing devices, in order to carry out routine analysis of large quantities of biological, environmental or materials samples; this may be important either for research or commercial analytical services.

7. Conclusions

The development of the new beam line will extend the capabilities of the Pelletron accelerator, both in the basic Physics

research (mostly in the X-ray and inner-shell processes related to heavy ion impact), and the applications in many fields of knowledge.

The high efficiency of the X-ray detection system, together with the possibility of applying several techniques simultaneously, allow fast, multielemental, and sensitive studies of many samples in a short time, reducing costs per sample in commercial analyses.

The still large unstudied area of X-rays and inner-shell processes induced by heavy ion impact will find a suitable device for conducting that research in Mexico. In a medium term, it is expected that even more complex studies will be followed, as the detection of several types of secondary radiation offers a deeper understanding of the many variables involved in those phenomena.

Acknowledgments

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