XAS

X-ray beamline dedicated for absorption measurements

Technology:

XANES and EXAFS measurements in transmission and fluorescence mode

- *Quick-XAFS*
- *Grazing incidence XAFS*
- *Energy range from 2.4 (Sulphur K edge) to 27 keV (Cd K-edge).*
- *Typical beam size is 8 mm (hor) x 1 mm (ver), range 1 x 1 mm to 20 x 2 mm*
- *Flux: Si (111): 6.0x10^{9} ph/s/mm^{2} @ 9 keV (measured at average current 100 mA)*

Equipment:

Double Crystal Monochromator Si(111) and Si(311), fluorescence and transmission detection.
Large experimental hutch with 0.5t crane for in-situ measurements.
Special chamber for low energy measurements, e.g. Sulphur

Category:
C. Particle Characterization in- and ex-situ

Institute:
Karlsruhe Institute of Technology (KIT)

Location:
Karlsruhe Institute of Technology
Institute for Synchrotron Radiation (ISS)
Building 348
Hermann-v.-Helmholtz-Platz 1
D-76344 Eggenstein-Leopoldshafen
Germany

Contact Details of Technology Expert:

Name: Dr. Stefan Mangold; Dr David Batchelor
Phone: +49 721 608-26073
Fax: +49 721 608-26789
E-mail: stefan.mangold@kit.edu; david.batchelor@kit.edu

Short technology description/Overview *(approx 300 words)*:

XAS is an X-ray absorption spectroscopy (XAS) beamline on a dipole magnet source. Due to the high flux the beamline is suited for highly diluted systems in bulk samples. XAS provides the local atomic geometry (XAFS) together with electronic structure information (XANES). The information is element specific and not limited to crystalline material i.e. it is complementary to X-Ray diffraction. Distances up to 6 Å with a resolution of ± 0.02 Å together with coordination number can be determined. Besides standard XAFS measured in transmission (detection limit ~5%) and fluorescence (detection limit 1mmol/L) modes the beamline offers Q-XAFS (Quick XAFS) allowing scans as quick as 30 seconds. Grazing incidence XAFS is also possible providing surface sensitivity in the 50 nm range. Cryogenically cooled measurements are also possible.

At present the beamline focuses on questions in nanomaterials, catalytic systems and environmental science.

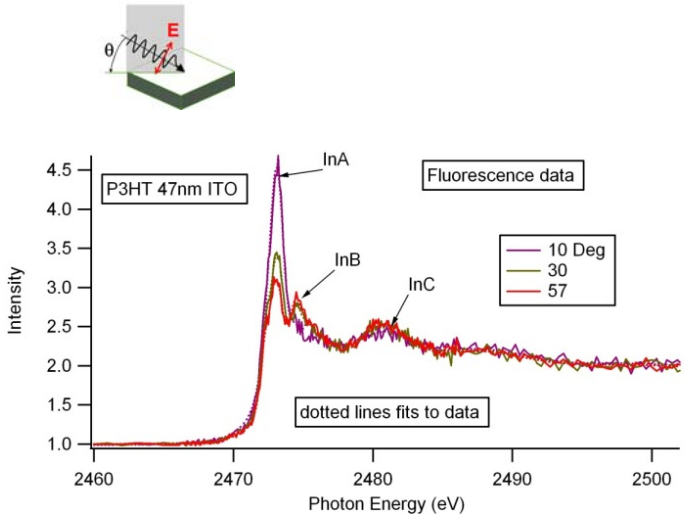
Main Features (Equipment Capabilities):

- Source Dipole
- Energy range 2.4 (Sulphur K edge) - 27 keV (Cd K-edge).
- Double crystal Si(111) and Si(311) monochromator, resolution (dE/E) 2E^{-4} and 1E^{-4} respectively.
- Mirror for higher order rejection for the low energies, e.g. Sulphur measurements
- High throughput sample stage
- Cryogenic stages:
  Closed cycle He-cryostat (15 K - 320 K, 0.1 K accuracy), liquid N_2 cryostat (-130 - +400 °C)
- 3 Ionisation chambers for transmission measurements, detection limit 5%
- Combined 2 Ionisation chambers for transmission measurements, Sulphur measurements
- Fluorescence detection 5 element Ge detector and 1 element SDD detector (140 eV resolution) detection limit 1mmol/l
- QXAFS 1-2 min fluorescence mode and 30-60 seconds in transmission

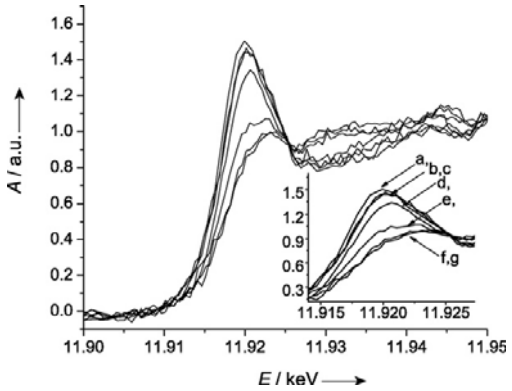
Limitations / constrains

- Spatial resolution limited to aperture size i.e. low flux density (radiation damage reduced - e.g advantageous for biological specimens)
- Handling of the beamline and the data evaluation needs knowledge about XAFS

Typical structures & designs



Angle dependence of the XANES from a thin nanometre polymer film which shows a clear orientation dependence: This is of importance to Solar Cell device characteristics
(Anka Annual report 2010, U.Aygul et al.).



Reduction of Gold to metallic state in the oxidation of oxidation dibenzylamine with $Au(OAc)_3/CeO_2$. The in-situ formed gold nano particles are responsible for catalysing the reaction
(L. Aschwanden et al. Journal of Molecular Catalysis A: Chemical 300 (2009) 111–115)

***Any further Information*:**

The techniques available at XAS fit in best with the Joint Research activities 1 and 3 and are complementary to XAFS investigations on the smaller tens of micron scale where radiation damage may play a significant role.

- *JRA 1 - Strategies to eliminate / reduce nanoparticle batch-to-batch variability*
- *JRA 3 - Development of Tools for characterisation of nanoparticles in situ in biological matrices*