ESRF	**Experiment title:** **DAFS Study of the phase transition of the quasi-2D tungsten bronze $(PO_2)_4(WO_3)_{2m}$,m=13**	**Experiment number:** HE-5
Beamline: ID24	**Date of experiment:** from: 25-1 l-96 to: 2-12-96	**Date of report:** 28/2/97
Shifts: 15	**Local contact(s):** M. Hagelstein	*Received at ESRF:* **2 SEP. 1997**

Names and affiliations of applicants (* indicates experimentalists):

HODEAU, J.L.*' Laboratoire de Cristallographie - CNRS, BP 166,Grenoble
BOS Sandra" "
FAVRE-NICOLIN Vincent * "
LORENZO J.Emilio* "
HESS Christoph LEPES - CNRS, BP 166,Grenoble
SCHLENKER Claire LEPES - CNRS, BP 166,Grenoble

Report : the monophosphate tungsten bronzes $(PO_2)_4(WO_3)_{2m}$ are a family of compounds that presents Charge Density Wave (CDW) transitions, due to electronic instabilities. One important physical property associated with this CDW state is the low-temperature periodic lattice distorsion (Peierls transition due to an electron-phonon coupling), which gives rise to a **superstructure** and to **satellite reflections.**

Since these satellite reflections are a **highly selective source of information about the CDW,** the study of both the **intensity** and **the phase** for these reflections is a direct way to quantify the atomic displacements associated with the CDW.

DAFS (Diffraction Anomalous Fine Structure) measurements consist in measuring diffracted intensities on a continuous range (from 200 eV to 1 keV) around the absorption edge of the resonant atom, thus yielding more information about the phase than MAD experiment (3 or 4 wavelengths, with a 30° max. precision for the phase).

The choice of a **dispersive optics** allows a **fast collection of I(E) spectra,** and eliminates an important source of systematic errors, as the whole setup (orientation, temperature,optics...) doesn't change.

Results : taking advantage of the small polychromatic focus point of ID24 (80 µm FW at 10%), the sample was easily aligned in the diffracting position, despite its very **small size** (200x100x10 µm). This demonstrates the feasibility of DAFS measurements on ID24 even for small crystals.

All diffraction images were collected using the rotating method, with a large 2D detector (image-plate with on-line reading). We thus collected 70 couples of images (the second one being taken with “finger slits” as energy markers, see report of exp n°MI-119 for details), both at room temperature and above the phase transition. An example of the images obtained is shown in fig. 1.

We have observed unexpected features, such as a superstructure due to stacking faults in the sample (fig. 2). This emphasizes the importance of a large **2D detector** to provide a large vision of what *actually* (expected or not) happens in the compound, which is of course very important for phase transitions.

The extraction of data from the images follows the process described for the experiment n°MI-1 19 (image corrections, integration, energy calibration, intensity normalization). But since the quantity of data (hundreds of reflections, 3 gigabytes of data) is very important, we are developping a program based on IDL language : this software will allow data reduction for all dispersive DAFS measurements in the rotating method, including indexation of reflections (main peaks or satellites ; with a refinement of experimental parameters), *automatic integration of I(E) spectra* with background substraction, and *collection of orientation parameters* for each reflection (to allow absorption and lorentz-polarization corrections in further refinement). The development of this program is well under way and data reduction for this experiment will follow.

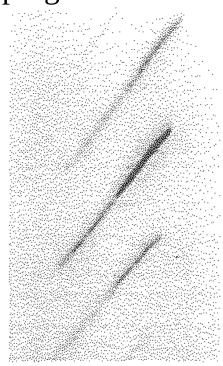

Fig 1. A row of satellite reflections. Each line correspond to a full I(E) spectra for one reflection : the energy varies linearly along the line, with longer wavelengths on the upper (right) end of the line, and shorter wavelengths on the other end. The step in intensity marks the W L_{III} edge. The energy range is ≈600 eV.

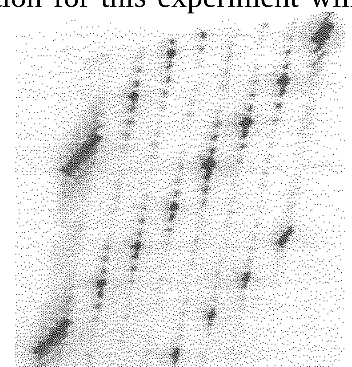

Fig.2. Superstructure observed in the sample, due to stacking faults (the structure of the crystal consists in slabs of 13 WO_6 octahedras, and some slabs have only 12). This shows a long-range order with $\lambda \approx 600$ Å along c*.For satellite reflections, this superstructure is replaced by diffuse scattering.
(this image was taken with "slits" used as energy markers, hence the slicing of the lines)