Oxy-nitrides characterization with a new ERD-TOF system

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Abstract

A new time-of-flight (TOF) camera was installed on Elastic Recoil Detection (ERD) measurement setup on the Tandem Accelerator at Université de Montréal. The camera consists of two timing detectors, developed and built by the Jyvaskyla group, that use a thin carbon foil and microchannel plates (MCP) to produce the start and stop signals. The position of the first detector is fixed at 18 cm from the target, while the position of the second detector can be varied between 50 and 90 cm from the first detector. This allows to increase time resolution by increasing the distance between the time-of-flight detectors or to increase solid angle by decreasing the distance. Moving the detector also helps determining the parameters such as the effective distance between detector foils and the delay time.

Compared to the original system, which had only one timing detector and relied on a surface barrier energy detector to measure both the energy and the stop signal, in the new system the energy is now obtained from the timing signal with much better resolution, reaching ~4 nm near the surface. There is also no more need to keep track of the surface barrier detector calibration for each element, including the pulse height defect.

We show examples of quantitative depth profiles of oxy-nitride layers that has been characterized with this new system. It allows quantitative depth profiling of targets that would be difficult to analyze with other techniques, especially when light elements such as hydrogen, carbon, or nitrogen are mixed in various proportions in a heavy element matrix.

Keywords: Elastic Recoil Detection, Ion beam analysis, ERD-TOF, ToF-ERDA

1. Introduction

Elastic Recoil Detection by Time-Of-Flight Analysis (ERD-TOF) is a very useful technique for quantitative thin film depth profiling of several elements in a single measurement. Its main advantage over Rutherford Backscattering Spectrometry (RBS) is that it provides a good sensitivity for light

elements, such as hydrogen, carbon, nitrogen and oxygen, even in matrices containing heavy elements. The fact that ToF-ERD measures both the energy and the velocity for every detected atom it enables to separate each individual element from the spectra. This eliminates the problem of the overlapping of the different elements in RBS.

The first ERD-TOF camera was developed by Groleau *et al.* at Université de Montréal in the early eighties [1]. This first camera consisted of a carbon foil combined with a microchannel plate as the start detector and the timing signal of a solid-state detector as the stop detector. Using this setup, they were able to obtain a timing resolution of 250 ps for 5.48 MeV alpha particles.

In this paper we present a new TOF camera for the ERD-TOF line at Université de Montréal. The time-of-flight unit consists of two detectors that use a thin carbon membrane and microchannel plates (MCP) to produce the start and stop signals. The time is now used to measure the energy with excellent resolution. We have reached 4 nm near-surface resolution, compared to about 8 nm with the old system. We show examples of quantitative depth profiles of oxy-nitride layers obtained with this new system.

2. Experimental setup

The schematic of the ERD-TOF setup is shown in Figure 1. As seen in the figure, the distance between the two timing units can be varied from 50 to 90 cm. This has two purposes. First, it can be used to tweak the depth accuracy and the solid angle. Increasing the length of the camera increases time of flight (and thus timing accuracy) and decreases the solid angle (down to 0.107 mSr), which also reduces the geometrical straggling due to the finite size of the detector. We observed depth resolutions differences of the order of 5% when increasing the camera length from 50 to 90 cm. Inversely, solid angle can be increased up to 0.273 mSr by decreasing the length of the camera. Second it is used to accurately measure the time-of-flight calibration parameters by varying the length of the camera. For instance an alpha radioactive source can be measured at different lengths to establish the energy as a function of time-of-

flight relation.

The timing units used in this new TOF camera were designed and built by the Jyväskylä group [2]. The main components of the timing units are schematically shown in Figure 2: (1) the 10 µg/cm^2 carbon foils, which emits and accelerates the electrons when an ion passes through it, (2) the "toblerone", which forms electric field-free volume for the electrons, (3) the electrostatic mirror, which reflects the electrons towards the base of the unit, (4) the MCP plates, which multiplies the electrons, and (5) the anode, which collects the electron signal. The two timing units are built differently. For the stop signal timing unit, the carbon foil and mirror are at -2800 V while the toblerone and MCP stack are at -1800 V. For the start signal timing unit, the carbon foil is grounded in order to avoid unwanted extra acceleration of the outgoing charged recoils if their charge state changes when passing through the foil, which would lead to a broadened energy distribution. The energy of the recoils is measured by a passivated implanted planar silicon (PIPS) detector. Another PIPS detector, placed at 16° from the TOF camera (46° scattering angle) at 15 cm from the sample surface, in combination with a stopping foil is used to monitor hydrogen as the detection efficiency of the TOF camera is low for the hydrogen.

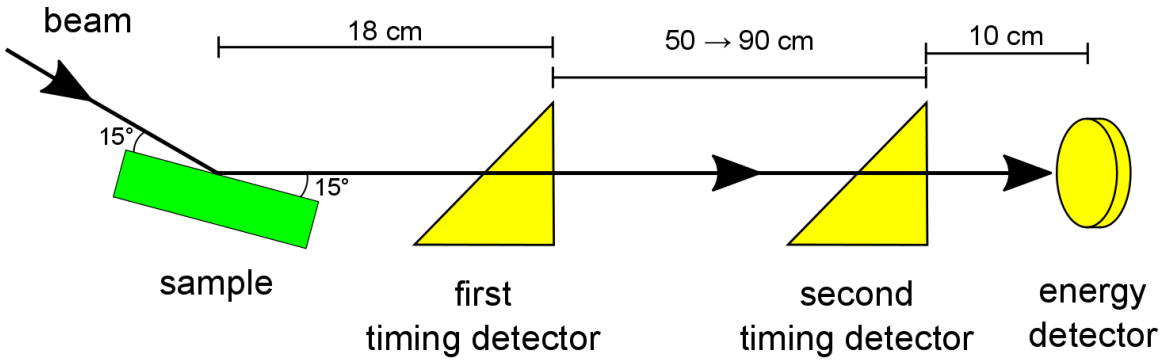

Figure 1. Schematics of the ERD-TOF setup. The scattering angle is 30° and the first timing detector is at 18 cm from the sample. The distance between the first and second timing detectors can be varied between 50 and 90 cm and the distance between the second timing detector and the energy detector is 10 cm.

Figure 3 shows the timing resolution of the TOF camera measured for 5.5 MeV alpha particles from

an Am-241 α-source. The first timing unit was at 20 cm from the source and the distance between the two timing units was 25 cm. A Gaussian fit of the data gives a full-width at half-maximum of 218 ps. The analysis presented in the next section was carried out with a 50 MeV Cu^{9+} beam provided by the 6 MV Tandem accelerator at Université de Montréal.

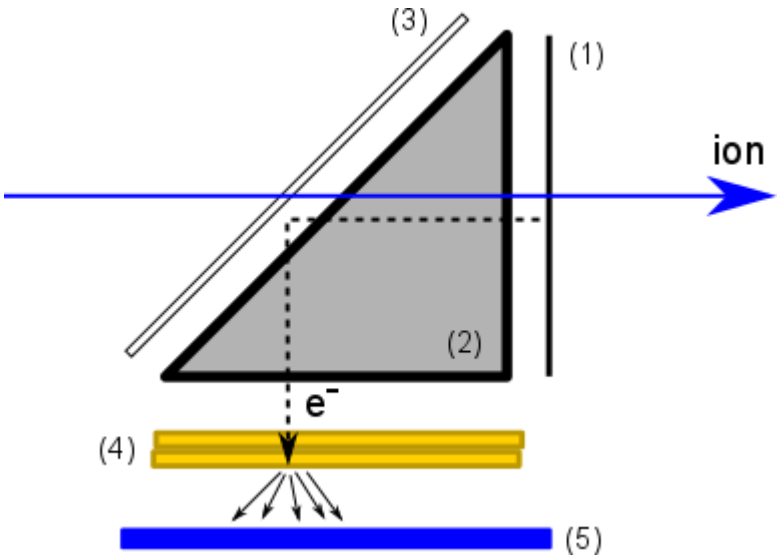

Figure 2. Schematical representation of a timing unit. (1) carbon membrane (2) toblerone (3) electrostatic mirror (4) MCP stack (5) collecting anode. When an ion passes through the carbon membrane, electrons (e^-) are emitted and accelerated by the toblerone. They are then deflected by the electrostatic mirror towards the MCP stack. The MCP stack multiplies the electrons, which are finally collected by the anode.

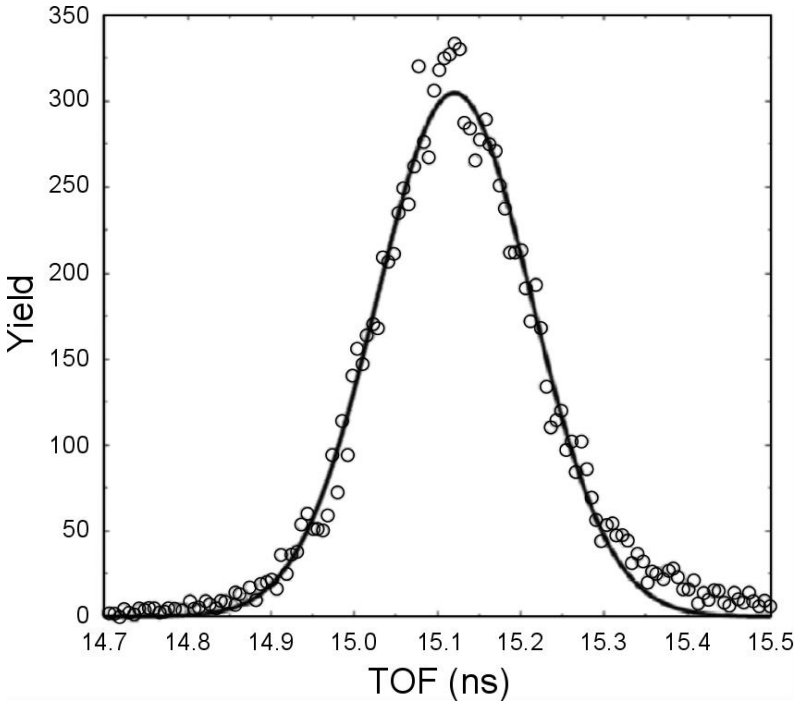

Figure 3. Timing resolution of the TOF camera. A Gaussian fit of the data gives a full-width at half-maximum of 218 ps.

3. Results

A SiONV(~50 nm) / SiON(~200 nm) layer on a silicon substrate was analyzed with a 50 MeV Cu^{9+} beam. The distance between the timing units was 79 cm and the solid angle was 0.13 mSr. Figure 4 shows the events plotted as time-of-flight vs. energy. About 1,000,000 co-incidence events were detected with ToF and E detectors, including the events related to the scattered Cu beam. One can see from the Fig. 4 that the background noise is mostly due to pile-up, i.e. more than one atom entering the detection system at the same time. Since scattering of the ion beam forms the majority of the events, a pile-up event is more likely to include a scattered ion, forming a vertical stripe above the "Cu beam" in the Figure 4. It is also seen that the stripes have a trapezoidal shape, especially near the high energy edge, where the bottom edge is sharp. This is due to the good timing resolution compared to the relatively poor energy resolution of the PIPS detectors, especially with heavy ions, which produces a relatively wide stripe.

The corresponding atomic concentration as a function of depth, were calculated iteratively from the

elemental energy spectra with the Allegria software [3]. Those depth profiles are shown in Figure 5. The hydrogen profile is obtained with the traditional ERD detector with a 10.16 μm mylar foil. In these profiles, we can identify a thin oxide layer at the surface, which is 2-3 nm thick. The full width at half maximum of the corresponding peak can be used to estimate the depth resolution, which was found to be around 4 nm for this measurement. Following this very thin oxide layer is the SiNV layer whose composition is found to be $Si_{0.37}O_{0.03}N_{0.43}V_{0.14}H_{0.13}$. Before the silicon substrate is the SiON layer, which composition was found to be $Si_{0.29}O_{0.55}N_{0.04}H_{0.12}$. These results would have been very difficult to obtain with traditional RBS as there was less than 5 % of the nitrogen in the matrix of the SiONV layer.

4. Summary

The ERD-TOF setup at Université de Montréal has been upgraded with a new TOF camera. This has led to an improvement in the depth resolution achieved by the system. The next steps to improve the system resolution will be to use heavier elements for the probing beam, which have higher stopping powers. Lower beam energies will also be used to improve hydrogen detection efficiency because the hydrogen stopping power in the carbon foils will increase with decreasing energy.

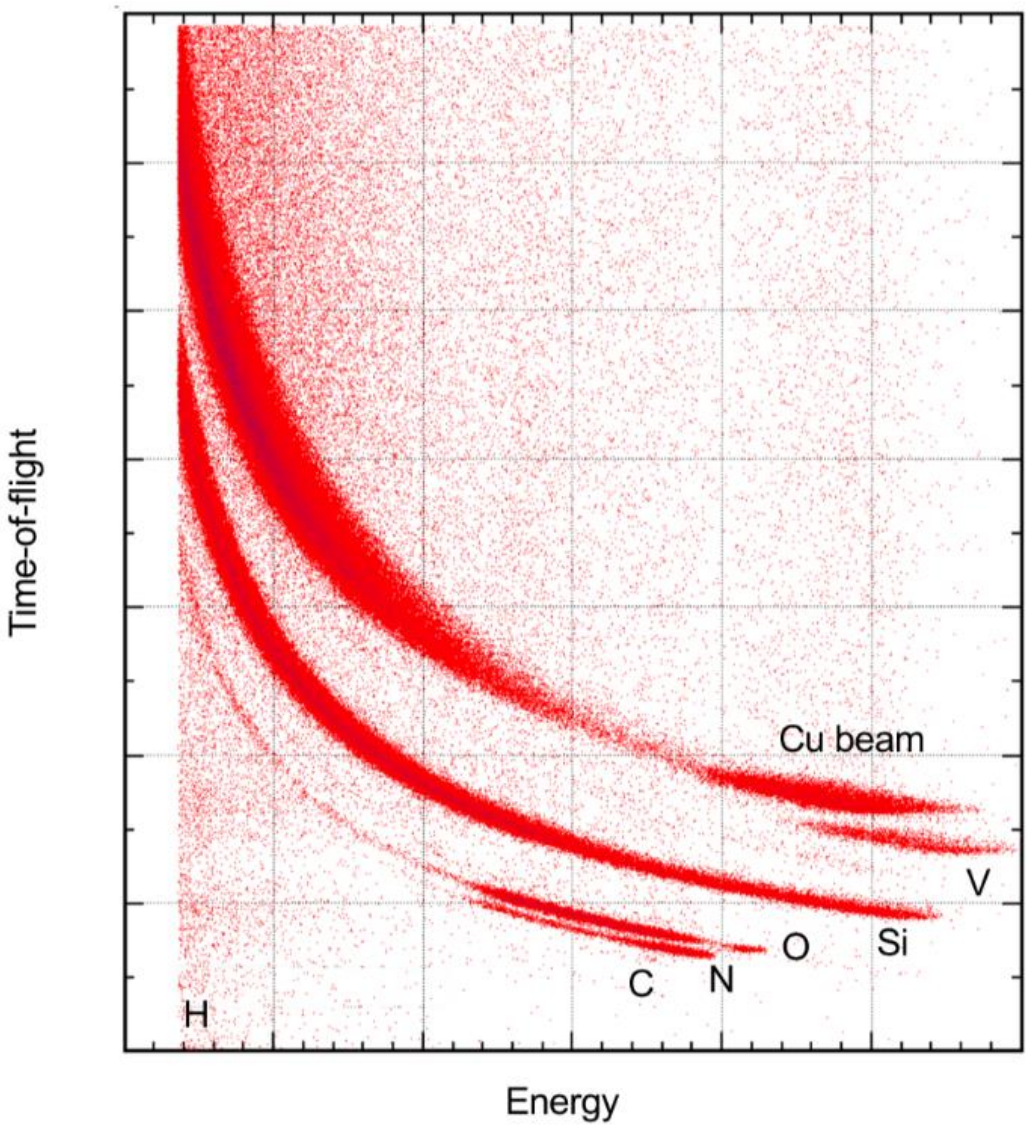

Figure 4. ERD-TOF events from a SiNV/SiON/Si sample probed by a 50 MeV Cu beam plotted as Time-of-Flight vs energy. Traces from Cu, V, Si, O, N, C and H can be seen.

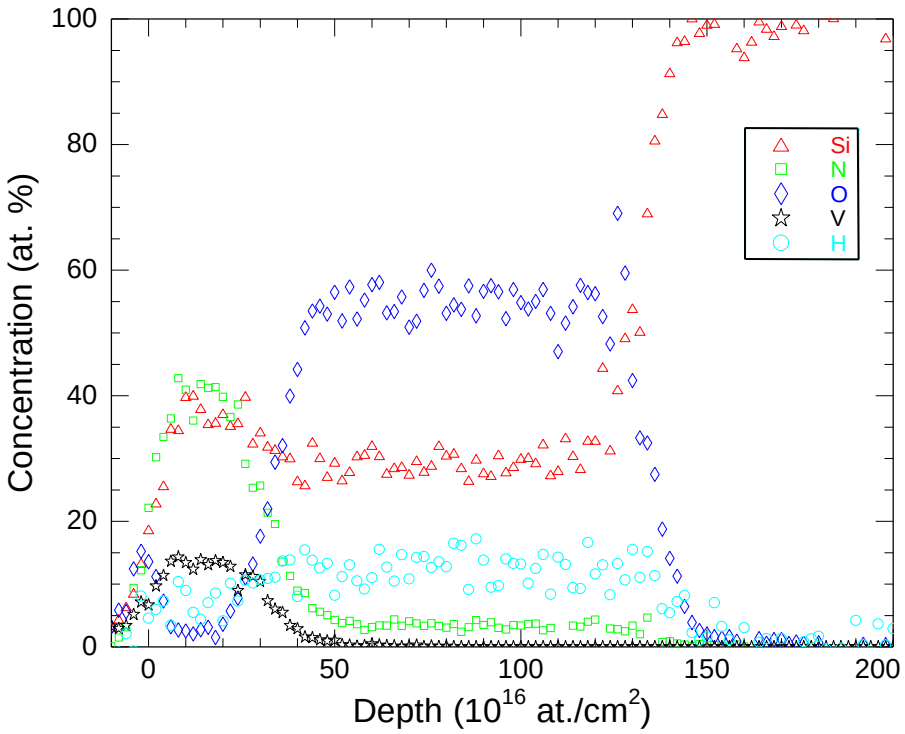

Figure 5. Atomic concentration vs depth for same sample as in Figure 4.

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References

[1] R. Groleau, S.C. Gujrathi, J.-P. Martin, Nucl. Instr. and Meth 218 (1983) 11.

[2] M. Laitinen, M. Rossi, J. Julin, Timo Sajavaara, Nucl. Instr. and Meth. in Phys. Res. B 336 (2014) 55.

[3] F. Schiettekatte, M. Chicoine, S. Gujrathi, P. Wei , K. Oxorn, Nucl. Instr. and Meth. in Phys. Res. B 219-220 (2004) 125.