X-ray Absorption Spectroscopy

X-ray absorption spectroscopy (XAS) techniques probe short- and medium-range order, yielding information on bond lengths, coordination numbers, local coordination geometry and the oxidation state of atoms for a wide range of solid and liquid systems. XAS experiments require an intense, tunable photon source only available at synchrotrons.

Features

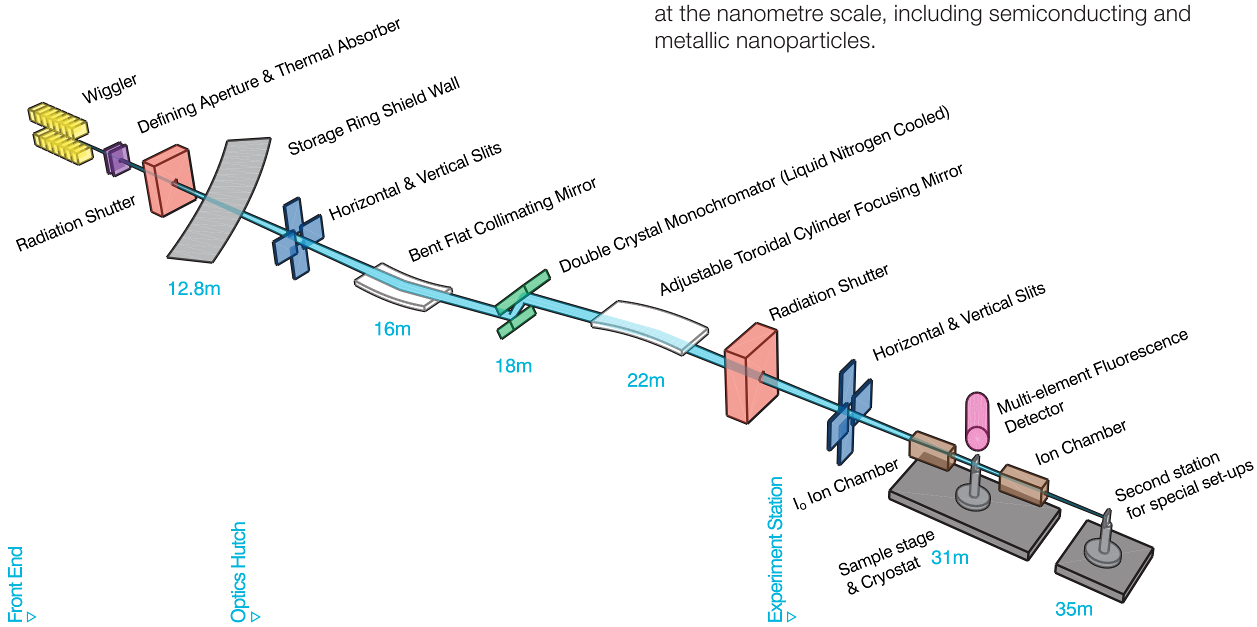
- medium and high energy (from atomic number Z=20 upwards) XAS, XANES (x-ray absorption near-edge structure), XAFS (extended x-ray absorption fine structure) and XES (x-ray emission spectroscopy)
- bright and highly stable scanned monochromatic photon beam covering a large energy range
- accessible elements are Ca to Nd (K edge) and Pm to U (L edge with $k > 15Å^{-1}$)
- the EXAFS region of an XAS spectrum provides structural information such as bond length, coordination number and disorder
- the XANES region provides chemical information such as local coordination geometry and oxidation state
- fast-scanning XAS can be used to develop methodologies for determining the structures of intermediates in biochemically-important enzymatic processes.

Applications

Around the world, XAS beamlines are in high demand for applications in the biological, chemical, earth, environmental, materials and physical sciences and engineering. The technique complements protein crystallography studies, and the two are frequently used in combination to determine challenging structures. Widely used by both specialists and non-specialists, XAS is a mature technology that is also enabling the advancement of new areas of science.

Examples

- identification of therapeutic target sites, such as the metal-binding sites that may be responsible for some of the pathological effects of Alzheimer's disease
- elucidation of the chemistry associated with enzyme catalysis, particularly in relation to the influence of redox or charge state on the electronic and molecular structure of transition metal complexes
- studies of metal complexes and mechanisms associated with the formation of ore metal deposits (Cu, Au, Ag) to assist prediction of economically important deposits
- characterisation of ion-implantation-induced disorder in semiconductor substrates, with application to electronic and photonic device fabrication
- investigation of novel properties exhibited by materials at the nanometre scale, including semiconducting and metallic nanoparticles.



Case study 1

Non-steroidal Cu anti-inflammatories

Technology from Sydney University with Biochemical Veterinary Research/Nature Vet and the University of Western Sydney involves new non-steroidal anti-inflammatory formulations that use copper complexation to minimise side-effects caused by the active ingredient, indomethacin. The researchers, led by Prof Peter Lay, significantly improved the copper-indomethacin formulations after using synchrotron techniques to determine the core structure and stability in formulations and investigate its transformation in cells. XAS data were collected on Cu Indo complexes. One complex was subsequently used in new formulations although crystals could not be obtained for XRD structure determination. The structure was therefore determined by XAFS analyses, validated against the structure of related Cu complexes for which the XRD structure was known.

Case study 2

Soil contamination from groundwater

Dr Peter Kappen and colleagues from La Trobe University and Environmental Resources Management Australia are using XAS to study chromium species in contaminated soils. Cr contamination problems can arise as a result of previous industrial practices. The problem is usually addressed by adding a reductant to the soil to transform highly toxic and soluble Cr(VI) into relatively insoluble and non-toxic Cr(III). Results show how soil takes up Cr from contaminated water. The next step will be to monitor the Cr speciation during remediation treatment. The work will assist the development of more efficient remediation strategies.

Case study 3

Amorphous metal nanocrystals

Dr Mark Ridgway from the Australian National University and his international colleagues are studying the fundamental processes that govern the formation and modification of atomic-scale nanocrystalline structures. Their findings include the identification of subtle structural differences relative to bulk material and the surprising discovery that nanocrystalline metals can be rendered amorphous by ion irradiation, an established technique for modifying bulk-phase materials. The transformation to an amorphous form is impossible with bulk metals and contrary to theoretical predictions.

Amorphous metals lack long-range order and crystalline defects, so they are stronger, harder, tougher and more elastic. Applications for such nanocrystalline-form metals include optical switches (photonics) and chemical catalysts.

Beamline specifications

Source	1.9T wiggler
Available energy range	4->50 keV
Optimal energy range	4 – 22 keV focussed with Si(111) 5.5 – 37 keV focussed with Si(331) 4 – 45 keV unfocussed
Resolution deltaE/E	Crystal dependent: Si(111) ~ 1.5×10^{-4} Si(311) ~ 4×10^{-5} Si(333) reflection from Si(111)
Nominal beam size at sample (horizontal x vertical)	Variable 5 (h) x 1 (v) mm to 0.5 (h) x 0.2 (v) mm
Photon flux at sample	> 5×10^{12} photons / second (4 – 22 keV) > 1×10^{11} photons / second using Si(311)
Harmonic content at 5-20 keV	< 10^{-5}
Techniques available	phase 1: XANES, EXAFS etc by transmission phase 2: fluorescence and secondary x-ray spectroscopy

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