FAME-UHD: a new beamline well adapted to nano environment sciences for high dilution and high resolution measurements

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8th International Conference on the Environmental Effects of Nanoparticles and Nanomaterials – July 3rd – 5th, 2013 – Aix en Provence

WHY?

X-ray Absorption Spectroscopy (XAS) is particularly suitable for nanomaterial studies, and more precisely their **toxicological** or **environmental** impact. The sensitivity of this spectroscopic probe to the local coordination structure of the target element is indeed limited to a few Å and thus particularly relevant for the nano-size of the particle. Furthermore, the particles themselves are often diluted (in a soil, biological system...). Two conditions are thus required for the collection of high-quality XAS data :

(i) Acquisitions need to be sensitive to a **very small amount of nanoparticles** in order to be as close as possible to the predictable concentration of these elements in the ecosystem (1 - 200 ppm),

(ii) Differences between bulk and nanostructured materials spectra are often subtle and make necessary to fully **optimize the quality and the energy resolution of the spectra**

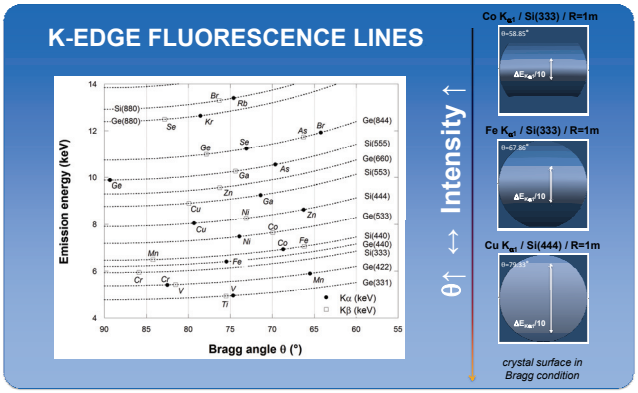
HOW ?

The new beamline will be complementary to the current one: FAME. Two parameters will be optimized

i) The **photon flux** on the sample will be increased, with optical elements allowing a collection of a large solid angle

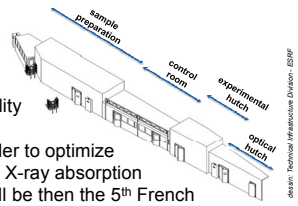
ii) The five crystals focusing spectrometer installed on FAME [Hazemann et al. 2009 ; Llorens et al. 2012] will be the basis of the high resolution detector installed on FAME-UHD. The new spectrometer will include more crystals (5 → 14 or 21) at a larger distance (1m) to increase both the **solid angle** collection and the **energy resolution**.

K-EDGE FLUORESCENCE LINES



WHERE & WHO ?

FAME-UHD will be installed on BM16 at the European Synchrotron Radiation Facility (ESRF). FAME & FAME-UHD will operate simultaneously with a common staff in order to optimize the complementarities between these two X-ray absorption spectroscopy instruments. FAME-UHD will be then the 5th French Collaborating Research Group beamline at the ESRF.



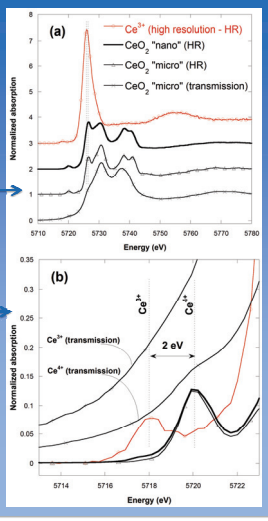
WHEN ?

The new beamline is under construction. Experiments with a crystal analyzer spectrometer are already possible on FAME. FAME-UHD will be opened to users in January 2016.

HIGH ENERGY RESOLUTION FLUORESCENCE DETECTED XANES

Measurement of fluorescence signals using crystal analyzer spectrometer instead of a solid state detector significantly improves X-ray Absorption Near Edge Structure (XANES) features. Measurements performed on CeO_2 micro- and nanoparticles, as well as Ce^{3+} reference, clearly showed the interest of high spectral resolution. All the XANES features of CeO_2 obtained classically just pop out using crystal analyzer.

For example, pre-edge peaks are not seen using classical measurement (transmission in this case) but clearly resolved in HR-XAS (Fig. b), which allows to clearly distinguish between 3+ and 4+ valence states. Spectral analysis is not only improved but simply made possible in many cases.



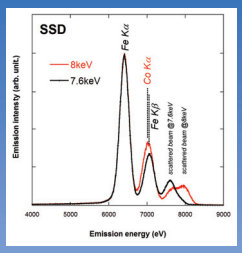
IMPROVED FLUORESCENCE SELECTIVITY

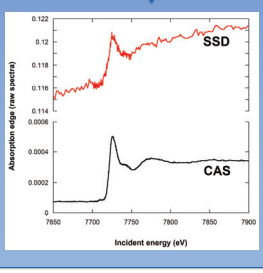
The use of a solid state detector (SSD) can find limitations when a high counting rate due to scattered beam and/or fluorescent signal from the matrix containing the diluted element does not allow to detect the signal of interest.

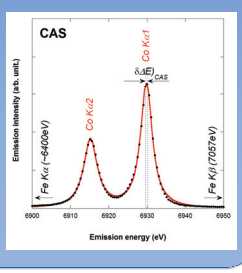
In the particular case of Co adsorbed on Fe oxide, the absorption spectrum at the Co K-edge is difficult to measure with a SSD detector because the Fe K_β fluorescence lines is superimposed to the Co K_α one ($\Delta E \sim 250eV$) .

With a CAS, the improved energy resolution ($\Delta E \sim 1eV$) allows to discriminate all the fluorescence lines.

Co K-edge XANES spectra have been then measured in total (SSD) and partial (CAS) fluorescence modes.







This project is financially supported by the french "grand emprunt" EquipEx (EcoX, ANR-10-EQPX-27-01), the CEA-CNRS CRG consortium and the INSU CNRS institute. Construction of the spectrometer prototype was financially supported by the INSU CNRS institute, ANR NANOSURF (coordinator: C. Chaneac, Laboratoire de Chimie de la Matière Condensée de Paris, France), ANR MESONNET (coordinator J.Y. Bottero, CEREGE), and CEREGE laboratory (Aix en Provence, France).

References

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• Llorens et al., "High energy resolution five-crystal spectrometer for high quality fluorescence and absorption measurements on an X-ray Absorption Spectroscopy beamline", *Review of Scientific Instruments* **83** (2012) 063104