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THE LABORATORY OF MATERIAL ANALYSIS WITH ION BEAMS LAMFI - USP

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ABSTRACT

LAMFI is a laboratory dedicated to the development and application of ion beam techniques for the analysis of bulk materials and thin films. Its main facilities comprise an 1.7MV Pelletron tandem accelerator and two analytical setups, one mainly for Rutherford Backscattering Spectrometry (RBS), and channeling, and another, for Particle Induced X-ray Emission (PIXE) analysis. Since its beginning in March 1992, over 10 institutions involving about 68 researchers, 42 graduate and 9 undergraduate students have been direct or indirectly users at LAMFI..

1. INTRODUCTION

LAMFI (Laboratório de Análise de Materiais por Feixe Iônico) is a laboratory dedicated to the development and application of ion beam techniques for the analysis of bulk materials and thin films. LAMFI is the result of a joint effort of different researcher groups from the University of São Paulo, which, since the seventies, have been applying nuclear techniques for the analysis of air pollution, environmental samples, semiconductors and thin films. Its main facilities comprise an 1.7MV Pelletron tandem accelerator and two analytical setups, one mainly for Rutherford Backscattering Spectrometry (RBS), and channeling, and another, for Particle Induced X-ray Emission (PIXE) analysis. LAMFI is a multi-user laboratory whose operation is supervised by a scientific committee formed by five representatives from the Institute of Physics (IFUSP) and two from the Engineering Faculty (EPUSP), both at the University of São Paulo. LAMFI is operated and maintained by a technical staff whose coordinator is indicated by the scientific committee.

2. THE FACILITIES

Right after arrival of the accelerator in 1992, the LAMFI was first installed on the 9th floor of the Pelletron Accelerator building. This allowed its immediate operation, while waiting for the conclusion of the its own building. Though in temporary installations, all the necessary resources needed to fully operate the accelerator and the analytical setups were available. In November 96, LAMFI was disassembled and moved to a new building. At the same time, the former technical coordinator, Prof. Juan Carlos Acquadro, decided to retire being substituted by Prof. Manfredo H. Tabacniks. In September 1997, LAMFI resumed its operation in the new installations.

2.1. The Accelerator

The main equipment at LAMFI is a 1.7MV NEC 5SDH tandem Pelletron accelerator with a gaseous N_2 stripper. Two ion sources, an RF alphatross with a rubidium charge exchange cell for He^- beam and a SNICS II (Secondary Negative Ions by Cesium Sputtering) for H^-, Li^-, C^-, Si^-, O^- and other heavier beams. The high energy side switching magnet, which is also used to control the beam energy, has 4 output lines respectively at ±30° and ±15° plus the zero degree line. Two complete analytical setups, each one with its own high vacuum chamber, vacuum pumps, detectors and data acquisition electronics, are connected to the switching magnet. The setup connected to the -15° line is mainly used for RBS and channeling analysis, while the +30° line is connected to a home-made PIXE analysis setup which is mainly used for environmental sample analysis. .

2.2. The analytical setups

The RBS station, shown in Figure 1, is a NEC high vacuum chamber (43cm ID and 15cm high) with a computer controlled goniometer with 5 degrees of freedom (x, y, z, θ, and ϕ). Surface barrier detectors can be mounted at any angle on top of a rotating table. A fixed reference SB detector is mounted at 170° to the beam. A simple and effective vacuum load lock completes the assembly. Data acquisition is done with two ORTEC 918 Multichannel Buffer controlled by an IBM-PC microcomputer. RUMP code for microcomputers (Doolittle, 1985; 1986; CGS, 1996) is used for spectra manipulation and analysis. One of the detectors on the rotating table with a 7μm thick Al absorber foil is used for Hydrogen analysis by Forward Recoil Spectrometry (FRS), (Baglin et al. 1992).

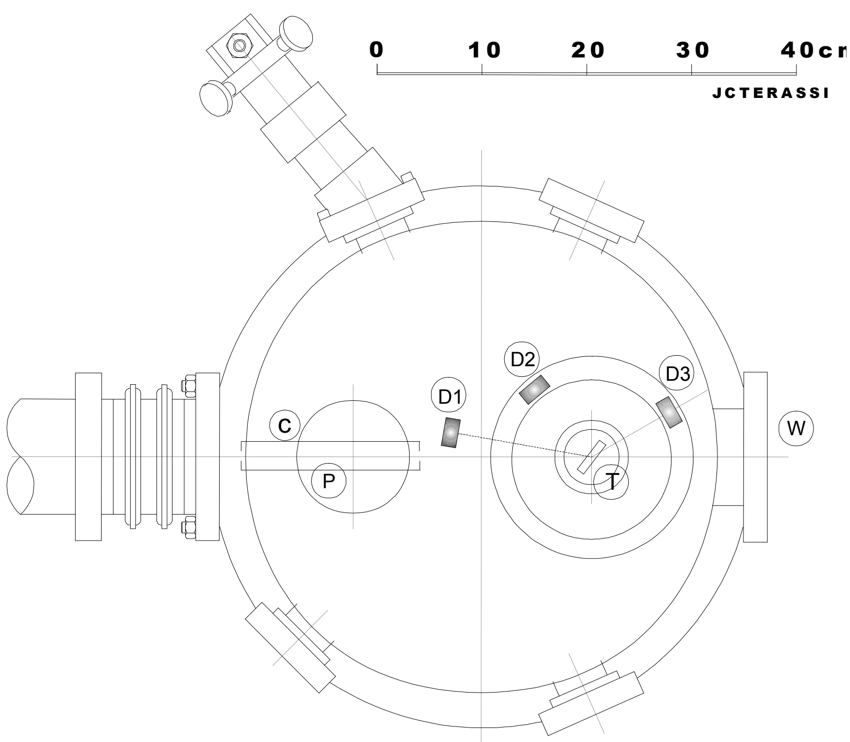

Figure 1. The RBS setup at LAMFI. (D1), (D2), (D3) Surface barrier detectors at 170°, 110° and 30°; (G) Goniometer and target holder; (P) Pumping port, (V) View port; (T) telescope for target positioning; (B) Beam line.

The PIXE station, depicted in Figure 2, is a home-made high vacuum chamber (15cm ID, 25cm high), that has been used since 1976 for PIXE analysis of air pollution at the Institute of Physics (Souza et al., 1976; Tabacniks, 1983). Initially installed on the 8MV Pelletron accelerator in the Nuclear Physics Department, and recently adapted to the new machine, the PIXE setup uses two Si(Li) KEVEX detectors with pulsed optical feedback for best resolution. One detector at 158° has a 7,1mm^2 crystal and resolution of 138 eV FWHM@MnKα. A 50μm thick Be absorber is used to avoid backscattered protons from hitting the detector. This detector and geometry was optimized for low energy X-ray detection. The detector for high energy X-rays has a 25mm^2 crystal, resolution of 145eV FWHM@MnKα, and is mounted at 90° to the target. A 550μm thick Mylar absorber is used to avoid low energy X-rays from being detected thus enhancing the high energy part of the spectrum on a clean background, without pile up events. Both detectors operate in vacuum. The use of two detectors respectively for low (1.3 to 5keV) and high (5 to 20keV) X-ray energies of the spectra, is intended to reduce data acquisition time while optimizing the X-ray yield and thus the detection limits. A computer controlled linear target holder for 18 samples completes the assembly. Inside the PIXE chamber are attachments to install surface barrier detectors that can be used for RBS measurements and/or beam monitoring when doing thick target analysis (Tabacniks, 1993; Martins and Tabacniks, 1993). The faraday cup accuracy for charge measurement was tested against the beam scattered in a thin gold foil into a surface barrier detector, with an agreement better than 1% (Martins, 1993).

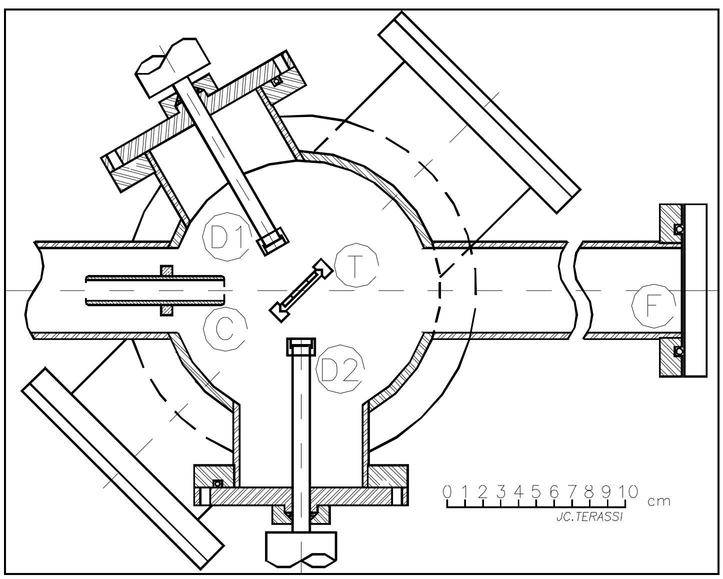

Figure 2. PIXE setup at LAMFI. (C) beam collimator, (F) Faraday cup, (T) Target holder, (D1) Low X-ray energy Si(Li) detector, (D2) High energy X-ray Si(Li) detector.

Data acquisition is done with an ORTEC 919 spectrum master ADC and multichannel, controlled by a Pentium type microcomputer. X-ray spectra analysis is done using AXIL software (V.Espen, et al.). PIXE analyses are usually run with 2.4MeV proton beam. Mass calibration is achieved analyzing homogeneous evaporated thin film standards (~50μg/cm^2) deposited on 2.5μm thick Mylar film, supplied by Micrommater, USA. Figure 3 shows the calibration curve for the São Paulo PIXE system. Figure 4 shows the result of 59 PIXE analysis of one multilayer target (Al/Ti/Zr/Mylar) used to monitor the calibration stability over a period of three months. Data were plotted in form of normalized residue to the average of the series according to $y = (x_i - \bar{x}) / \bar{x}$, where x_i is each individual data point. Standard deviation was 10% for Al data 4.6%, for Ti and 3.3%. for Zr The higher deviation for Al and Ti may be respectively due to higher gradient of the X-ray ionization cross section in respect to energy.

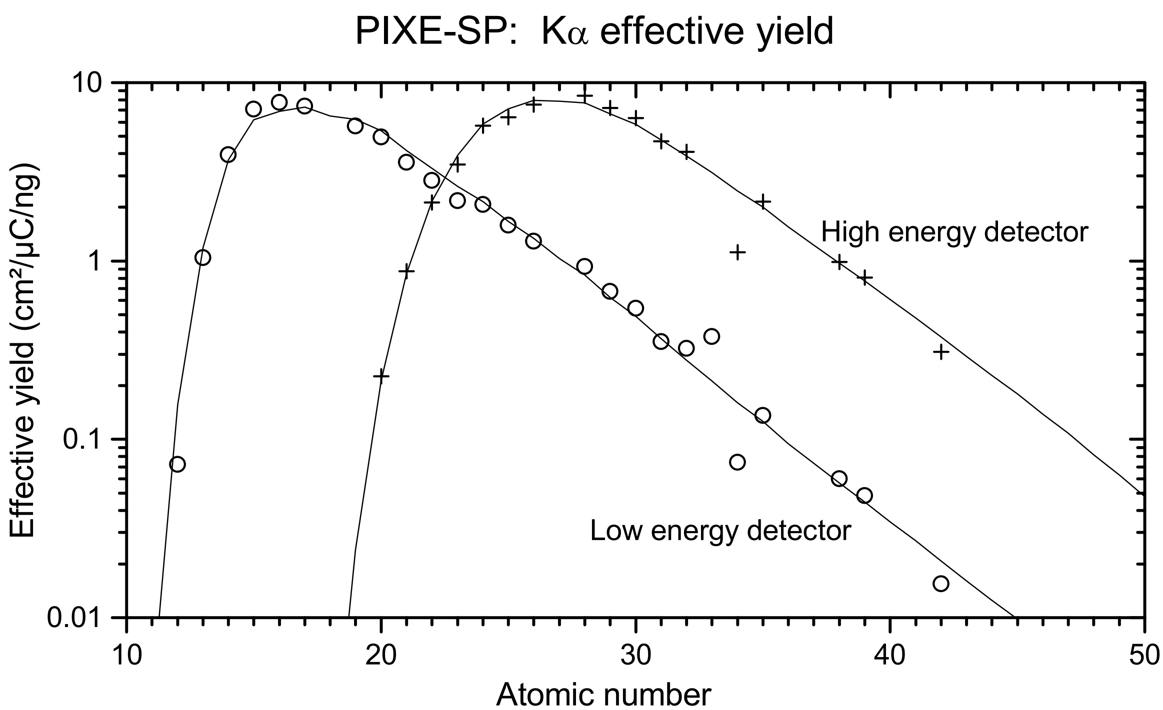

Figure 3. Calibration curves for the São Paulo PIXE system (adapted from Nascimento, 1997).

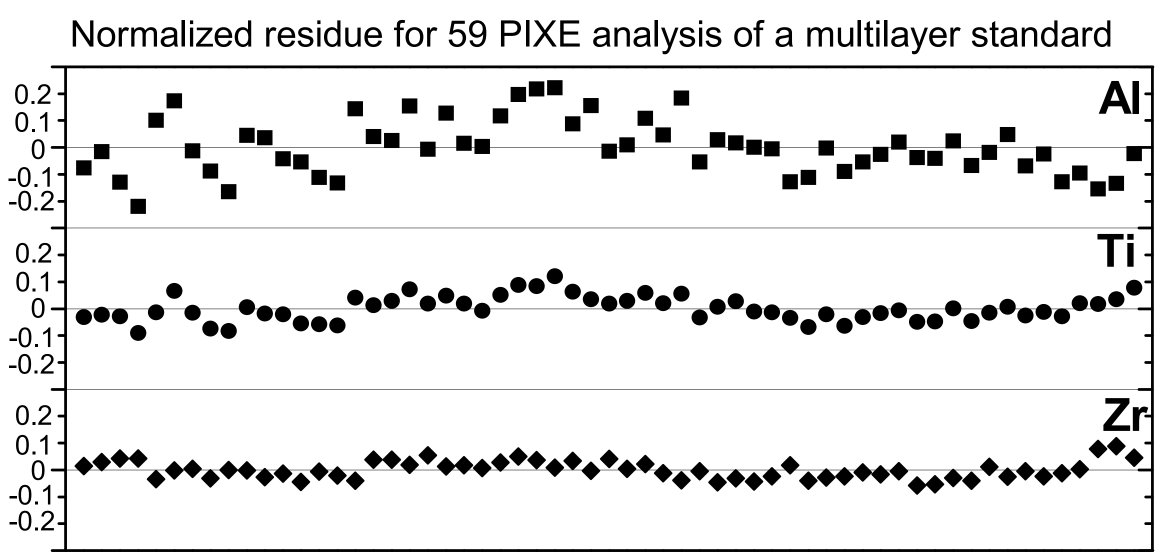

Figure 4. Calibration stability of the São Paulo PIXE system over a period of three months. (adapted from Yamasoe, 1994).

3. ALLOCATION AND USE OF MACHINE TIME AT LAMFI

LAMFI started its activities in March 1992 with all available machine time allocated to the final users. The following is a partial statistics of the accelerator time and its use, from available data over different periods. Extrapolation of these data is possible, because user profile and technical conditions at LAMFI did not change substantially over the past five years. Figure 5 shows the fraction of the effective working days at the accelerator allocated to final users, from 1994 to 1996. The difference to 100% is due to failures, preventive maintenance and vacations. The major user groups at LAMFI are listed below and the allocation and division of the machine time among them are shown in Figure 6. :

GEPA	Group for Air Pollution Studies, IFUSP Coordinator: Prof. Dr. P.E. Artaxo
LSI	Laboratory of Integrated Systems**.** EPUSP. Coordinator: Prof. Dr. N. I. Morimoto
LMM	Laboratory of Magnetic Materials, IFUSP. Coordinator: Prof. Dr. F.P. Missell
OTHERS	Profs. J.C. Acquadro and M.H. Tabacniks and other researchers from IFUSP and other institutions.

Figure 5 shows that about 2.5 months per year were used for accelerator maintenance, tests, and vacations[1]. Analysis time at LAMFI used to be distributed according to Figure 6. GEPA, thus PIXE analysis, used about 50% of the available time. RBS analysis was assigned to the other half of the available time. Malfunctioning, power failures and technical problems reduced the overall efficiency to about 79% in 1995 and 47% in 1996, when the Pelletron Laboratory had its central air/water conditioning system renewed with many interruptions and failures, thus affecting all the work done in the building. In the new installations it is expected to reduce these kind of problems to a minimum.

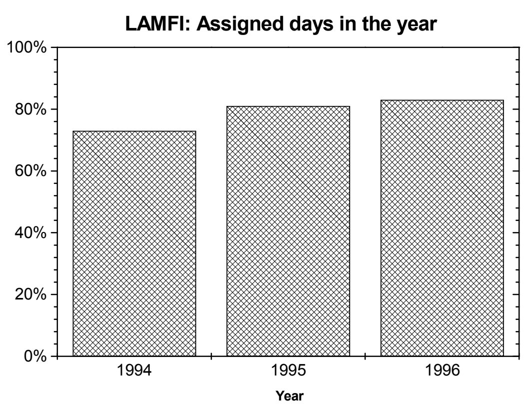

Figure 5. Fraction of accelerator running days allocated to the final user.

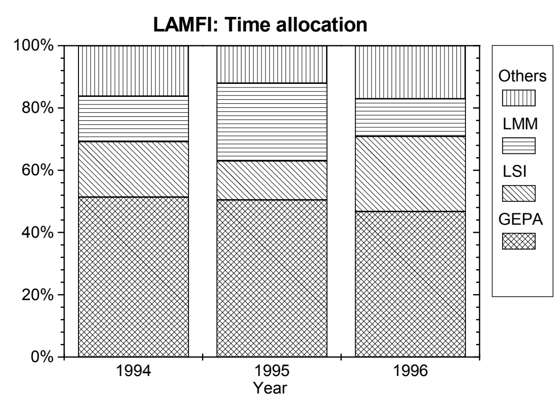

Figure 6. Allocated machine time among major users

5. USERS AND RESEARCHERS AT LAMFI

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[1] LAMFI used to close for vacation time due to the small number of technicians to operate the accelerator. Nowadays, with adequate training of students and users, it may be possible to avoid such interruptions.

A list of institutions that were or are users at LAMFI is shown below.

- Department of Applied Physics (DFA), IFUSP.
- Department Physics of Materials and Mechanics (DFMM), IFUSP.
- Department of Nuclear Physics (DFN), IFUSP
- Department of Experimental Physics (DFE), IFUSP
- Laboratory of Integrated Systems (LSI), Integrated Microsystems Division. Department of Electric Engineering, EPUSP
- Nucleus for Research and Development of Agricultural Instrumentation, NPDIA, of the National Center for Research and Development in Agriculture, EMBRAPA. São Paulo.
- Institute for Nuclear and Energy Research, IPEN, São Paulo
- Institute of Physics, University of Campinas, UNICAMP
- Federal University of Rio Grande Do Sul, UFRGS
- Pontifical Catholic University of Rio de Janeiro, PUC-RJ

These institutions represent over 68 researchers, 42 graduate and 9 undergraduate students that have been direct or indirectly users at LAMFI.

6. FINANCIAL RESOURCES

LAMFI has been financed by scientific agencies in Brazil with basis on the scientific production of its users. There are two types of resources available at LAMFI: grants assigned directly to LAMFI, mainly from the Research Supporting Foundation of the State of São Paulo (FAPESP), which are controlled by its technical coordinator, and contributions from major users, in the form of equipment or payment for a few unforeseen expenses.

7. RECOMMENDATIONS OF THE INTERNATIONAL EVALUATION COMMITTEE

From August 11 to 13, 1997, LAMFI and its activities were evaluated by an international committee specially invited for the task, under the sponsorship of the Research Supporting Foundation of the State of São Paulo (FAPESP). The committee consisted of:

Dr. John E.E. Baglin, IBM Almaden Research Center, CA, EUA
Dr. James W. Mayer, Professor of Science and Engineering, Arizona State University, EUA
Prof. Dr. Israel J.R. Baumvol, Federal University of Rio Grande do Sul
Prof. Dr. Alejandro Szanto de Toledo, DFN-IFUSP
Prof. Dr. Mahir Saleh Hussein, DFN-IFUSP
Dr. Johan Biersack, Visiting Scientist, DFN-IFUSP

In their final report (Baglin, et al. 1997), these scientists concluded that LAMFI is in an ideal position to make positive contributions to the Brazilian scientific and engineering communities and to the University of São Paulo. In order to optimize the work done at LAMFI the committee suggested the construction and installation of a new basic analysis chamber for RBS, FRS and

PIXE analysis, to replace the present one. The committee also suggested that a three-tiered staff team composed by a technical person to operate the accelerator, a graduate or post-doc to carry out specialized analyses, and a faculty member be implemented. In order to fulfill this last recommendation it is necessary only to hire the graduate or post-doc specialist. Except for the current staff composition, the committee did not find any major weakness in the LAMFI organization and infrastructure.

8. CONCLUSIONS

In a new building, under new technical coordination and based on five years of experience, LAMFI has now the conditions to expand its analytical options and tune the experimental setups to maximum performance.

There is a solid group of users that provide scientific support to the work done at LAMFI. This support can be and is being used to subsidize new proposals for grants and scholarships. On the other hand, the need for a graduate or post-doc fellow to carry out specialized analyses and manage the Laboratory is increasing with the introduction of the new analytical options and the installation of new equipment.

The PIXE setup will be fully automated to account for the thousands of samples that have to be analyzed on a systematic and controlled way and, in the future, it should also allow automatic and unattended overnight data acquisition. The RBS system needs to be upgraded and data acquisition, charge collection, beam collimation, target holder, have to be improved. The addition of FRS (ERDA) for Hydrogen analysis, which is an important complement for the RBS analysis, is being undertaken. This option should be available soon.

In order to increase the involvement of users with LAMFI and, at the same time, decrease demand on staff, highly skilled technicians and students of the users' laboratories are encouraged to help with testing, maintaining and upgrading some of laboratory's equipment. This practice may not be possible in "professional" laboratories, but is welcome in a University, and may represent a great educational opportunity for everybody. The result is a higher commitment of the users and a very prompt feedback on the analytical conditions.

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