A.6: Soft x-ray absorption measurement on Indus-2 soft x-ray reflectivity beamline using x-ray fluorescence signal

Soft x-ray absorption spectroscopy (SXAS) is a powerful tool to analyze the chemical and electronic properties of materials. For the absorption measurements, one needs the ratio of transmitted beam to the incident photon beam intensity. In the soft x-ray region (< 2000 eV), the transmission measurements are very challenging because they require ultrathin freestanding samples (< 50 nm thickness), which are difficult to produce. Therefore, the SXAS measurements in the soft x-ray region are performed in indirect mode by measuring the total electron yield (TEY) or x-ray fluorescence signal. The TEY technique is surface sensitive, as the electron signal is collected from the top within few nanometer thickness (2-3 nm). The resultant absorption data are affected by surface contamination, if any. The SXAS signal collected in fluorescence mode has higher depth sensitivity (> 100 nm) as the penetration depth of the soft x-ray photons is relatively large, and therefore the fluorescence based technique is useful to probe the bulk properties. Charge neutrality is another major advantage of the fluorescence based x-ray absorption measurement technique. Thus, non-conducting samples can also be measured easily.

At Indus-2 soft x-ray reflectivity beamline, BL-03, a soft x-ray fluorescence (SXF) based x-ray absorption measurement setup is designed, installed, and commissioned. A separate vacuum chamber is installed in downstream of the existing reflectometer station with a vacuum isolation valve. A separate vacuum pumping arrangement is made for the SXF chamber. At a time four samples of ~10×10 mm^2 size can be mounted. The SXF chamber operates in ~1×10^{-6} mbar vacuum. A vacuum compatible energy dispersive silicon drift detector (SDD) is used for soft x-ray fluorescence measurement.

In order to protect the detector from the exposure to ambient while sample loading/unloading, the detector is mounted on a retractable linear movement feedthrough and kept in a detector vessel, isolated with a vacuum valve. The detector can be brought close to the sample during the measurement. Along with mechanical vacuum assembly, a LabView based instrument control and data acquisition software is also developed. The software is used to control the detector and the beamline monochromator together so that the energy dependent x-ray absorption measurements can be performed. The SXAS measurements in the fluorescence mode are successfully performed on various samples. Figure A.6.1 shows the SDD detector based SXAS setup installed in downstream of the reflectometer station. Figure A.6.2 shows a measured SXAS spectra of CrN and Cr thin film sample using the nitrogen *K* emission line. The setup can be used for elemental identification of different low *Z* elements, e.g., carbon, nitrogen, oxygen, etc., which are otherwise not feasible in the hard x-ray based fluorescence measurement setup operating in air environment.

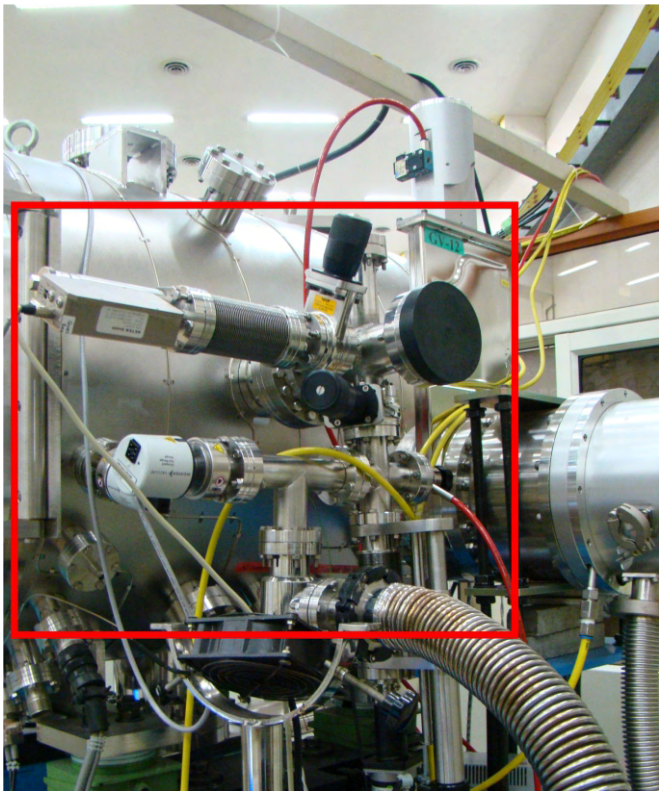

Fig. A.6.1: Soft x-ray fluorescence based absorption measurement setup shown in the red marked area is installed in the downstream of the reflectometer station.

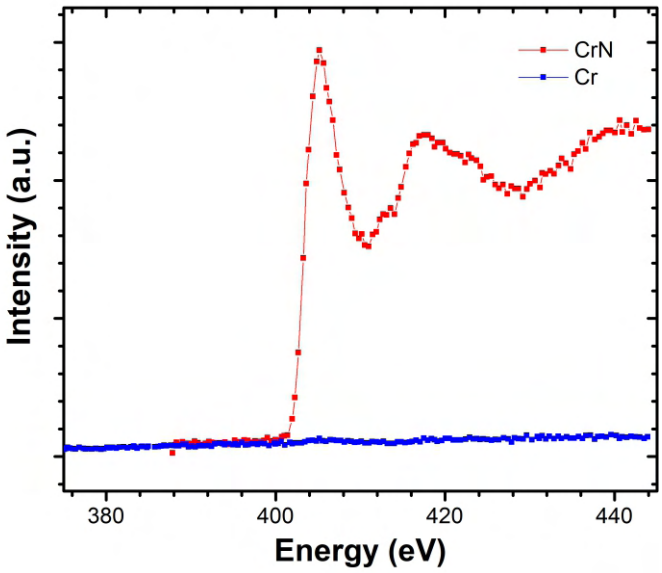

Fig. A.6.2: SXAS spectra of CrN and Cr thin film samples measured using the nitrogen K fluorescence signal.

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