EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Chemical environment of the uncovered $AsHg_8$ compact structure responsible for the p-type electrical conductivity in As:HgCdTe	**Experiment number**: 30-02-994
Beamline: BM30B	**Date of experiment**: from: 03/12/2010 to: 07/12/2010	**Date of report**: 21/02/2013
Shifts: 12 (16b)	**Local contact(s)**: O. Proux	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Xavier BIQUARD*, CEA-Grenoble

Philippe BALLET*, CEA-Grenoble

Report:

Overview:

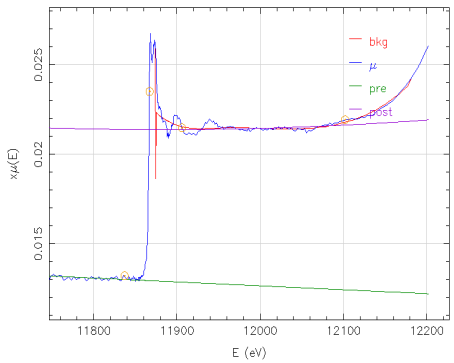
This experiment was dedicated to the study of the $AsHg_8$ compact structure (X. Biquard et al., J. Appl. Phys. 106, 103501 (2009)) that was discovered in 2009 with ESRF's experiment MA-442 (sample 24875_01).
The 30-elements Canberra detector was used (500ns shaping time), ROI of each detector's element were individually adjusted with minimal width to select only the As fluorescence and avoid as much as possible Hg fluorescence. Monochromator's glitch at 12010eV was systematically removed.
A relatively large number of samples (>20) were made available specifically to conduct this experiment. These samples cover the full extend of meaningful As concentration, ranging from low (1.0 10^{17} at/cm^3) to highly doped (1.7 10^{19} at/cm^3) As:HgCdTe. 3 types of sample exist:

- as-grown samples
- n-type samples that have undergone a single low-temperature heating (#200°C) to fill out Hg vacancies
- p-type samples that have undergone first a high-temperature activation annealing (3h@370°C) under Hg overpressure before a low-temperature heating

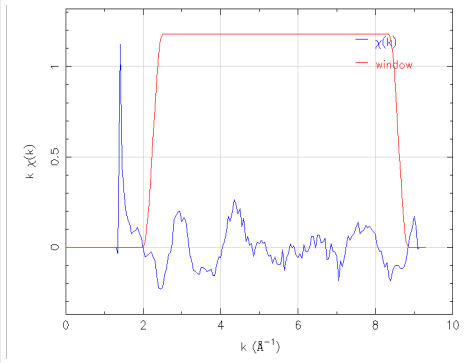
It is in this latter type of p-type samples that is found the $AsHg_8$ compact structure.

Measurements:

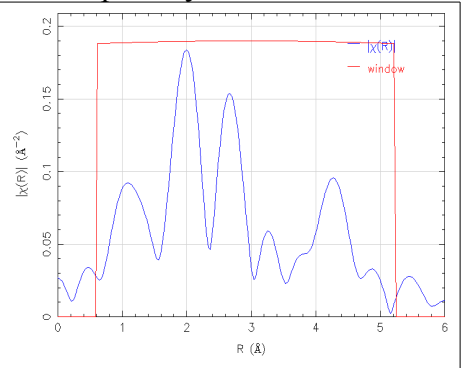
To check the experimental setup, we started by recording a single spectrum on our reference p-type sample (24875_01). XAS spectra was of good quality, EXAFS oscillations were clearly visible until k=9Å^{-1}. In R-space, the first shell of 8Hg was clearly visible as well as a second shell. It is this second shell we want to clearly identified as constituted of Te. We note the presence of a low-k/low-R contribution probably due to a small oxydation of this sample elaborated 1½ year ago despite it was stored under primary vacuum.



XAS spectra with white line at 11868eV and secondary at 11874eV. Edge step is 0.00844



kχ(k) oscillation ranges from 2.0 to 8.85Å^{-1}

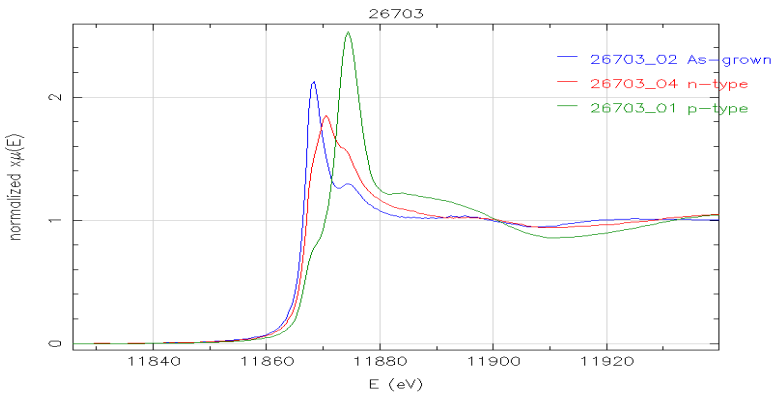


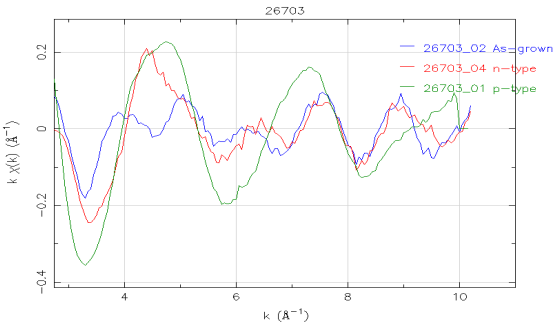
|χ(R)| shows Hg double contributions at pseudo-R 1.99/2.67Å, second shell at 4.27Å and probably oxide contribution 1.1Å

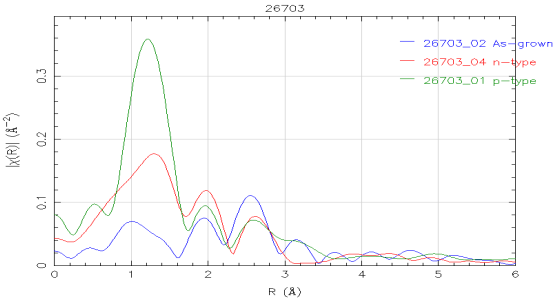
Here is the table of all our recorded samples:

Name	Type	Edge Jump (10^{-3})	As content (at/cm^3)	Comment
24875_01	P	8.44	5 10^{17}	Plate centre, reference sample
****				
26667_03	As-grown	9.38	5.5 10^{17}	Plate centre, 5 spectra,
26667_02	N	7.61	4.5 10^{17}	Plate centre, 7 spectra
26667_01	P	7.34	4.5 10^{17}	Plate centre, 4 spectra
****				
26697_01	P		<1.10^{17}	Not enough As
****				
26699_03	As-grown	40.78	2.4 10^{18}	Plate center, 16 spectra
26699_05		16.57	9.7 10^{17}	Plate corner, 9 spectra
26699_14		18.45	1.1 10^{18}	Plate corner, 1 spectrum
26699_04	N	32.47	1.9 10^{18}	Plate center, 11 spectra
26699_01	P	29.41	1.7 10^{18}	Plate center, 12 spectra
26669_08		13.20	7.8 10^{17}	Plate corner, 5 spectra
****				
26702_01	P	46.08	2.7 10^{18}	Plate centre, 1 spectrum
26702_08		17.12	1.0 10^{18}	Plate corner, 1 spectrum
****				
26703_02	As-grown	215.8	1.3 10^{19}	Plate centre, 1 spectrum
26703_04	N	218.6	1.3 10^{19}	Plate centre, 1 spectrum
26703_01	P	240.1	1.4 10^{19}	Plate centre, 1 spectrum

5 plates (ZnCdTe 2” substrate) were specifically realised to conduct our study. In each plate, 16 samples were cut, central samples being numbered 1, 2, 3 and 4 where As fluorescence intensity show a good homogeneity. Corner samples are outside the homogeneity zone extend of the plasma source. Samples from plates 26667 and 26699 were ideal for our study: low enough to avoid As clustering and high enough to extend EXAFS oscillations as far as possible. Data reduction was conducted on-site during experiment and sadly, we noticed that all p-type samples were completely oxidised as illustrated on highly-doped plate number 26703:







Sample edge shifts by 6eV from metallic edge 11867eV to oxidised arsenic as 11872.9eV, the white line does the same, kχ(k) EXAFS oscillations display a completely decreasing intensity and |χ(R)| shows a single short-distance contribution.

Several months later, it was shown that the brand new oven that was used for high-temperature annealing had in fact a micro-leak localised near a current feed-through: oxygen was present during annealing thus explaining the fully oxidised p-type samples. Therefore, our experiment could not reach its goal but we nevertheless fully used available beam time to record good quality spectra from our samples.