REPORT

Beamtime 26-01-857 DUBBLE
16.12.2009 – 21.12.2009

Annemie Adriaens[1], Mark Dowsett[2], Koodlur Lokesh[1], Matthew Hand[2]
[1] Universiteit Gent, Vakgroep Analytische Chemie, Krijgslaan 281-S12, 9000 Gent
[2] University of Warwick, Department of Physics, Coventry CV4 7AL, UK

In December 2008 and March 2009 we made our first measurements on DUBBLE using the proof of concept XEOL detection system shown in Figure 1. The objective was to obtain improved signal to noise ratio, compared with our pilot studies on SRS station 7.1 (see cover story in Analytical Chemistry on 15 November 2008 [1]). By virtue of the higher flux on DUBBLE, and a new large aperture light-optical system with optical bandpass filtering, we separated electronic state information (giving e.g. near-edge resonances in specific optical bands) from the site specific (i.e. X-ray fluorescence-like) parts of the spectrum, revealing information channels which were effectively identical with conventional XAS (but potentially optically imageable on the micron scale), and others resulting from direct excitation of states in the valence band.

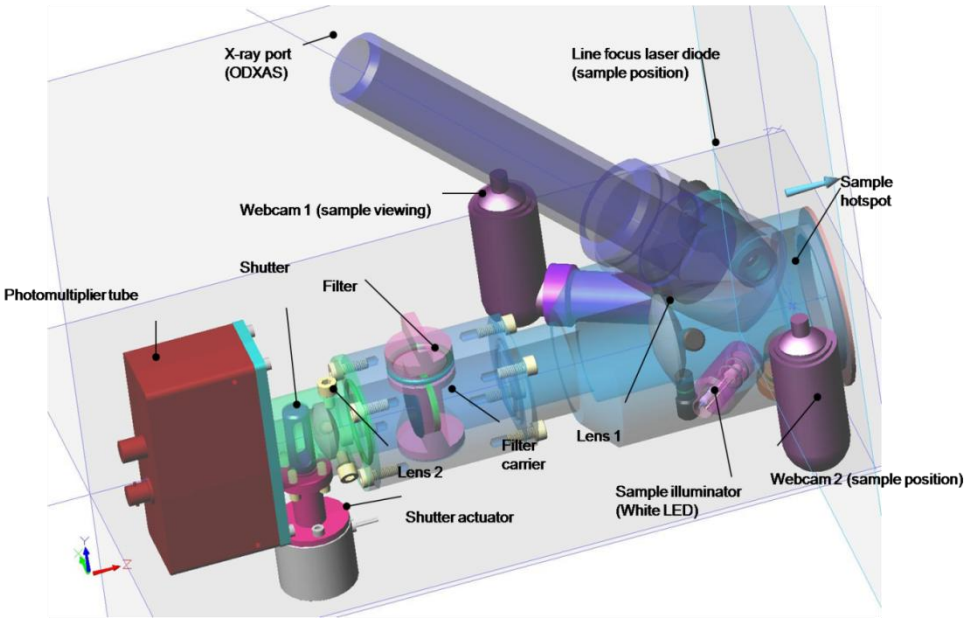

Figure 1.

Just as we have done for copper compounds [1], the aim of this beamtime was to start up a comparative XANES/EXAFS library of spectra from lead corrosion products, acquired from the optical emission and from conventional fluorescence measurements. Originally the idea

was to perform both measurements at the same time (XEOL and XAS) and we actually tried to set-up the equipment, but the electronics of DUBBLE did not allow us to acquire and process two signals at the same time. Sergey Nikitenko, beamline scientist of DUBBLE, will look into this problem.
As a result, we performed separate measurements: (i) XAS in transmission mode, (ii) XAS in fluorescence mode and XEOL of the following lead compounds lead (II) oxide, lead (IV) oxide, lead acetate, lead carbonate, lead chloride, lead sulphate and lead powder. For the transmission experiments, the powders where diluted with boron nitride and pressed into a pellet. Spectra were recorded at the LIII edge of lead (13.06 eV).
In addition preliminary experiments were done on gold substrates modified with N4-macrocyclic molecules. Measurements here were performed at the Au LIII edge (11.92 eV).

[1] M.G. Dowsett, A. Adriaens, Cell for Simultaneous Synchrotron Radiation X-ray and Electrochemical Corrosion Measurements on Cultural Heritage Metals and Other Materials Analytical Chemistry 78(10) (2006) 3360.