ESRF

| Experiment title: Combination of x-ray diffraction and x-ray absorption spectroscopy at BM29 | Experiment number: MI215 |
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| **Beamline:** BM29 | **Date of Experiment:** from: to: | **Date of Report:** |
| **Shifts:** | **Local contact(s):** A. Filipponi, M. Borowski | *Received at ESRF:* |


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**Report:**

Several efforts have been devoted in the last years (since the experiments connected with proposal MI124 and MI215) to implement an experimental setup which combines x-ray absorption spectroscopy and x-ray diffraction at BM29. Among the various attempts we explored (a) the possibility to detect the radiation diffracted in the vertical plane using a position sensitive detector (b) the possibility to couple a $2\theta$ (angular scanning) diffractometer arm with the transmission EXAFS setup, and (c) the possibility to use a fixed angle collimator.

The latter option resulted the most versatile since it involves only a very simple instrumental addition to the EXAFS setup. The diffraction pattern is recorded by scanning the monochromator energy in a suitable range (energy scanning x-ray diffraction).

A full description of the present BM29 experimental setup for the investigation of condensed matter under extreme conditions of high-temperature and high-pressure, which combines various control and detection systems suitable to perform x-ray absorption spectroscopy, x-ray absorption temperature scans [1], and energy scanning x-ray diffraction (ESXD) is given in a extended paper recently accepted for publication in Rev. Sci. Instr. [2].

The experimental setup can be combined with two sample environment devices; the L' Aquila-Camerino oven [3] for high-temperature studies up to 3000 K in high vacuum and the Paris-Edinburgh press [4] suitable for high-pressure high-temperature studies in the range 0.1-7 GPa and temperatures up to 1500 K.

An example of the performances of the ESXD setup of BM29 is reported in the figures below that show the diffraction scans obtained for samples of Pd powder in graphite or $Al_2O_3$. In the figure on the right a collection of x-ray diffraction energy scans at fixed $2\theta \approx 15.6°$ of the Pd(200) and Pd(111) peaks for samples of $Pd/Al_2O_3$ (A and B) and Pd/graphite (sample C) is reported. The energy scales for Pd(200) and Pd(111) are scaled by $2/\sqrt{3}$ for a direct comparison. The room temperature reference is marked as a vertical line, for sample C the peak is shifted since the lattice is contaminated with C. Upon increasing temperature the peaks (measured in the range 1200-1800 K) shift to lower energies due to the lattice expansion. The minimum FWHM of the diffraction peaks achieved so far is about 20 eV at 20 keV which guarantees a lattice spacing determination with a sensitivity of $10^{-4}$.

The EXAFS spectra collected on solid, liquid, and undercooled liquid Pd using the same BM29 setup have been the subject of a separate publication [5].


[1] A. Filipponi, M. Borowski, P. W. Loeffen, S. De Panfilis, A. Di Cicco, F. Sperandini, and M. Giorgetti, J. Phys.: Condens. Matter **10**, 235 (1998).

[2] A. Filipponi, M. Borowski, D. T. Bowron, S. Ansell, S. De Panfilis, A. Di Cicco and J. P. Itiè, Rev. Sci. Instr. (to be published).

[3] A. Filipponi and A. Di Cicco, Nucl. Instr. and Meth. in Phys. Res. B **93**, 302 (1994).

[4] J. M. Besson, R. J. Nelmes, G. Hamel, J. S. Loveday, G. Weill,and S. Hull, Physica B **180** & **181**, 907 (1992).

[5] A. Filipponi, A. Di Cicco, and S. De Panfilis, Phys. Rev. Lett. **83**, 560 (1999).