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# External beam IBA set-up with large-area thin $Si_3N_4$ window

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ABSTRACT

A compact external beam setup has been constructed for Particle Induced X-ray Emission (PIXE) and Nuclear Reaction (NRA) analyses. The key issue in the design has been to obtain a wide beam spot size with maximized beam current utilizing a thin $Si_3N_4$ exit window. The employed specific exit window support enables use of foils with thickness of 100 nm for a beam spot size of 4 mm in diameter. The durable thin foil and the large beam spot size will be especially important for the complementary external beam NRA measurements. The path between the exit foil and sample is filled with flowing helium to minimize radiation hazard as well as energy loss and straggling, and to cool the samples. For sample-independent beam current monitoring and irradiation fluence measurement, indirect charge integration, based on secondary electron current measurement from a beam profilometer, is utilized.



## 1. Introduction

External ion beams have gained wide use especially in the analysis of objects related to arts and archeology due to clear advantages over conventional in-vacuum measurements [1]. This has often been the driving force for the development of external beam facilities.

Of the ion beam based techniques utilizing external beams Particle Induced X-ray Emission (PIXE) is the most common. However, in many cases also elemental depth profiling is preferred. As PIXE is not feasible for such determinations other ion beam based techniques should be utilized, e.g., Rutherford Backscattering Spectrometry (RBS) or Nuclear Reaction Analysis (NRA) [2]. Such measurements though require as thin as possible exit windows to guarantee minor energy straggling and thus good energy and depth resolution. In this respect the use of thin $Si_3N_4$ membranes as exit windows for ion beam analysis (IBA) set-ups has become more popular as even N-15 beams have been utilized successfully for hydrogen depth profiling via nuclear reactions [3]. The limiting factor has been the rather small beam size area in case of thinner foils and in case of larger area exit foils their thickness. For example, in case of 100 nm thick membrane the exit window diameter has been typically 1 mm [1,4] and in case of 3 mm diameter membrane the thickness has been of the order of 500 nm [5].

The aim of this work was to develop an external beam arrangement enabling measurements with good sensitivity and to construct and test an exit window support set-up allowing the use of thin 100 nm $Si_3N_4$ exit membranes providing a large beam spot size up to 4 mm in diameter on the sample. This enables use of higher total beam currents with still rather low beam current density improving detection sensitivity and reducing the measurement time and risk of beam induced sample damage. It also enables the analysis of a large area of the sample without the need to scan the beam over the sample. With large area averaging of composition of inhomogeneous samples, like historical potteries and ceramics, effect of the single mineral grains can be avoided. The concept should find usage especially in PIXE setups employing complementary external beam NRA measurements where high beam currents with minimum energy straggling are often required to achieve good sensitivity.

Direct beam dose measurement in external beam experiments is difficult due to the ionization of air molecules and due to the different conductivities of sample surfaces [6,7]. Here we present a method of beam dose estimation that overcomes these difficulties. The method is based on accurate current measurement from a beam profilometer (BPM) prior to the beam exit foil. The method is independent of sample conductivity and ionization issues. In this work, the method was successfully applied to external-beam PIXE analysis.

## 2. Experimental set-up

### 2.1. Beam exit system

The constructed experimental arrangement is shown in Fig. 1. To be able to analyze huge number of samples, we upgraded the

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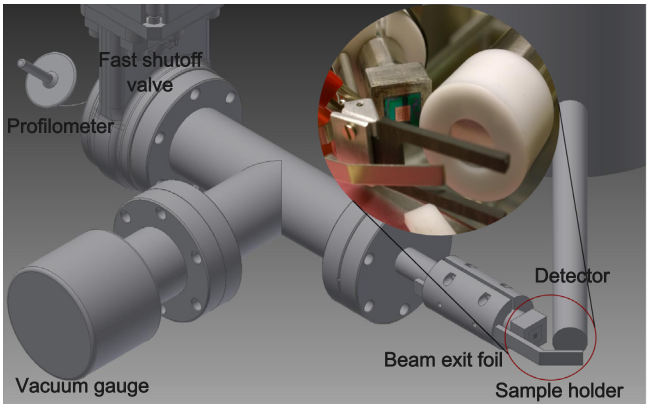


**Fig. 1.** General layout of the external beam set-up and a close-up of the beam extraction window.

external beam setup for fast measurements of historical ceramic samples by PIXE, to reduce measurement time and to improve the minimum detection limits of the technique. An important condition affecting the construction was to achieve a low a beam power per sample unit volume as possible. This can be achieved either by sample cooling and/or increasing the beam spot size on the sample. In the present configuration a flow of helium gas is used for sample cooling and the spot of the exposing beam is maximized by employing a special exit window support. The basic idea of the arrangement follows the lines realized in our earlier setups [8–10], with several improvements.

One of the main considerations in designing the present beam exit setup has been to achieve as low a background yield as possible for PIXE analyses. All components which can be in contact with the ion beam have been made of graphite. To minimize ion energy loss and straggling the space between the exit foil and the sample is filled with constantly flowing helium gas at atmospheric pressure. The helium atmosphere also aids elimination of possible hazardous radionuclides that can be produced if the space is filled with normal air.

Pneumatically actuated fast closing isolation valve was installed in order to protect accelerator vacuum against instantaneous vacuum failure in case the silicon nitride window is ruptured. Installed valve is a series 752 valve from VAT, capable of closing the valve blade within 10 ms.

### 2.2. Exit window

The main criterion in the selection of proper exit window material has been the minimum feasible foil thickness combined with the requirement of maximum beam spot size. In this respect $Si_3N_4$ membranes are the best choice. In the present system the beam spot size could be increased by the use of a carbon support under the exit window. The carbon support is perforated by $7 \times 7$ holes of 0.3 mm in diameter within an area of 10.9 mm$^2$. The effective beam spot area due to the carbon support is 3.5 mm$^2$.

Commercial $Si_3N_4$ membranes from Silson Ltd. with thicknesses of 30, 100, and 500 nm and with window area of $4 \times 4$ mm$^2$ were tested. The membrane has a supporting silicon frame. The membranes were glued from the frames to the aluminum which is surrounding the carbon piece supporting the membrane. Epoxy glue was used. Test results together with the energy loss and energy straggling for 3 MeV protons in various membranes are provided in Table 1.

The $Si_3N_4$ membrane was carefully placed on top of the carbon grid. Then each vertex of the supporting frame was carefully glued from top minimizing the amount of glue going under the frame. Tests were done both with the supporting carbon grid and without it. When the glue at the vertices had dried, the glue was applied on top of the sides of the frame. The thin 30 nm $Si_3N_4$ membranes broke during or prior to gluing due to the slight frictional forces present between the foil and the carbon grid. In cases they survived the gluing, they broke during the vacuum tests due to increased frictional forces between the membrane and the carbon grid caused by movement of the membrane due to the pressure difference. It was possible for a 100 nm thick window supported by the carbon grid to survive both gluing and vacuum tests. However this was relatively difficult to achieve and the finishing of the carbon grid and gluing the foil must be done with particular care. The 500 nm foil easily survived gluing and vacuum testing (both supported and not supported).

For routine PIXE measurements, the thicker foil is more robust to use and, compared to the proton energy loss of roughly 44 keV and straggling of 13 keV in 20 mm of 1-bar He [11], the energy loss is not significantly larger in a 500 nm window than in a 100 nm window. Concerning the PIXE method such energy loss and straggling values are insignificant. For these reasons, a 500 nm foil was used for the current integration study discussed in the following section. We decided to use the graphite grid support for the foil in order to minimize the magnitude of the possible pressure pulse to the vacuum in case of membrane rupture and to minimize the bending of the foil and the resulting uneven energy loss.

To sum up, tension tests of the $Si_3N_4$ foils were affected by rough edges of the graphite support and small particles between membrane and support. Foil thicknesses from 100 nm and up can be used as extraction windows with graphite grid support. The use of the thin 100 nm foil enables complementary measurements with external beam NRA [12] with the same setup as discussed in more detail in Section 2.4.

### 2.3. Beam current normalization

Numerous different arrangements for beam current monitoring during external beam measurements have been reported in the literature [1,6]. In our configuration the beam current monitoring is carried out with two methods. In the first method the current is measured from the sample holder. In the second method the current is estimated by measuring the real-time secondary electron current from a Beam Profile Monitor (BPM), manufactured by National Electrostatics Corporation.

**Table 1**
Results for the tested ($4 \times 4$ mm$^2$) $Si_3N_4$ windows. The corresponding calculated energy loss and straggling values are provided for 3 MeV protons.

| Foil thickness [nm] | Supported | Result | Energy loss [keV] | Straggling FWHM [keV] |
|---|---|---|---|---|
| 30 | No | Breaks during assembly or vacuum testing | 0.96 | 1.23 |
| 30 | Yes | Breaks during assembly or vacuum testing | 0.96 | 1.23 |
| 100 | No | Breaks during vacuum testing | 3.16 | 3.17 |
| 100 | Yes | Holds | 3.16 | 3.17 |
| 500 | No | Holds | 15.89 | 8.64 |
| 500 | Yes | Holds | 15.89 | 8.64 |