Nuclear Instruments and Methods in Physics Research B 268 (2010) 1911–1915

Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research B

journal homepage: www.elsevier.com/locate/nimb

Preliminary experiments: High-energy alpha PIXE in archaeometry

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ARTICLE INFO

Article history:
Available online 25 February 2010

Keywords:
Alpha particle
High-energy
PIXE
Cross-sections

ABSTRACT

This paper describes the work realized at the "Centre Européen d'Archéométrie" to highlight the utility of high-energy alpha PIXE in the particular field of archaeometry and to introduce the developments done and to be done to complete the knowledge of high-energy alpha PIXE.

It starts with the comparison of the yield and the noise background between several alpha particle beams and the comparison between alpha particle and proton beams on different thick and thin references. After, this paper depicts the developments done at the "Institut de Physique Nucléaire, Atomique et Spectroscopie" to perform such high-energy experiments, first on standards and later on cultural heritage objects. Moreover, it introduces the problematics of such beams for the quantification in PIXE by the intermediary of the knowledge of the ionization and X-ray production cross-sections and also the developments done to answer to this serious lack in the databases.

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1. Introduction

Particle induced X-ray emission (PIXE) is a standard and well-known ion beam analysis (IBA) technique, nevertheless the method has still some unresolved factors.

The "Centre Européen d'Archéométrie" (CEA) is already active in conventional IBA thanks to a dedicated beam line performing both PIXE and PIGE analysis on cultural heritage objects in air [1–3].

Besides performing conventional 3 MeV proton beam analysis, a project has been launched to complete the knowledge of PIXE.

Indeed, if PIXE is applied to the study of a work of art for several years, most of the laboratories involved in such a field use low-energy beams mostly limited by the kind of accelerator available.

The Liège center owns a "CGR 520 MeV" cyclotron which permits us to reach higher energies up to 20 MeV with both alpha particle and proton beams.

The use of high-energy beams in the field of archaeometry is rare, however they have been already successfully performed [4,5].

We will not give any details of high-energy proton PIXE but concentrate instead on high-energy alpha particle beams. Such beams are still not often used in our domain and we foresee a long and interesting work to develop them.

In archaeometry, due to their weak penetration power (compared to protons), alpha particles are generally given up in favour of protons. The use of high-energy beams could compensate for this characteristic and permit other prospects than the conventional 3 MeV proton beam analysis.

Moreover, our cyclotron permits the adaptation of the energy of the beam, and hence the penetration of the beam, to the desired analyzed layer thickness.

Also, thanks to their higher mass, alpha particles induce a weaker primary Bremsstrahlung background than protons.

The weaker primary Bremsstrahlung background combined with the increase of the yield of the X-rays emitted by the elements present in the matter and of the penetration power of the alpha particles with the energy beams lead to reconsideration of the application of such beams for the study of archaeological works of art.

The limit of detection for several elements, or the ratio between the yield and the background noise (from both primary alpha particles and secondary electrons [6]) had been studied here to highlight the perspicacity of high-energy alpha PIXE. It has been also compared to conventional proton PIXE. The limit of detection has already been studied for low-energy alpha PIXE and already shown to give a considerable improvement [7].

Due to the weak number of applications of high-energy alpha particle beams in archaeometry, all the physics parameters used for the quantification of the different elements present in the sample are still unknown (ionization and production cross-sections, etc.). The experiments reported here are the starting point of a new project aiming to measure the production and ionization cross-sections with high-energy alpha particles.

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doi:10.1016/j.nimb.2010.02.096

2. Highlighting

2.1. High-energy alpha particle beams

High-energy alpha particle beams are rarely used however they could be really useful in several particular cases where the conventional analyses would appear insufficient. We have therefore performed analyses on thin and thick samples to highlight their characteristics and their advantages to be used in PIXE.

The experiments have been carried out in air on the direct beam line of the cyclotron with both high-energy alpha particle (from 6 to 12 MeV) and proton (3 MeV) beams on thin and thick references.

The control of the fluence has been carried out thanks to two separated methods.

The first method is based on the detection of the back-scattered ions by the back side of the extraction nozzle, gilded with gold, with a PIPS detector. Fig. 1 shows a scheme of the extraction nozzle. The fluence is measured by the number of counts in the RBS spectrum in a relative energy range. Then, as the gold peak moves with the energy of the incident particles of the beam, the number of counts measured in the same energy range does not fit exactly with the same fluence for different beams, which only differ by their energy. This results in an imprecision in the control of the fluence.

The second method is based on the collection of the charge induced by the crossing of the beam through the extraction nozzle. It has the advantage to be independent of the energy of the beams but unfortunately the precision on the control of the fluence is degraded by the instability of the beam position inside the line which may vary a little bit with time. Then we used these two complementary methods to get the most precise fluence control possible.

A three-axis moving system permitted us to position all the references in the same geometry thanks to a laser, inserted in the beam line by intermittence, coupled to a CCD camera.

The X-rays emitted by the samples were collected with a Si(Li) X-ray detector and the Titan pulse processor from "e2V scientific instruments" mounted at 40° with respect to the normal of the sample surface. The use of an ultra-thin polymer AP3.7 "Moxtek" window [8] led us to construct a deflector, using permanent magnets, to prevent any damages in the 10 mm^2 crystal which could be induced by the ions back-scattered from the samples. The deflector had also been successfully tested for beam energy up to 14 MeV. A 1 μm carbon foil had been added to this device to suppress the visible light.

Moreover, helium gas was allowed to flow in the one centimeter gap between the sample and deflector to avoid energy straggling, angular scattering of the beam and absorption of the light X-rays emitted by the samples.

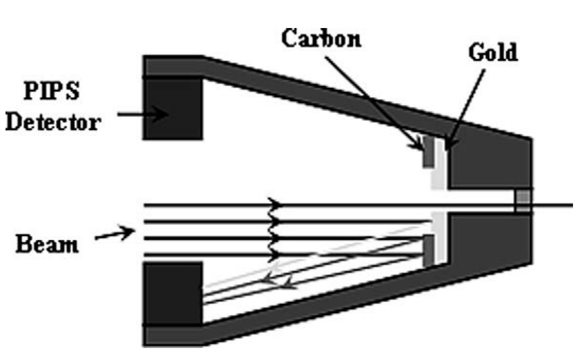

Fig. 1. Scheme of the extraction nozzle. The control of the fluence is here performed thanks to the detection of the back-scattered ions by the gold layer.

2.2. Thin standards

2.2.1. Comparison between high-energy alpha particle beams

In order to highlight the performance of high-energy alpha particle beams for the study of thin cultural heritage objects, the first experiments had been carried out to compare different beams of the same ion type which only differ by their energy.

In this work, we have compared alpha particle beams from 6 to 12 MeV on "Micromatter" [9] thin references. The samples represent the whole element range that could be found present in works of art, from Na to Bi.

Fig. 2 shows the comparison of the yield calculated thanks to the fit of the spectra with "Gupixwin" [10] for more than 40 different elements. This method was applied for all the results presented in this article.

We have directly observed an increase of the yield with the energy of the beam for all the elements. The production and ionization cross-sections are still increasing with the energy of the beam in this energy range and decreasing with the atomic number of the sample. This experimental result is in good agreement with the different theoretical cross-sections models.

But the energy of the beam could not be increased indefinitely. A limit needs to be defined. Indeed, if the yield is increasing with the energy, the noise background is also increasing. Then, as Fig. 3 shows, the limit of detection and the peak-to-noise ratio are limiting factors. Here, we have represented the yield divided by the (yield + noise) to have a maximum ratio equal to one in a perfect case, without any noise.

The noise has been also calculated with "Gupixwin" thanks to the relation, found in the Gupix literature, between the limit of detection calculated by the software and the noise background.

We have calculated a ratio very close to one for light elements ($13 < Z < 26$) from Al to Fe with the three different beams. On the other hand, we have noticed that for heavier elements the ratios differ between the different beams. For intermediate elements ($27 < Z < 33$), the ratio is between 0.6 and 0.95. If the 6 MeV beam directly showed lower results, it is impossible to define the best energy between the 9 and 12 MeV. There is then no advantage to increase too high the energy of the beam. On the other hand, for the heavy elements ($Z > 34$) the ratio still diminishes but the 12 MeV beam gives, with no contest, the best results.

The energy of the beam must then be adapted as a function of the elements present in the sample to have the best analysis of thin cultural heritage objects.

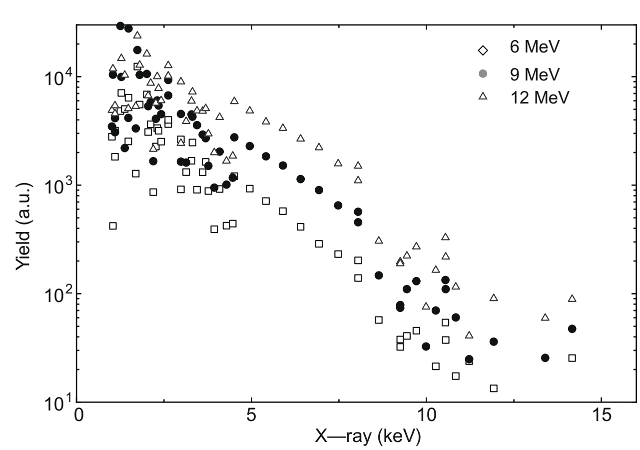

Fig. 2. Comparison of the yield for different X-ray energies obtained on PIXE spectra with 40 different elements from Na to Bi with high-energy alpha particle beams (6, 9 and 12 MeV) on thin "Micromatter" references.

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