A.3: Synchrotron induced total reflection X-ray fluorescence measurements on Indus-2

Total reflection X-ray fluorescence (TXRF) spectrometry is a well-established technique for elemental analysis of solid and liquid materials at the parts per billion (ppb) levels. The technique finds several applications in a variety of fields (viz. geology, archaeology, biomedical science and material science etc.). The TXRF spectrometry is one of the variations of the EDXRF method in which the specimen is excited by the primary x-ray beam at a glancing angle less than the critical angle at which total external reflection occurs, and secondly the specimen is placed on a plane polished surface. The grazing incidence mode of excitation almost completely eliminates the large Compton scattering of the primary x-ray beam from the sample bulk, which usually limits the detection sensitivities in the conventional EDXRF method.

Now-a-days, from the application point of view, TXRF spectrometry has become quite popular in semiconductor industry for analysing trace and ultra-trace elemental impurities in semiconductor devices and wafers, introduced during manufacture procedure. Even, several synchrotron radiation based TXRF facilities have been built, for industrial utilization of the technique in semiconductor technology. The present state-of-the-art is the elemental sensitivities in the TXRF method has been obtained in femtogram range by employing synchrotron radiation as an excitation source. In addition to trace elemental analysis, the TXRF technique also finds applications for surface and near-surface layer characterization. The grazing incidence X-ray fluorescence (GIXRF) technique has the potential of being one of the most powerful and versatile methods for characterization of layered materials because it combines features of both x-ray reflectivity and x-ray fluorescence. GIXRF enables the non-destructive determination of layer thickness, density, interface roughness and depth profiling for an element inside a layer structure.

Considering several advantages of a synchrotron induced total reflection fluorescence measurements we have setup a TXRF experimental station on the microfocus x-ray fluorescence beamline (BL-16) of Indus-2 synchrotron radiation facility. The TXRF experimental station comprises of a 2-circle goniometer installed on a vibration free optics table. To measure specular reflected x-ray intensity from a flat substrate, a scintillator detector has been installed on the exit arm of the 2-circle goniometer. Figure A.3.1 shows the photographic view of the TXRF setup installed on the BL-16 beamline.

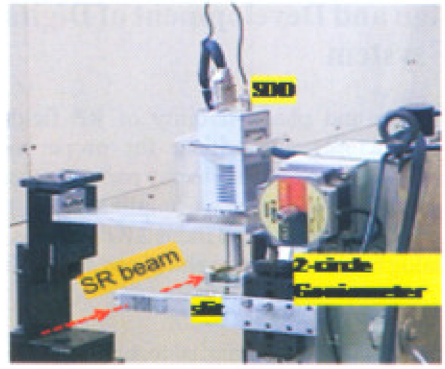

Fig. A.3.1: Photograph of the BL-16 TXRF experimental station.

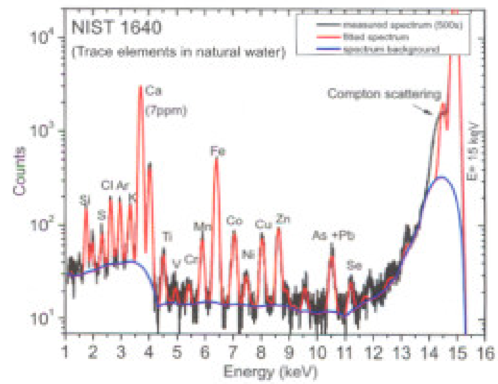

Fig. A.3.2: TXRF spectrum of a NIST 610 (trace elements in natural water) measured at incidence x-ray energy of 15 keV.

To validate the TXRF methodology a standard reference material (NIST-1640: trace elements in natural water) has been analyzed. Figure A.3.2 depicts a representative TXRF spectrum of the NIST 1640 sample measured using collimated synchrotron x-ray energy of 15 keV. The TXRF spectrum was recorded for acquisition time 300 s. Almost all trace elements have been detected in the NIST sample. The detection sensitivities obtained for various elements (Ca - U) were found to be in the range of sub ppb to ~ 20 ppb.

In summary, a synchrotron based TXRF spectrometer has been installed and commissioned on the micro focus x-ray fluorescence (BL-16) beamline Indus-2 synchrotron source. High incident photon flux for excitation, wide energy tenability and very high detection sensitivities make the SR-TXRF technique a valuable tool for ultra-trace element analysis. It is now operational and available for the users.

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