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Nuclear Instruments and Methods in Physics Research B 249 (2006) 915-917

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# The CIBA high resolution RBS facility

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Available online 30 May 2006

#### Abstract

Recently, a high resolution RBS facility was installed at the Centre for Ion Beam Applications (CIBA) at the National University of Singapore (NUS). The magnetic spectrometer comprises of a 90° double focusing sector magnet and a 2D-MCP focal plane detector. The UHV scattering chamber is equipped with a five axis goniometer for channeling applications, the setup is similar to that used by the Kyoto group [K. Kimura, M. Kimura, Y. Mori, M. Maehara, H. Fukuyama, AIP Conf. Proc. 475 (1999) 500]. State of the art performance characteristics were expected, because the system operates in conjunction with the NUS Singletron accelerator, which provides ion beams with very favorable phase space characteristics. Well resolved spectra of 10 nm SiO<sub>2</sub>/Si are shown, and an energy resolution of 0.9 keV FWHM was measured at the leading Si edge of a SiO<sub>2</sub> sample at 300 keV and 80° scattering angle, which is close to the value expected from kinematic broadening alone in this case (~0.7 keV).

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PACS: 68.55.-a

Keywords: High resolution RBS; Magnetic spectrometer

# 1. Introduction

Technological advances in microelectronics, optoelectronics and photonics rely to a large extent on the use of precisely manufactured thin film structures. Because of the recent advent of nanotechnology as a rapidly increasing research field (with typical dimensions in the 1-100 monolayer range), the ability to fully and nondestructively characterize such structures is critical for further development. RBS, with alpha particles as analytical probes, is the most versatile technique available for the nondestructive and quantitative depth-profiling of thin films, if high atomic mass films/structures on lighter substrates are concerned. However, the depth resolution of conventional RBS is limited by the energy resolution of the Si detectors used. In normal incidence, with e.g. a 1 MeV alpha beam, the depth resolution for Au is around 10 nm. The use of grazing incidence geometries improves this, but the increase in small

angle scattering results in an increase in the path length effect [11]. This, together with limitations in the attainable geometry, typical limits the depth resolution to around 3 nm. Possible ways to improve on this number include the utilization of heavy ions with the associated large stopping powers or the development of detector systems with high relative energy resolution ( $\Delta E/E$ ). The IBA community has worked for many years on both approaches, and over the last few years it has become clear that sub-nanometer depth resolution is becoming a critical necessity. For light ions, several approaches to increase energy resolution are possible, among them time of flight spectrometers [2], electrostatic analyzers, and magnetic spectrometers [3–9]. The use of a magnetic spectrometer can improve the depth resolution by an order of magnitude or more, allowing monolayer depth resolution for RBS and even for ERDA in certain circumstances [10,11]. Very sophisticated spectrometers have been built, mostly for work in the MeV region, however the Kyoto group of Kimura has taken a somewhat different approach and used a compact 90° sector magnet to built a small system for

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<sup>0168-583</sup>X/\$ - see front matter @ 2006 Elsevier B.V. All rights reserved. doi:10.1016/j.nimb.2006.03.198

 ${\sim}500~{\rm keV}$  alpha scattering, utilizing the stopping power maximum and increased cross sections in this region.

At the CIBA [12] an extensive program of collaborative and consultative IBA work with research institutes and local industry is ongoing for a decade, and in order to be able to satisfy the increasing demand for ultrathin film analysis, a HRBS500 magnetic spectrometer system manufactured by Kobelco Steel [13], was recently installed at the 45° beamline. The endstation is operated in conjunction with the CIBA Singletron 3 MV accelerator [14], that provides light ion beams with very favorable phase space characteristics, i.e. a large brightness  $(20 \text{ pA}/(\mu m^2 \text{ mrad}^2 \text{ MeV}))$ coupled with and low energy instability (20 eV peak to peak at 1.88 MeV), This high beam energy stability minimizes possible contributions to the system energy resolution from energy variations. Here we report on the characterization of the new facility, and demonstrate the capabilities of the spectrometer.

#### 2. The HRBS spectrometer

The HRBS endstation, sketched in Fig. 1, is based around a UHV scattering chamber with a base pressure below  $5 \times 10^{-9}$  bar. A computer controlled five axis goniometer enables sample translations in x, y and z as well as rotations up to 90° and 30° around vertical and horizontal axes, respectively. The spectrometer is similar to the system described in [1], with a relatively simple  $90^{\circ}$  sector magnet with 26.6° inclined boundaries for double focusing, with a bending radius of 175 mm and a mass energy product  $(mE/q^2)$  of 2.1 MeV. A 100 mm long and 15 mm high z-stack multichannel plate (MCP) with a resistive anode readout is used as a position sensitive detector in the focal plane of the spectrometer. The exit boundary is modified from a straight line to rotate the focal plane to be perpendicular to the central exit trajectory, in order to avoid the rapid changes seen in the MCP efficiency that occur for oblique incidence angles. This is shown in Fig. 1, where the trajectories of particles with  $\varepsilon = E/E_0 = 0.88$ , 1 and 1.15 are indicated for the case of a geometry with a solid angle of 0.4 msr.



Fig. 1. Sketch of the CIBA high resolution RBS endstation, the median plane trajectories of particles with  $\varepsilon = E/E_0 = 0.88$ , 1 and 1.15 are indicated.

The magnet chamber is mounted on rails and can be rotated around the scattering chamber so that scattering angles of 30°, 50°, 65°, 80°, 95°, 110° and 125° can be arranged, allowing for both backscattering and forward recoil measurements. A load lock system allows rapid sample changes in the UHV chamber.

#### 2.1. Calibration measurements

A set of 16 measurements were carried out using a thin (<1 nm) HfO/Si sample and a 300 keV alpha beam  $(I \sim 20 \text{ nA}, \text{ with scattering angle } \varphi = 80^{\circ} \text{ and beam to target-normal angle } \theta \text{ of } 60^{\circ}$ ). A range of *B* field settings are used to sweet the Hf peak over the MCP surface. Such a *B*-field sweep is equivalent to an energy calibration, because the trajectories of ions with energy *E* are identical to those with energy  $E_0$  (assumed to represent the central trajectory at  $B_0$ ) if the *B* field is varied according to  $(B/B_0)^{1/2}$ . Fig. 2 shows the Hf peaks, normalized to the integrated charge, of nine of the resulting raw position spectra. The surface (high channel) edges are clearly sharper then the (low



Fig. 2. Position spectra of thin HfO/Si samples, taken at B field values as indicated, details see main text.



Fig. 3. Comparison of leading edge positions with calculations from matrix theory and the SIMION simulation code.



Fig. 4. HRBS spectra of 10 nm SiO<sub>2</sub>/Si, the full lines are RUMP simulations. The Si and O surface edges are indicated.

energy) HfO/Si inner interface edges. These spectra were fitted with double error functions to extract the positions of the leading edges on the MCP surface. Fig. 3 then compares these fitted positions  $X((B_0/B)^2) = X(E/E_0) = X(\varepsilon)$ with values calculated from first order matrix theory [15] for a straight edge magnet (assuming the fringe field extends half a pole gap) and also with simulations using the ray tracing software SIMION [16] which models both the fringe field and the curved exit boundary. The magnet works as designed, as reasonable agreement is seen for the SIMION calculations, and even the matrix calculation is not far off.

The extraction of energy spectra from the raw position spectra is a more complex process than in conventional RBS. The energy/position relationship is not strictly linear and the energy spectra f(E) need to be calculated from the position spectra  $F(\varepsilon)$  according to [1]:

$$f(E) = F(X(E/E_0)) \frac{1}{E_0} \frac{\mathrm{d}X}{\mathrm{d}\varepsilon} \Big|_{\varepsilon = E/E_0}$$

Fig. 4 shows two spectra taken from a SiO<sub>2</sub>/Si thin film standard sample [17], with a certified thickness of 10.8(2) nm. A 300 keV  $\alpha$  beam and a scattering angle of  $\varphi = 66^{\circ}$  were used, with the target normal set at  $\theta = 55^{\circ}$ and  $\theta = 35^{\circ}$  with respect to the beam. A RUMP [18] simulation fits the data reasonably well, except for the region of channel 80–100 in the  $\theta = 55^{\circ}$  spectrum, where some residual channeling is seen. The thickness of the SiO<sub>2</sub> film assumed in the simulations is 10.5 nm. The Si surface edges indicate an energy resolution of 1.2 keV, a value that includes effects from possible surface contaminations of the sample. The best resolution observed so far, 900 eV for a Si surface edge, is rather close the purely kinematic broadening expected due to the change of the kinematic factor over the solid angle subtended by the spectrometer. This amounted to  $\sim$ 700 eV in this case, and so the performance of the spectrometer is as expected.

## 3. Conclusions

We have installed and tested a double focusing 90° sector magnet spectrometer at the 45° beamline at the Singletron accelerator at CIBA, and the system performs as expected. The high resolution RBS facility with sub 1 keV energy resolution is now operational. Our further plans include a number of applied projects utilizing the system, among them the analysis ultrathin high k dielectrics, thin strained Si structures and ultrathin silicides (NiSi, CoSi<sub>2</sub>). We also plan to couple the scattering chamber with a MBE growth chamber in order to allow in situ analysis.

## References

- K. Kimura, M. Kimura, Y. Mori, M. Maehara, H. Fukuyama, AIP Conf. Proc. 475 (1999) 500.
- [2] M. Döbeli, P.C. Haubert, R.P. Livi, S.J. Spicklemire, D.L. Wheathers, T.A. Tombrello, Nucl. Instr. and Meth. B 47 (1990) 148.
- [3] W.A. Lanford, B. Anderberg, H. Enge, B. Hjorvarsson, Nucl. Instr. and Meth. B 136 (1998) 1177.
- [4] J. Vrijmoeth, P.M. Zagwijn, J.W.M. Frenken, J.F. van der Veen, Phys. Rev. Lett. 67 (1991) 1134.
- [5] K. Kimura, K. Ohshima, M. Mannami, Appl. Phys. Lett. 64 (1994) 2232.
- [6] T. Enders, M. Rilli, H.D. Carstanjen, Nucl. Instr. and Meth. B 64 (1992) 817.
- [7] O. Kruse, H.D. Carstanjen, Nucl. Instr. and Meth. B 89 (1994) 191.
- [8] W.M. Arnoldbik, W. Wolfswinkel, D.K. Inia, V.G.G. Verleun, S. Lobner, J.A. Reinders, F. Labohm, D.O. Boerma, Nucl. Instr. and Meth. B 118 (1996) 567.
- [9] R. Grötzschel, Ch. Klein, O. Kruse, Nucl. Instr. and Meth. B 183 (2001) 3.
- [10] K. Kimura, K. Ohshima, K. Nakajima, Y. Fujii, M. Mannami, H.-J. Gossmann, Nucl. Instr. and Meth. B 99 (1995) 472.
- [11] G. Dollinger, C.M. Frey, A. Bergmaier, T. Faestermann, Nucl. Instr. and Meth. B 136–138 (1998) 603.
- [12] F. Watt, J.A. Van Kan, I. Rajta, A.A. Bettiol, T.F. Choo, M.B.H. Breese, T. Osipowicz, Nucl. Instr. and Meth. B 210 (2003) 14.
- [13] KOBE STEEL LTD., Machinery & Engineering Company, Kobe, Japan.
- [14] D.J.W. Mous, R.G. Haitsma, T. Butz, R.H. Flagmeyer, D. Lehmann, J. Vogt, Nucl. Instr. and Meth. B 130 (1997) 31.
- [15] S. Penner, Rev. Sci. Instr. 32 (1961) 150.
- [16] SIMION 7, Scientific Instrument Services, 1027 Old York Rd, Ringoes, NJ 08551.
- [17] CRM 564, Community Bureau of Reference. Available from: <a href="http://www.irmm.jrc.be">http://www.irmm.jrc.be</a>>.
- [18] L.R. Doolittle, Nucl. Instr. and Meth. B 9 (1985) 344.