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A new mapping acquisition and processing system for simultaneous PIXE-RBS analysis with external beam

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ABSTRACT

The combination of ion beam analysis techniques is particularly fruitful for the study of cultural heritage objects. For several years, the AGLAE facility of the Louvre laboratory has been implementing these techniques with an external beam. The recent set-up permits to carry out PIXE, PIGE and RBS simultaneously on the same analyzed spot with a particle beam of approximately 20 μm diameter.

A new mapping system has been developed in order to provide elemental concentration maps from the PIXE and RBS spectra. This system combines the Genie2000 spectroscopy software with a homemade software that creates maps by handling acquisition with the object position. Each pixel of each PIXE and RBS maps contains the spectrum normalised by the dose. After analysing each pixel of the PIXE maps (low and high energy X-ray spectra) with the Gupixwin peak-fitting software, quantitative elemental concentrations are obtained for the major and trace elements. This paper presents the quantitative elemental maps extracted from the PIXE spectra and the development of RBS data processing for light element distribution and thin layer characterization. Examples on rock painting and lustrous ceramics will be presented.

1. Introduction

The combination of PIXE, PIGE and RBS is particularly fruitful for the study of cultural heritage objects. For many years, the experimental set-up of the AGLAE facility of the Louvre laboratory has been built up to implement these techniques with an external beam. Single-spot mode PIXE with the external microbeam has been successfully used on art objects and samples that are not compatible with analyses in vacuum [1–3]. For the analysis of layered structures such as painting [4], lustrous ceramics [5] and altered or corroded objects [6,7], the simultaneous combination of PIXE and RBS has been then developed and is now routinely used.

In addition of point analysis, elemental maps are full of information about spatial distribution, reconstruction of worn away structure and location of region of interest [8]. In the past years, PIXE imaging was carried out for the analysis of Egyptian papyrus [9] or ink [10]. In order to combine PIXE with RBS, a new mapping system has been recently developed for collecting ion beam analysis data and generating multi-detector images. In this paper, the new system providing elemental concentration maps from PIXE spectra and elemental distribution maps from RBS spectra are described and examples are presented.

2. Experimental setup

The AGLAE facility of the Centre de Recherche et de Restauration des Musées de France is based on a 2 MV tandem accelerator (Pelletron 6SDH-2). The installation has been described in details in several previous papers [11,12]. The simultaneous PIXE-RBS experiment conditions are the following. For the PIXE detection, two Si(Li) detectors oriented at 45° to the particle beam record the X-ray spectrum [13]. The low energy X-rays (0.5–15 keV) are detected by a Gresham detector of 10 mm^2, with a Moxtek AP3.3 entrance window (energy resolution of 145 eV at 5.9 keV). A continuous flow of He is used to eject air from the volume between the detector crystal and the sample. The higher energy X-rays (4–40 keV) are recorded by a Gresham detector of 30 mm^2, with a 8 μm Be entrance window (energy resolution of 152 eV at 5.9 keV). A filter (50 μm Al, or other materials according to the analyzed matrix) is added to absorb the intense matrix X-rays that could saturate the electronic amplifier and pulse processor. This configuration is quite powerful in providing the panoramic elemental composition of the material, from major (generally light) elements to minor and trace elements (intermediate and heavy).

For RBS analysis, a PIPS surface barrier detector from Canberra (PD 50 mm^2-resolution 12 keV) is used. A special housing under vacuum with a Si_3N_4 entrance windows has been made in order to obtain RBS spectra of a good quality (40 keV resolution with 3 MeV protons).

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An important factor for quantitative analysis is the measurement of the number of incident particles. Under conventional vacuum conditions, this is simply done by current integration on an insulated chamber. This technique cannot longer be used in the case of external beam due to the loss of secondary electrons. Since several years, the irradiation dose is measured with the Si X-ray line of the Si_3N_4 membrane of the exit nozzle with a Si PIN detector (XR-100 from Amptek with a 9 inches flexible cable and PX2CR power supply and amplifier).

For mapping, the current facilities allow the external beam to be scanned over an area of few mm^2 [14–17]. For most of our applications, the area of interest is larger and the elemental mapping is obtained by moving the sample instead of scanning the beam. The sample is fixed on a remote controlled stage equipped with high precision stepping motors.

3. Acquisition system

The external beamline is set up with the Genie™ 2000 Spectroscopy Software from Canberra. This software is used to control acquisition of multiple detectors independently and simultaneously and to display spectra. The electronic system of the two PIXE detectors is composed of an analog pulse processor "Titan" from Gresham and an analog ADC8701 (Analog-to-Digital Converter) from Canberra. For the particle detector, a 142A preamplifier and a DSP9660 (Digital Signal Processor) from Canberra are used for pulse processing and signal digitizing. The ADC and DSP are linked to a Canberra AIM 550 (Acquisition Interface Module) connected to the Ethernet network. So, the workstation can be localised far away from the experimental beamline.

The dose is monitored by integrating the Si–K line emitted by the Si_3N_4 window with a SCA (Single Channel Analyser) from Ortec, adjusted on the Si pulse height. For single-spot analysis, a home made software called "Orion" developed with LabView© controls all the devices useful for external IBA: the beam extraction by opening/closing the Faraday cup, helium gas flux, dose monitoring, detector counting rate, laser pointer, light, spectrum acquisition, experimental condition record, sample information.

4. Mapping system

Due to our specific external set-up and to the existing acquisition system and dose monitoring, no software was commercially available to control them and generate maps. A new software called "AGLAE_MapS" has been developed (Fig. 1). It uses the acquisition system Genie2000 described above and generates maps by a combination of the spectra and position of the objects. The sample positions are controlled with linear motor stages from Newport Corporation.

For each position, Genie2000 acquires spectra of all detectors according to a preset dose value. When the sample is moving, the acquisition operates continually. Between each position, AGLAE_MapS clears the spectra, resets the dose counter, acquires the data, save all spectra and move the sample to next position. At the end of every line of movement, AGLAE_MapS saves all the spectra of each detector in a file with the EDF format (ESRF Data Format). The EDF format is suited to large data sets and it allows to use all free softwares developed by the BLISS (Beam Line Instrumentation Software Support of ESRF [18]), such as PyMCA [19] which is basically dedicated to the visualization and analysis of energy-dispersive X-ray fluorescence data. Finally, AGLAE_MapS generates two PIXE (low and high energy X-rays) and one RBS maps simultaneously. Each pixel of the map contains a spectrum normalised by the preset dose value.

5. Elemental quantitative maps

For extracting elemental concentration from the PIXE maps, each pixel must be processed.

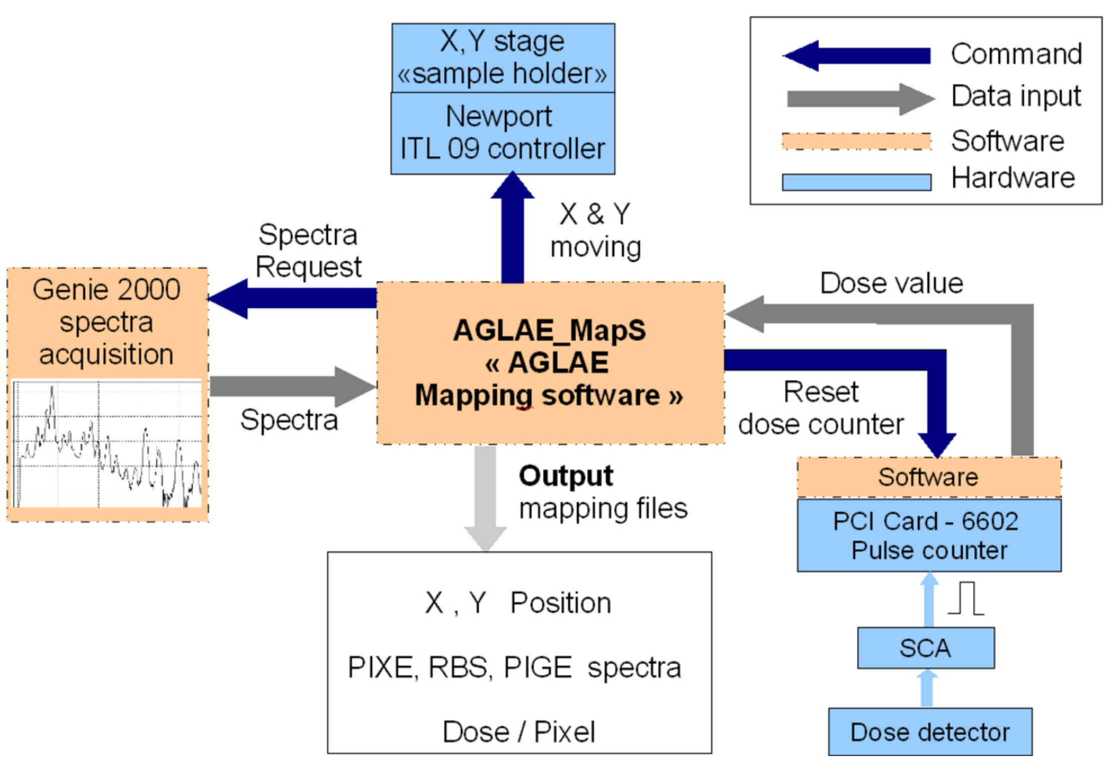

Fig. 1. Principle of the «AGLAE_MapS» mapping system.