	Experiment title: **High-resolution measurements of x-ray RRS of Si and Al and their oxides**	**Experiment number**: HE-1615
Beamline: ID21	**Date of experiment**: from: 14.4.04 to: 20.4.04	**Date of report**: 25.8.04
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Report:

Introduction

The application of the total reflection x-ray fluorescence (TXRF) method [1] combined with intense synchrotron x-ray sources offers new possibilities for measuring very low concentrations of light element impurities on the surface of silicon wafers. Several experiments [2, 3, 4] have shown that the detection limit of aluminium impurities on the silicon surface is limited by the presence of the x-ray resonant Raman scattering (RRS). For excitation energies tuned below the Si K-absorption edge to avoid the intense Si K fluorescence x-ray line the RRS structure of silicon is indeed overlapping with the Al K x-ray peak. In the analysis of low-resolution data a precise knowledge of the background profile that is mainly due to the x-ray Raman scattering is therefore crucial.

Experiment

The resonant x-ray Raman scattering spectra of Al, Si and Al_2O_3 and SiO_2 were measured at the ESRF beam line ID21 by means of high resolution x-ray spectroscopy, using a von Hamos Bragg-type curved crystal spectrometer [5]. As the Bragg angle domain covered by this instrument extends from 24.4° to 61.1°, for the observation of the 1400 – 1800 eV x-ray spectra of interest the spectrometer was equipped with an ADP (101) crystal (2d = 10.642 Å). The diffracted x-rays were recorded with a 26.8 mm long and 8 mm high position-sensitive back illuminated CCD detector consisting of 1340 columns and 400 rows with a pixel size of 20×20 μm^2. At ID21 the spectrometer was installed downstream of the STXM microscope chamber to which it was connected through a ~200 cm long evacuated pipe (Fig. 1).

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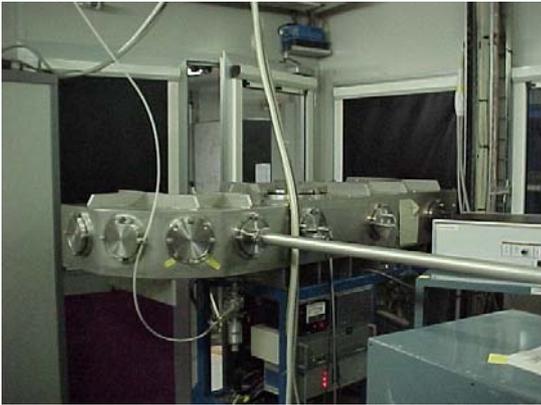
Fig. 1: Photograph of the von Hamos spectrometer as installed at the ESRF beam line ID21.
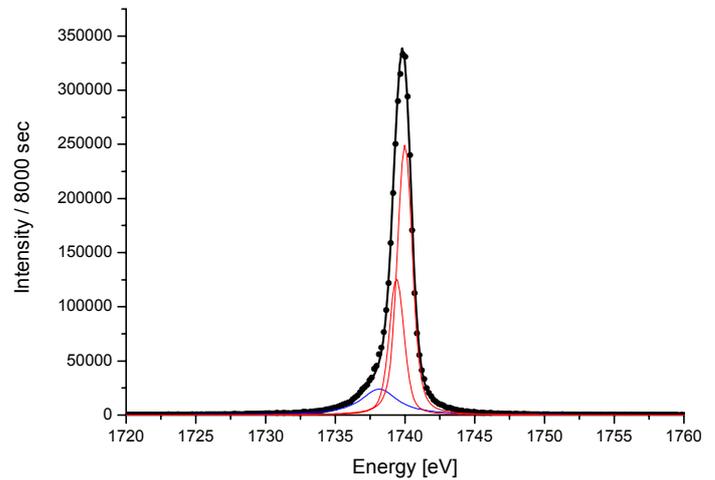

Fig. 2: High-resolution Kα$_{1,2}$ x-ray spectrum of Si. The dots correspond to the experimental data, the black line to the total fit of the spectrum.

The x-ray beam delivered by the wiggler was monochromatized by means of two 20 Å Ni/B$_4$C multilayers. Residual photons above 4 keV were suppressed with a Ni mirror. Thanks to this monochromator $10^{12} – 10^{13}$ incident photons/s were obtained on the samples with an energy resolution of ~10 eV. For all measurements the angle between the incoming beam and the sample surface was 20°. Solid targets consisting of 25 mm high × 12 mm wide × 1 mm thick sheets were used. The specified purity of the samples was better than 99.99% for Al and Si and 97.5% and 99.9%, respectively, for Al$_2$O$_3$ and SiO$_2$.

The energy calibration of the spectrometer and the determination of the instrumental response were performed by measuring the Kα x-ray lines (2p-1s transitions) of Al and Si. For illustration the Si Kα spectrum observed at an excitation energy of 1895 eV is presented in Fig. 2. The two diagram lines and associated unresolved M-satellites originating from shake processes in the M-shell as a result of the 1s photoionization were fitted each with a single Voigt function (red curves). A third Voigt profile (blue line) was used to account for the asymmetric response of the ADP crystal.

Data analysis and results
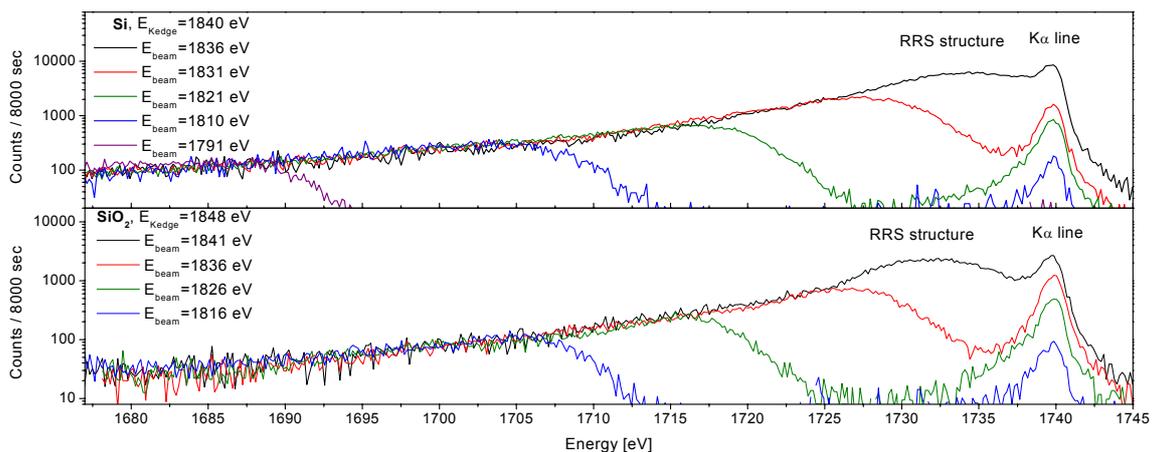
Fig. 3: high-resolution RRS spectra of Si and SiO$_2$ for different excitation energies

Data were taken at several excitation energies between 1790 eV and 1900 eV for Si and SiO$_2$ (see Fig. 3), respectively between 1540 eV and 1600 eV for Al and Al$_2$O$_3$. The beam energy calibration was based on measurements of the K-absorption edges of Al and Si, using the edge energy values reported in [6]. For each target the intensities of the different spectra were normalized off-line for the number of incident photons and acquisition time and corrected for the beam intensity profile on the sample.

The RRS x-ray spectra were analyzed by means of the least-squares-fit program PeakFit (SYSTAT Software Inc. products, Richmond, CA 94904-2028, USA), employing several juxtaposed Pearson IV functions to reproduce the shape of the observed structures. From the fitted spectra the evolution of the integrated intensity of the RRS structure was then determined as a function of the excitation energy. For illustration results obtained for Si and SiO_2 are presented below (Fig. 4).
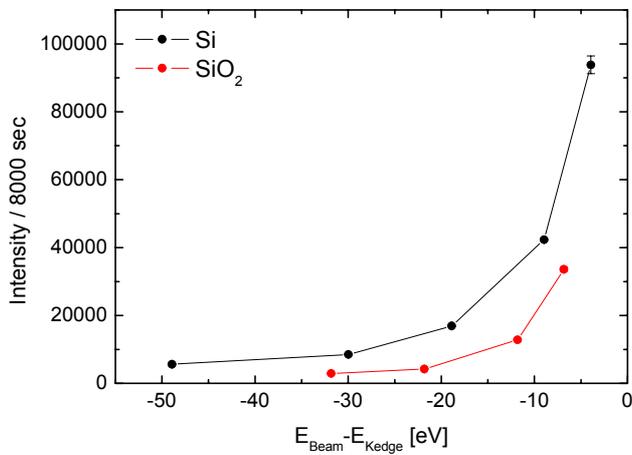

Fig. 4: Integrated intensity of the RRS structure of Si and SiO_2 as a function of the excitation energy.

The measured RRS x-ray spectra of Si, SiO_2, Al and Al_2O_3 will be compared (work in progress) with theoretical predictions from the nonrelativistic dipole approximation (NDA) calculations of Gavrila [7]. In this way the NDA approach, which is used for modeling the RRS features in the TXRF spectra [3], can be tested accurately, using present high-resolution data. In addition, we plan to investigate the influence of solid state and chemical effects by comparing the RRS structures measured for elemental targets and their oxides.

References

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