Large areas elemental mapping by ion beam analysis techniques

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Abstract. The external beam line of the Laboratory for Material Analysis with Ion Beams (LAMFI) is a versatile setup for multi-technique analysis. X-ray detectors for Particle Induced X-rays Emission (PIXE) measurements, a Gamma-ray detector for Particle Induced Gamma-ray Emission (PIGE), and a particle detector for scattering analysis, such as Rutherford Backscattering Spectrometry (RBS), were already installed. In this work, we present some results, using a large (60-cm range) XYZ computer controlled sample positioning system, completely developed and build in our laboratory. The XYZ stage was installed at the external beam line and its high spacial resolution (better than 5 µm over the full range) enables positioning the sample with high accuracy and high reproducibility. The combination of a sub-millimeter beam with the large range XYZ robotic stage is being used to produce elemental maps of large areas in samples like paintings, ceramics, stones, fossils, and all sort of samples. Due to its particular characteristics, this is a unique device in the sense of multi-technique analysis of large areas. With the continuous development of the external beam line at LAMFI, coupled to the robotic XYZ stage, it is becoming a robust and reliable option for regular analysis of trace elements (Z > 5) competing with the traditional in-vacuum ion-beam-analysis with the advantage of automatic rastering.

1. Introduction
Among most of the analytical techniques, there is an effort to develop imaging capabilities. This is because the correlation of the element concentration with the position on the sample can give more information on heterogeneous samples, enabling not just studies related to formation and solid phases, but also the element maps can be used as sample maps for locating interesting spots for detailed analysis. It can be found in literature many studies that produce images using XRF, XPS, EDX, PIXE, and others. But these studies are usually performed in small areas and most of them are performed in vacuum environment (which imposes restrictions on the samples that can be studied).

The external beam line in the Laboratory of Material Analysis with Ion Beams (LAMFI) is a versatile setup for multi-technique analysis using ion beams. Recently, a setup upgrade included the installation of a particle detector enabling in-air particle scattering analysis [1]. X-ray detectors for Particle Induced X-rays Emission (PIXE) measurements and Gamma-ray detector for Particle Induced Gamma-ray Emission (PIGE) measurements was already installed, and some studies using this analytical station have been reported [2-5]. With the continuous
development of our external beam line, it is becoming a robust multi-analysis station for matrix
and trace elements (Z > 5) that meets applications in disparate areas. The great advantage of
using this analytical station is that it is virtually no necessary any sample preparation, what
increases the broad of samples that can be studied.

Following the imaging tendency, we developed a 3D robotic system for sample positioning.
The system was completely developed in our laboratory, and was coupled to the external beam
line. This is a high resolution machine that enables to position the sample in the external beam
line with high accuracy and high reproducibility. With the automation of this robotic device and
the development of the electronic interface that establish communication between the computer
that controls the positioning system and the detector electronics, we are capable to produce
elemental mapping of large areas by rastering the broad beam (0.7-mm diameter) provided by
the accelerator. Comparing capabilities with other similar setups for imaging using ion beam
analysis techniques that can be found in other laboratories around the world, the low resolution
of ours, mainly due to the broad beam, makes the observation of small details in the sample
a weakness of our device. On the other hand, what makes our device unique is the ability to
raster large areas, and to obtain elemental maps.

2. Methodology

The computer that controls the robotic sample holder is also able to communicate with the
detector electronics and automatically record the detector spectra and the sample position
coordinates. Keeping track of all this parameters it is possible to construct elemental maps
using the signal collected by the detectors in a spectra corresponding to a certain element and
its respective position coordinate in the sample surface. Fig. 1 illustrate the rastering process
and the variables that can be chosen by the user in the software of control.

![Figure 1. Illustration of the rastering process. The steps (dx and dy) and the size of the
boundaries of a rectangular region (Lx and Ly) can be chosen by the user in the software of
control. It is also a variable to be chosen the acquisition time in each point.](image-url)
3. Results
To explore the potentiality of the rastering mode in the external beam setup to elaborate elemental maps we performed the ratering in a test sample. This sample is a structured Cu foil on top of a Ta foil. The structure of the Cu foil is composed of some holes of different sizes, in order to explore the resolution limits and the size of the minor structure that can be observed. Fig. 2 shows a picture of the test sample.

![Image of test sample with labels 5mm, 2.5mm, and 1.0mm]

**Figure 2.** Picture of the test sample. The holes were fabricated with different diameters to explore the minor structure that can be observed.

For this test sample we used the robotic device to raster the broad beam with steps of 1.0 mm and measured the PIXE spectra for each point. The map obtained is presented in Fig. 3. With this measurements we were able to create maps of Cu and Ta, and despite the superposition of the K-lines of Cu and the L-lines of Ta, it is possible to observe the structures in the Cu foil.

Despite the size of the pixels that indicates the low spatial resolution of the image, we can identify all the structures of the Cu foil. The 1-mm diameter holes seems to be near the spatial resolution limit defined by the beam size.

In the beam exit at the external beam setup, there is a collimator that restricts the beam diameter to 1.0 mm. In order to exploit what is the best rastering resolution obtained, we performed a rastering of a 20.0x20.0-mm² area of a metallic grade. Fig. 4 shows a map of intensities for the Fe-Kα line along the measured area.

It is possible to observe that, even with the treatment of the image, the 1-mm step produced a pixelized image of the grade. A much better result is obtained using 0.7-mm step, thus this value was used as default for this beam size. To improve the spatial resolution, some other smaller collimator have been made (1.0 mm, 0.5 mm and 0.25 mm), providing the option of beam size that best suits each analysis.

The image generated in the 1.0-mm step rastering took 3 hours to be completed, while the 0.7-mm step image took 6.3 hours. Tests of movement of the equipment showed that the system takes, on average, 12 seconds between the measurement of two consecutive points with an acquisition time of 15 seconds in each spot. We estimate that 90% of this time is due to the resetting process of the detectors, meaning that there is room for improvements replacing the electronics by a faster model.
4. Discussion and Conclusions

The external beam setup under development at LAMFI is becoming a versatile analysis station for ion beam measurements. As it can be seen in the theoretical efficiency curve for the x-rays detectors used for PIXE measurements (see Fig. 7), a wide range of elements can be detected. This curve was calculated using the detector efficiency provided by the manufacturer [8], the
windows x-rays absorptions, and the solid angle of the detectors.

![Figure 5](image.png)

**Figure 5.** Theoretical relative curves of efficiency for the two x-rays detectors used for PIXE measurements at the external beam setup. Respective energies for the ka lines are marked as reference.

Also, the development of a robotic machine capable of indexing the position of PIXE measurements in a sample is showing to be very useful for production of elemental maps. The rastering of the broad beam produced by the accelerator can create maps with sub-millimeter resolution of large areas. This innovative use of a broad beam can open new perspectives of research and measurements in our laboratory.

5. References

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