

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 using the **Electronic Report Submission Application:**

<http://193.49.43.2:8080/smis/servlet/UserUtils?start>

Reports supporting requests for additional beam time

Reports can now 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.

**Experiment title:**

Evolution of the concentration profile at the interfaces of multilayers made from an ideal binary system : Mo-V

Experiment number:
Si-1065

Beamline:**Date of experiment:**

from: 01-28-05 to: 04-02-05

Date of report:

1 march 2006

Shifts: 18

Local contact(s): Hubert Renevier

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Report:

Fundamental questions still remain on diffusion at the nanoscale. There are indications that short range diffusion is no more Fickian and display linear-with-time kinetics. The group of Prof. Beke in Debrecen, Hungary [1] has shown recently that diffusionnal asymmetry, i.e. the strong concentration dependence of the diffusion coefficients (eg. The diffusion in B is much more faster than in A), controls the interdiffusion profile in an A/B couple. When the asymmetry is large enough and A-B is a miscible system intermixing preserves the sharpness of interface. In the same way an initially smeared out interface may sharpen after annealing [1]. These surprising theoretical results lack of experimental evidences. We would verify these predictions on the model system Mo-V. This system is perfectly miscible and displays a large asymmetry in diffusion coefficients, mostly caused by the difference in melting points between the two elements [2]. Moreover, the lattice parameter misfit is small enough: 3.8 %, which ensures a good crystalline quality of the multilayers.

The method used here is anomalous diffraction i.e. energy dependent diffraction while crossing the absorption edge of Mo. The strong variation of Mo x-ray scattering factor in the vicinity of the edge induces a variation in the Mo/V contrast and hence gives chemical sensitivity to diffraction. Theoretical results indicate that Mo diffuses in V layers and V does not diffuse in Mo layers. It is then better to do experiments

at the Mo K edge. This kind of experiments has already shown its ability to clearly determine the concentration and interplanar distance across the interfaces [3].

As noted above we want to evidence here interface sharpening as a result of annealing in Mo/V (001) multilayers. To fulfill that purpose we studied two kinds of samples: either samples prepared with sharp initial interfaces or having diffuse initial interfaces. In this latter case, the interfaces extend over 7-8 planes with a linear concentration dependence. The Mo-V superlattices are grown on MgO (001) by magnetron sputtering at 650 °C. For these two kind of samples, we have looked at as grown samples and samples annealed at 700 °C for 60 min.

The experiments which have been done are:

- long range high angles symmetric $\theta/2\theta$ scans below the absorption edge to refine interplanar distance.
- long range small angles symmetric $\theta/2\theta$ scans below the absorption edge to refine concentration profile (only for sample with diffuse interface).
- energy scans (across Mo K edge at 19.95 keV) of the different satellites in the 1st order symmetric diffraction to extract the concentration profile.
- X ray diffraction in grazing incidence to measure the in-plane lattice parameter.

Unfortunately, none of the the dafs measurements reveal a significant change in the global shape of the scan (fig 1). The only significant change was found in the relative intensities of the satellites of the high angles symmetric scans (fig 2).

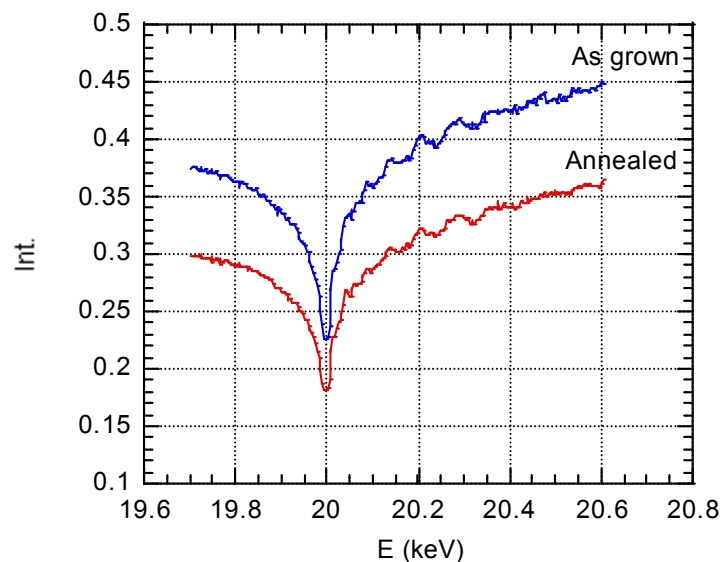


Figure 1: Typical intensities changes measured at the top of one satellite crossing the Mo adsorption edge.

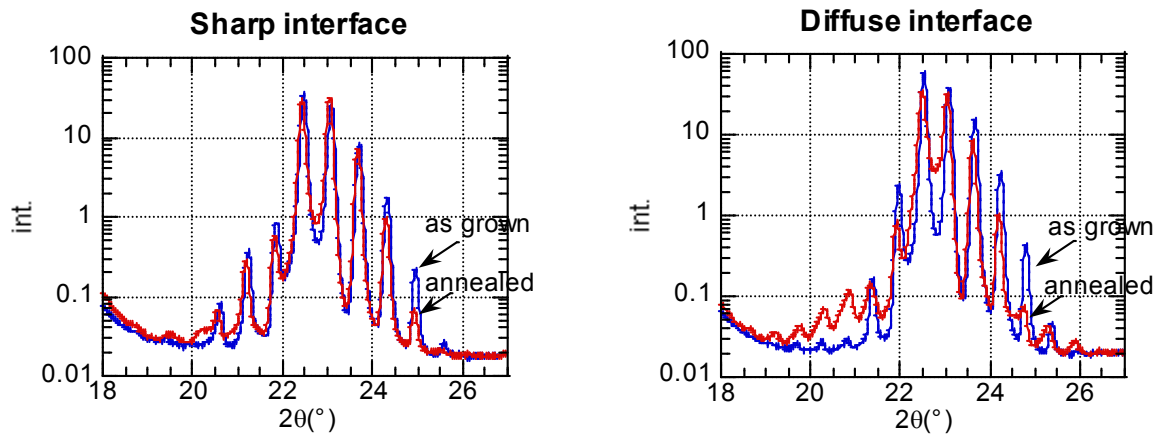


Figure 2: high angles symmetric scans at 19.9 keV for the 4 samples

Several informations can be extract straightforwardly when we compare the symmetric scans measured for the samples as grown to the ones measured for the sample annealed :

- Increase of the intensity of the satellite corresponding to the average lattice parameter of the multilayers
- decrease of intensities of the satellites at large angles
- no change of the period of the multilayer
- small decrease in the average perpendicular lattice parameter of the multilayer

Now, we are working on the refinement of the perpendicular structure of the multilayer using Suprex 9.0 to simulate the high angles and small angles symmetric scans.

- [1] Z. Erdelyi, I. A. Szabo and D. L. Beke, Phys. Rev. Lett. 89 (2002) 165901.
- [2] Z. Erdélyi, D. L. Beke, P. Nemes and G.A. Langer, Phil. Mag. A 79 (1999) 1757.
- [3] T. Bigault, F. Bocquet, S. Labat, O. Thomas and H. Renevier, Phys. Rev. B 64 (2001) 125414.