| | **Experiment title:** **Time resolved study of the reversible phase change mechanism used in rewritable DVD** | **Experiment number** HS-2095 |
|---|---|---|
| **Beamline**: ID22 | **Date of experiment**: from: 23/05/2003 to: 28/05/2003 | **Date of report**: 25/02/2004 |
| **Shifts:** 15 | **Local contact(s)**: Alexandre SIMIONOVICI, Gema MARTINEZ-CREATO | *Received at ESRF:* |


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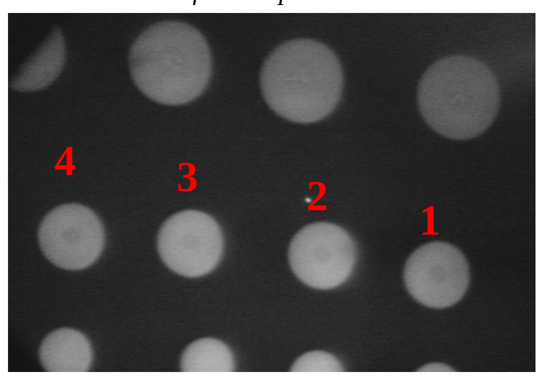
**Report:**

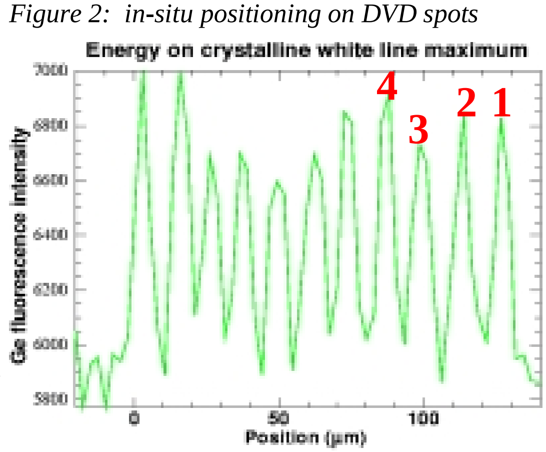
To be able to successfully conduct this experiment, the main experimental challenge was to make sure that the microscopic X-ray spot always probe the same and unique enlarged DVD spot of Ø7µm (see picture 1). Also, as we probed a 20 nm thin layer, the X-ray beam flux had to be made maximal: the distance through air was kept at a minimum and the solid state energy-resolved detector was brought as close as possible to the DVD disk. Then, to keep the dead time of the detector below 5%, its shaping time was diminished to 250ns. For stabilities sake, the undulator harmonic was kept constant to the first one.

*Picture 1: view of DVD spot number 1 to 4*




– Once obtained the most intense X-ray beam spot of Ø4-5µm, we noticed that the X-ray spot had instabilities, jumping around by 2.5µm vertically and 4µm horizontally. According to the beam position monitors, these instabilities came from the machine that was delivering its last Single-Bunch mode. As these too strong jumps prevented us from conducting our experiment, we had to wait for 24 hours for them to disappear spontaneously.
– Once stabilized in position, we use a golden cross to pre-align the microscope. Then, taking advantage of the 1.1eV energy shift of the maximum of the white line when going from amorphous state to crystalline state, we did an in-situ positioning of the X-ray beam on a chosen single crystalline DVD spot as illustrated of figure 2 where we followed a series of 9 identically spaced crystalline spots.

*Figure 2: in-situ positioning on DVD spots*



Once the X-ray beam impact position on the DVD was in-situ determined, we check that we were able to record in a reasonable acquisition time a correct XAS spectra at the Ge K-edge, either on a crystalline zone or an amorphous zone. And this was indeed the case as is clearly illustrated on Figure 3 where these recorded spectra are identical (with less signal) with reference ones obtained using a large X-ray spot of Ø500µm (direct beam going through the KB).

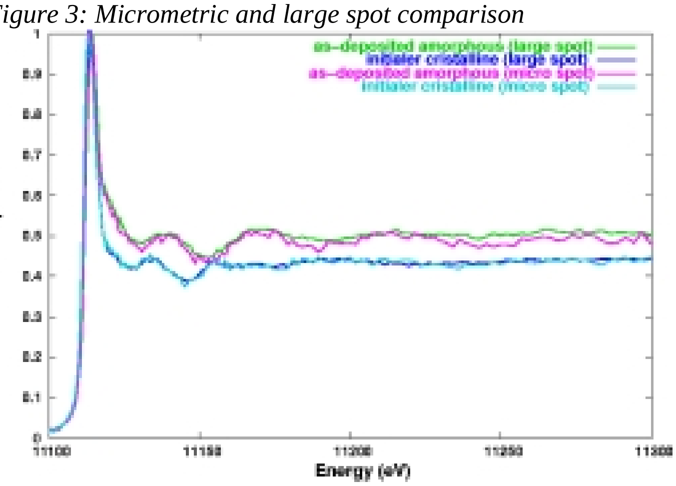



*Figure 3: Micrometric and large spot comparison*

Finally, we positioned the micro spot on the crystalline single DVD spot number 1 and then number 2 (see picture 1), and we recorded XAS spectra. As is illustrated on figure 4, the recorded spectra are not identical to the one that were recorded on the large crystalline zone: the edge is situated between amorphous and crystalline, as well as the mean level of EXAFS oscillations. And indeed, looking closer to picture l, the DVD spot white colour is not uniform but exhibits a darker Ø2µm centre: those enlarged DVD spot are not of a uniform crystalline phase but certainly present an amorphous centre.

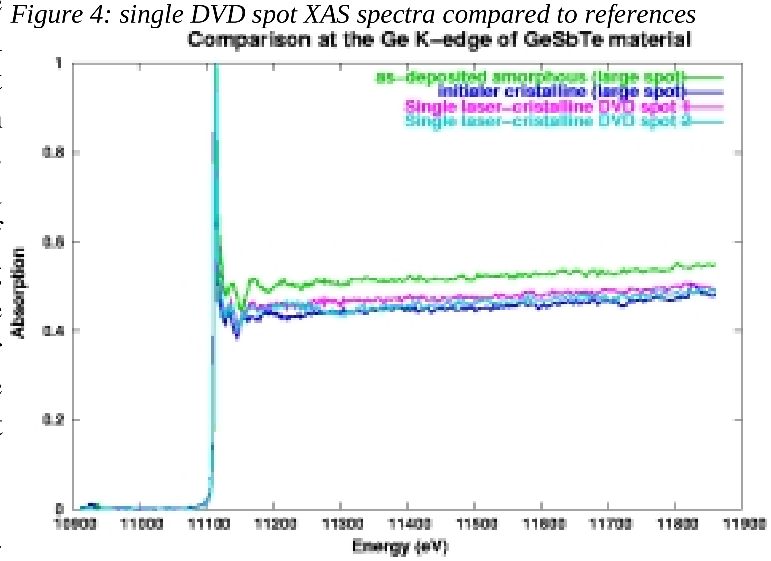



*Figure 4: single DVD spot XAS spectra compared to references*

Moreover, recorded spectra of DVD spot 2 presents an anomalous drop around 11300eV. To understand the origin of this drop, we have analysed the K$\alpha$ fluorescence intensity of the 1.5% of Ti located inside the aluminium layer. As the Ti fluorescence is more the 6keV away from the Ge edge, its intensity as a function of energy is a monotonous and slow varying function as illustrated on the top part of figure 5. But the most important, is that – for an unknown reason yet – the Ti intensity is 13% larger when situated below an amorphous zone than a crystalline zone: the Ti intensity is therefore an in situ means to check position of the X-ray beam at any given energy. And looking now to the low part of figure 5, one clearly see a Ti intensity drop around 11300eV for the DVD spot 2: the X-ray µ-spot has moved out of the DVD spot. Therefore, the beamline has to be more finely tune to ensure a sufficient µ-spot stability on the whole 1 keV energy range.

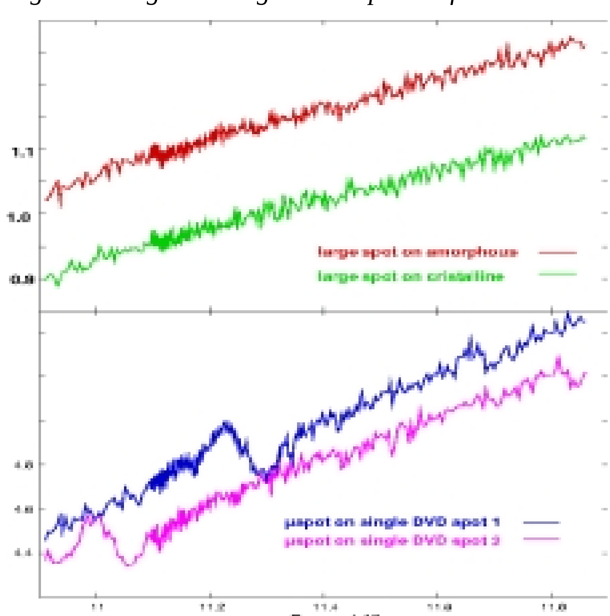



*Figure 5: large and single DVD spots Ti fluorescence*