Surface XAFS Using a Total-Electron-Yield Detector

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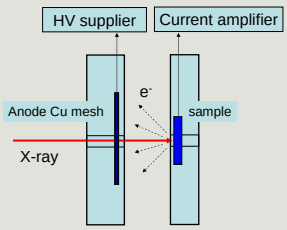
Introduction

When a beam of X-rays of a few keV impinges upon matter, it produces secondary emissions including X-ray fluorescence, primary, Auger and secondary electrons. By tuning the energy of the incident synchrotron X-ray beam and measuring the intensity of total-electron-yield (TEY) as a function of X-ray energy, the X-ray absorption fine structure spectra (XAFS) at the absorption edge of a specific element can be collected, due to the fact that the collected TEY is closely associated with the X-ray absorption coefficient of the element.

A simple detector using this principle was designed at the XDD beamline with photon energy range of 2.5-10 keV at the *Singapore Synchrotron Light Source* (SSLS). Compared with conventional XAFS using transmission and fluorescence mode, the XAFS in TEY mode is a surface sensitive technique and it works efficiently for thick and concentrated specimens that cannot be measured by other modes of XAFS, moreover, it also suffers less from the self-absorption effect as in the fluorescence mode.

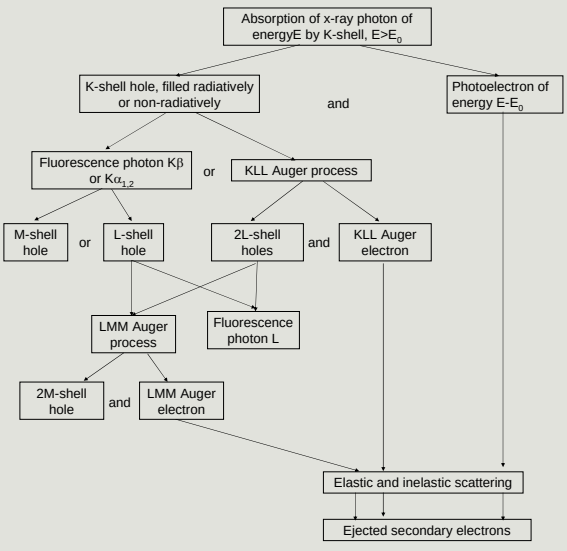
Mechanical Design

A graph showing the total-electron-yield detector consisting of two parts, a Cu mesh as anode and sample sitting on the aluminum plate, the outer shell is stainless steel



Layout of the TEY detector, a high voltage is applied to the anode, sample current is measured by a current amplifier

Methodology



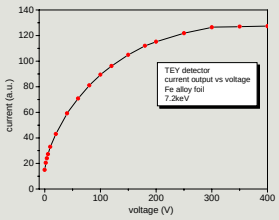
A figure showing the non-radiative or radiative decaying process due to the absorption of X-rays of energy E, which is related to processes in the total-electron-yield current production.

The sampling depth for total-electron-yield XAFS is determined mainly by the effective penetration range R_p of the Auger electrons, which is the distance over which they travel and deposit their excess energy. Experimental results of R_p on initial electron energy E of 1-10 keV for various materials can be approximated by:

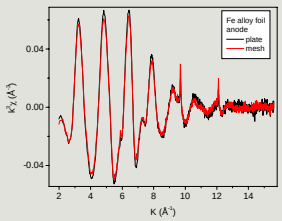
$$R_p \approx 1000E^{1.4}/\rho$$

Where R_p in angstroms, and E in keV, ρ is the material's density in g/cm^3

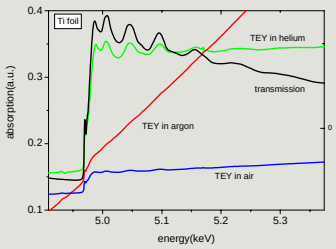
Performance test



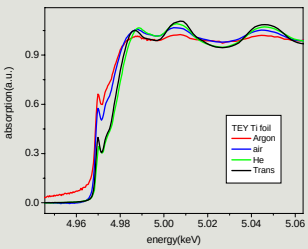
Sample current as a function of applied voltage, measured in a Fe-alloy foil at X-rays of 7.2keV. The current approaches to saturation at 300V.



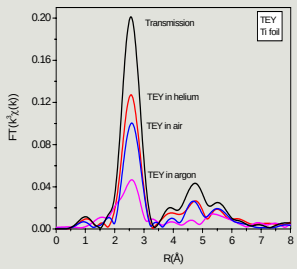
XAFS spectra of Fe-alloy foil (Fe K-edge) measured at two kinds of anodes, respectively: aluminum plate and copper mesh, the XAFS shows close amplitudes



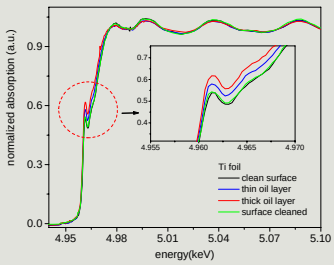
X-ray absorption spectra of titanium foil (Ti K-edge) measured under various ambient filled in TEY detector: helium, air and argon, compared to spectrum measured in transmission mode.



Normalized X-ray absorption spectra of titanium foil (Ti K-edge) measured at various ambient and compared to transmission mode.

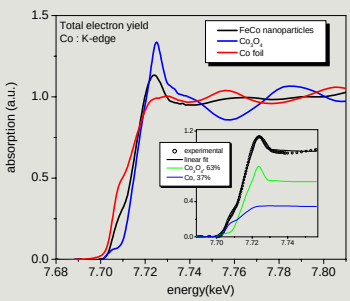


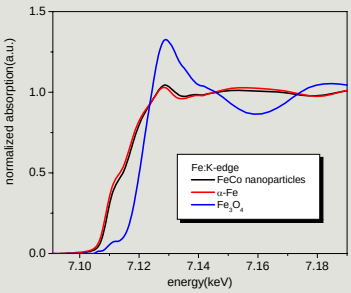
A comparison of Fourier transformed spectra of titanium foil at various ambient and transmission mode. The spectra of TEY mode show reduced amplitudes.



Normalized X-ray absorption spectra of titanium foil (Ti K-edge): clean surface, surface contaminated with a thin and a thick layer of pump oil, and oil-cleaned surface, the shoulders enlarged in the inset show the fine structure.

Fe-Co alloy nanoparticles





XAFS spectra obtained from the ball-milled nanoparticles of Fe-Co alloy investigated by XAFS in the total-electron-yield mode. By comparing with standard references and a linear fit, it can be determined that 63% of Co in FeCo nanoparticles are oxidized. In a same comparison with standard Fe-containing compounds, the XAFS spectrum at the Fe K-edge looks similar to that of Fe foil, indicating that Co is found at the surface of nanoparticles, partially oxidized due to exposure to air, while Fe most likely sit in the core part of nanoparticles.

Conclusion

The performance test shows that this technique is very sensitive to the surface status and the ambient filled in the detector. Using XAFS in total-electron-yield mode it is expected to obtain elemental information such as valence state, local symmetry, spatial configuration of atoms, specifically at the surface or near surface. It is being equipped at the XDD beamline of Singapore Synchrotron Light Source for users to do XAFS experiments.