A.8: Improvement of elemental detection sensitivities in XRF technique by using synchrotron source

X-ray fluorescence (XRF) spectroscopy is a powerful non-destructive technique for elemental analysis of solid and liquid materials at micro and trace levels.

In a conventional laboratory based XRF technique, the element detection sensitivities are largely limited to the mg g^{-1} (ppm) range due to the large spectral background produced by the Compton scattered x-rays from the specimen. The total reflection x-ray fluorescence (TXRF) is another variant of the energy dispersive-XRF (EDXRF) technique, where the complexity of the large spectral background is eliminated to a great extent owing to the high reflectivity on the flat surface and low penetration depth of the primary x-ray beam in the substrate material, on which the incident x-rays are allowed to impinge at glancing incidence angles. All these features improve the detection sensitivities of the TXRF technique to ~ 2-3 orders of magnitude or better compared to the conventional XRF, typically in the range of parts per billion (ppb) levels for most of the elements.

Another approach to reduce the spectral background in x-ray fluorescence measurements is to utilize linearly polarized primary radiation for the sample excitation. Based on the classical dipole oscillator approach, it is well understood that the emission profile of scattered radiation is anisotropic in the case of a polarized incident x-ray beam. Maximal signal to noise ratio can be realized if the fluorescence signal is measured in a position, where the contribution of anisotropic scattered radiation is minimal.

In a recent work, we have shown that how polarization properties of the synchrotron radiation can help in limiting the large spectral background in the total reflection x-ray fluorescence (TXRF) technique. For details please refer; Das G. et al., J. Anal. At. Spectrom., 29, 2405-2413, 2014. The experiment was conducted on the BL-16 beamline of Indus-2 synchrotron facility to study anisotropic nature of the scattered synchrotron photons. The anisotropic distribution profile of the scattered radiation imposes a constraint to place an x-ray spectroscopy detector in a condition where the contribution of the scattered background is minimal. The experimental TXRF measurements performed on the standard reference materials and for a few TXRF methodological samples showed that the horizontal detection geometry provides approximately one order of magnitude better signal to background ratio, in contrast to the conventional vertical detection geometry, thus improving TXRF detection sensitivities to ~ 20-60% for most of the elements. The Fig. A.8.1 illustrates a typical geometrical configuration used for the TXRF measurements on the BL-16 beamline. The Fig. A.8.2 shows the calculated scattering probability density for the Compton scattered x-rays emitted from a specimen as a function of scattering angle θ and azimuthal angle ϕ assuming an incident x-ray energy of 15 keV. The computation results closely corroborate with the experimental TXRF measurements performed on the standard reference materials.

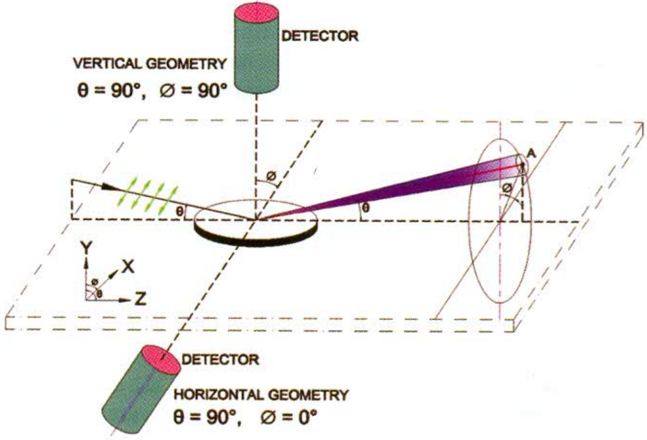

Fig. A.8.1: Experimental setup used for the angle dependent X-ray fluorescence measurements. The spectroscopy detector is placed in the vertical geometry (i.e., θ= π/2, ϕ = π/2) as well as the horizontal geometry (i.e., θ= π/2, ϕ = 0) to collect the XRF spectra.

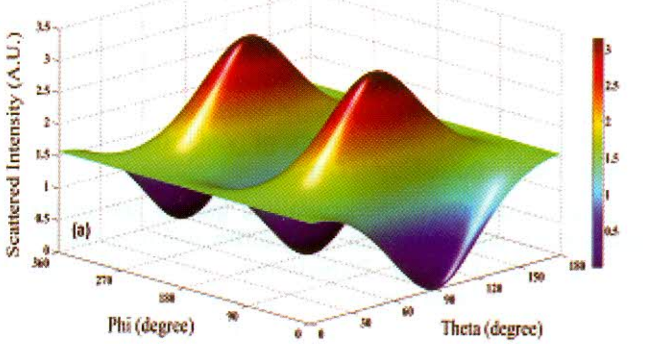

Fig. A.8.2: Angular distribution of the Compton scattered photons as a function of scattering angle (θ) and azimuthal angle (ϕ) at an incident x-ray energy of 15 keV.

The results reported in the present work provide a general guideline for mounting a spectroscopy x-ray detector, especially, for the synchrotron based grazing incidence X-ray fluorescence and total reflection X-ray fluorescence measurements to achieve better signal to spectral background contrast.

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