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# MeV-SIMS yield measurements using a Si-PIN diode as a primary ion current counter



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BEAM INTERACTIONS WITH MATERIALS AND ATOMS

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### ABSTRACT

Megaelectronvolt-Secondary Ion Mass Spectrometry (MeV-SIMS) is an emerging Ion Beam Analysis technique for molecular speciation and submicron imaging. Various setups have been constructed in the recent years. Still a systematic investigation on the dependence of MeV-SIMS yields on different ion beam parameters is missing. A reliable measurement method of the beam current down to the attoampere range is needed for this investigation. Therefore, a new detector has been added to the MeV-SIMS setup at the Ruđer Bošković Institute (RBI), which measures the current directly using a Si PIN-diode. In this work, we present the constructed system, its characteristics, and results of the first yield measurements. These measurements have already identified important factors that have to be considered while constructing a MeV SIMS setup.

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#### 1. Introduction

Megaelectronvolt-Secondary Ion Mass Spectrometry (MeV-SIMS) employs charged particles (primary ions) with a kinetic energy in the MeV range in order to eject particles (called secondary ions if charged) from a sample surface. For ions with a kinetic energy in that range, the dominant stopping mechanism is electronic stopping, leading to the increased desorption of high mass molecules [1–5]. As the electronic stopping power depends on the impinging ion's energy, charge and atomic number, a study of the secondary ion yield (secondary ions detected divided by the primary ions applied to the sample) as a function of these parameters is essential. Earlier works by Hakansson et al. [6] and Albers et al. [7] have shown strong yield dependence on these experimental parameters for lower mass sputtering and a similar dependence is likely for sputtering high mass molecules as well.

An example of the strong yield dependence on the electronic stopping can be seen in the comparison of two spectra from the amino acid Glycine (m = 76.04 Da) using 10 MeV Cl<sup>5+</sup> and 11 MeV O<sup>4+</sup> primary ions (Fig. 1). The absolute yield per primary ion hitting the sample achieved by the heavier chlorine ions compared to the oxygen primary ions is about 6 times higher even though kinetic energies are similar. This shows the necessity to investigate the

dependency of the secondary ion yields on primary ion characteristics. The results could be used to optimize the MeV-SIMS setup, in order to achieve the best possible efficiency. Also, a deeper understanding of the secondary ion sputtering process can be gained from a yield database spanning over a wide range of primary ion types and energies. This may also offer the future promise of quantitative MeV-SIMS.

The accuracy of any yield measurement depends on the precise and continuous measurement of the primary ion beam current. The method to measure this current and applications of this current measurement approach will be described in the following section.

## 2. Materials and methods

A description of the RBI MeV SIMS setup can be found elsewhere [8].

Any investigation of the yield dependence on different experimental factors requires a precise measurement of the primary ion current. As the current is in the attoampere range and the beam should be monitored continuously, current measurement through conventional indirect techniques like Rutherford Backscattering Spectrometry (RBS) or direct current measurement in a Faraday cup cannot be applied. Moreover, direct measurement of the primary ion fluence by a particle detector behind the sample, (i.e. Scanning Transmission Ion Microscopy (STIM) detector, only works for thin samples transparent to the primary ions.

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**Fig. 1.** Comparison of the Glycine yield achieved using chlorine and oxygen primary ions with similar energies. The yield value on the *y*-axis is the yield for the RBI setup determined using the system described here.

Taking all this into account, we decided to use a system that consists of a detector that intercepts the beam periodically. That concept showed to be the most reliable, reproducible and versatile way to control the current of primary ions impinging on the sample in the target chamber, while not altering the beam characteristics or restricting the application to a specific type of sample.

The schematic drawing of the setup installed on our microbeam line is shown in Fig. 2. The realization of such a system for ion beam current measurement utilizes three main parts: a detector for the incoming ions (beam current monitoring diode), a motor to move the detector in and out from the beam and a control mechanism for the motor.

In order to detect the incoming ions, a suitable detector with 100% detection efficiency and good radiation hardness is required. A Hamamatsu S1223-01 Si-PIN photodiode was chosen. Its low cost and wide availability makes it easily replaceable if damage due to excessive ion bombardment significantly degrades its performance.

This monitoring diode is mounted on a holder driven by a stepper motor, which allows for easy programmable movement between the two positions. Since the Hamamatsu S1223-01 is light, small and can be mounted close to the motor axis, almost any stepper motor can be selected. The only important factor is the torque the motor can provide, as it has to move the diode in and out of the microbeam axis in a very short time period.

The system is placed in the microbeam line to intercept the beam axis immediately after the collimator slits (defining typically a  $1 \times 1 \text{ mm}^2$  beam area) and prior to the scanning coils and focus-

ing quadrupole triplet (Fig. 3). With its sensitive area of  $3.6 \times 3.6 \text{ mm}^2$ , this diode (when well aligned) has 100% efficiency for all ions that enter the microprobe focusing system. The stepper motor is mounted outside the beam line, and connected to the diode holder by means of a rotary vacuum feedthrough. This vacuum feedthrough is particular simple and inexpensive, it consists of a simple radial shaft seal. Still no degradation of the vacuum could be measured along the beamline and tests have shown that vacuum can be maintained at  $10^{-7}$  mbar. Influence of the possible stray magnetic fields (i.e. from the stepper motor coils) that may degrade microbeam focusing could not be detected.

The last part of the setup consists of the motor controller and the driver. They control the motors movement and provide the power necessary for the motor to run. We have selected a simple solution based on the Arduino Uno microcontroller together with a Big Easy stepper motor driver.

The alignment of the monitoring diode in the beamline is done by monitoring the area burnt by the beam spot on a piece of paper which is placed at the diodes position. After observing the beam shape at that particular position, the diode is mounted in a way to cover the entire beam spot at that position. The whole system is controlled by a set of switches, which are used to monitor the amplitude and periodicity of the diode movement. These switches can also be used to choose different operation modes, which include the complete manual control of the diode (used to optimize beam current), or to switch to a second operation mode that uses an aluminum plate with a thin gold layer mounted on the diode holder to measure high currents indirectly using the RBS signal from this target (e.g. with a proton current of  $\approx 100$  pA for PIXE and RBS).

#### 2.1. Calibration process

During a typical MeV-SIMS measurement using the described setup, the monitoring diode measures the ion current for 0.5 s, then moves out of the beam and waits for 0.5 s before rotating back into the beam. As 280 ms are needed for the movement in and for the movement out of the beam, this time has to be added for a total measurement cycle period of 1.28 s. Hence, the time the monitoring diode measures the beam is not equal to the time the beam hits the sample. This, in addition with other possible effects like the beam shape and position on the Si-PIN diode as well as transmission losses along the way through the ion lenses (the distance monitoring diode and sample holder is  $820 \pm 20$  mm), makes it necessary to determine a calibration factor that relates the number of ions that actually hit the sample.



Fig. 2. Closer look at the diode in the position out of the beam with the important parts indicated. A good source for stepper-rmotors and their wiring is www. schmalzhaus.com.

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