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Experiment Report Form

	Experiment title: Thin film delamination study during in situ compressive testing by Scanning micro X-ray Diffraction	**Experiment number**: 32-02-658
Beamline: BM 32	**Date of experiment**: from: 06/09 to: 12/09/2007	**Date of report**: 09/10/2007
Shifts: 15	**Local contact(s)**: Jean-Sébastien MICHA	*Received at ESRF:*

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Report:

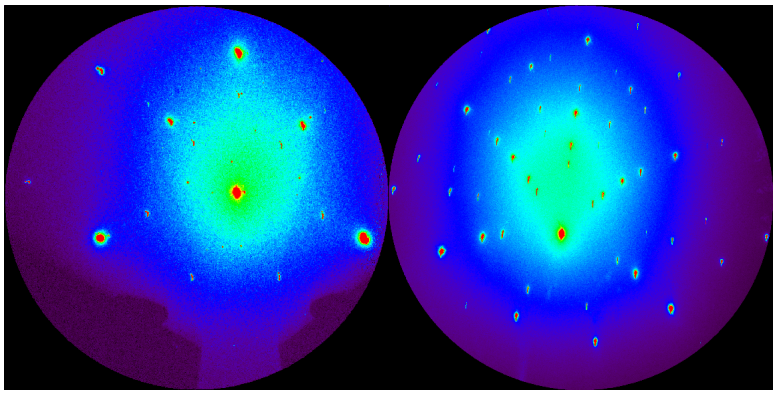
Our objective was the study of in situ delamination of supported thin films during compression test. For that purpose, scanning x-ray diffraction is used to measure elastic strain maps in the film and the substrate using mono and white beam respectively since the film is polycrystalline with nanometric grain size and the substrate is single crystal.

Carrying out of the experiment

One day and half shifts have been necessary for installing the end station beam line comprising the KB's, the sample stage, the optical microscope and the CCD camera for diffraction recording, and for optimizing the micro focus white beam mode. The CCD and the microscope cannot be used at the same time which is not convenient since the observation of the thin film surface during compression test is essential for following delamination phenomenon. Furthermore, the removing of the microscope after each loading step leads to vibrations and thus focusing perturbations. Several tests have been done using this mode but the beam size was not as satisfactory as expected: 1 µm in the horizontal plane and 3 µm in the vertical plane because of constant vibrations located at the KB's. Let us notice that the first optimized beam size for white beam mode (http://www.esrf.eu/UsersAndScience/Experiments/CRG/BM32/Microdiffraction) obtained in April 2006 was 0.5 x 0.7 μm^2. Concerning the monochromatic mode, the adjustments were terrible and dramatically time consuming. Finally, one day x-ray beam has been used for the project. Owing to the slow recording time process for mapping stresses in the film, only three loading steps have been achieved; two before buckling and one after.

Results

The analysis of all the measurements performed during the compressive test is not yet finished. The dedicated XMAS software is used for that purpose. The quality of the data obtained with the white beam is better for ESRF compared to ALS since the energy range is larger for ESRF (5-30 keV) than for ALS (5-15 keV). This is clearly visible on figure 1 where the number of Laue spot is larger for ESRF diagram.



(a) **(b)**

Figure 1: Laue diagrams obtained (a) at ALS and (b) at ESRF using a micro focused white beam on a LiF single crystal. The CCD camera is similar in both cases as well as the diffraction geometry

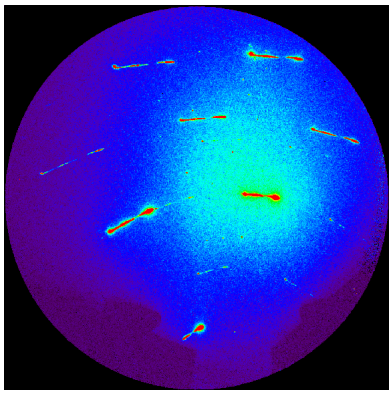
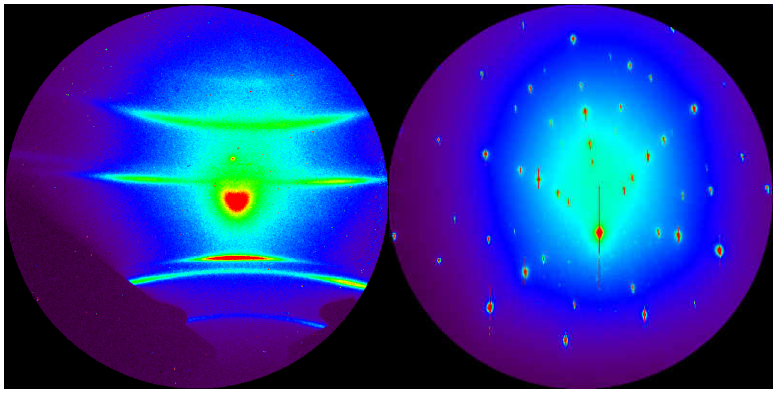


Figure 2: Laue pattern measured on The LiF single crystal substrate deformed up to 10 MPa.

When the plastic regime is reached in the LiF substrate, streaky spots appear in the Laue pattern as shown in figure 2. This effect is directly related to gliding dislocations whereas elastic strains are extracted form Laue distortion. This type of information cannot be easily extracted from the pattern and specific routines must be developed.

Concerning the monochromatic data, the recording time is 30 times larger that the one fixed for Laue measurements which is in fact limited by the read out of the camera (about 6 sec. for the MAR used here). A typical powder diffraction diagram is given on figure 3 (a). Maximum intensities on the rings correspond to the pole directions and are related to <111> fibber texture of gold films. Contributions of the LiF substrate are also non negligible, and some consecutive undesirable perturbations during analysis of the gold signal may exist. Strain/stress refinements are under progress. The main difficulty here is to get reliable and precise strains from powder diffraction rings. A specific routine is used to extract the strain tensor from all rings.



(a) **(b)**

Figure 3: 2D diagrams measured in gold coated LiF substrate with (a) mono and (b) polychromatic x-ray beams. The sample in (a) is prepared by ion beam sputtering whereas the films in (b) is obtained thanks to an evaporation system.

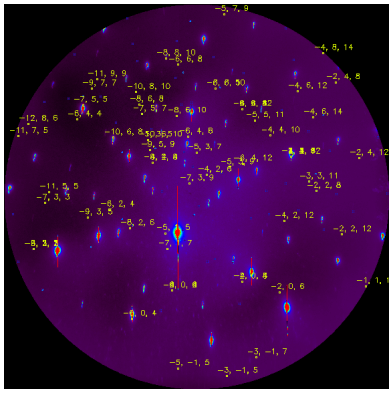


Figure 4: Indexed gold Laue pattern using XMAS software. The background has been subtracted.

Conclusions

Previous feasibility experiments done at ALS and ESRF on gold thin films deposited on LiF substrates have evidenced two main difficulties related first to the sample geometry which must be perfect whereas in plane strain gradients appear in the substrates generating thus heterogeneous buckling and secondly to the weak diffracted intensities in the monochromatic mode by the polycrystalline gold films containing nano grains. This last point is crucial and recent measurements done at ESRF have demonstrated this kind of measurements are almost impossible to achieve on this beam line owing mainly to the particularities of the set up. Indeed, it is a temporary experiment which means new adjustments at each measurements period (an average of two per year) which leads more often to decreasing performance (focused beam size due to KB's stability problems). Finally, the monochromator configuration does not allow reasonable fast poly / mono chromatic switching. Let us recall that the use of monochromatic x-ray beam for studying films is due to the small grain size produces by sputtering ion beam deposition techniques. A recent experiment done on an electron gun evaporation system has provided thin films with larger grain sizes. Using higher deposition speeds (or atom flux up to 0.3 nm / sec.) and a moderated deposition temperature (around 300°C), it is then possible to prepare gold films with grain size larger than the beam size and thus to get a Laue diffraction pattern (see figure 3 (b)). This has been verified last september at ESRF with a 1x3 μm^2 beam; the gold Laue pattern is easily indexed using XMAS (figure 4). The average recording time is 30 times larger in case of mono compared to poly and thus mono experiments are time consuming. However, the use of polycrabonate substrate will be difficult for such film preparation.

In summary, it is preferable to map the strain/stress with white beam in both the substrate (when appropriate) and the film, and to use occasionally the monochromatic beam to get the energy of one Laue spot and thus the dilatational part of the strain tensor (The Laue measurements give only the deviatoric part).

These first experiments have been the subject of several oral and poster presentations:

GOUDEAU P., RENAULT P.-O., COLIN J., COUPEAU C., FOUCHER F., GEANDIER G., TAMURA N.
Thin film delamination study during in situ compressive testing by Scanning micro X-ray Diffraction
MECA SENS IV, 24-26 sept 2007, Vienne (Austria).

GEANDIER G., MALARD B., GOUDEAU PH., TAMURA N.
Strain mapping by non destructive method : Laue microdiffraction
ECM24, 22-27 Août 2007, Marrakech, Maroc
ACTA CRYST. A63 (2007) S70-S71.

GOUDEAU P., GEANDIER G., RENAULT P.-O., TAMURA N., COUPEAU C., FOUCHER F.
Thin film delamination study during in situ compressive testing by Scanning micro X-ray Diffraction
ECM24, 22-27 Août 2007, Marrakech, Maroc
ACTA CRYST. A63 (2007) S235.

GOUDEAU P., RENAULT P.-O., COLIN J., COUPEAU C., FOUCHER F., GEANDIER G., TAMURA N.
Thin film delamination study during in situ compressive testing by Scanning micro X-ray Diffraction
Poster, Workshop « Nanomaterials: microstructural and mechanical characterizations, simulations », Poitiers (France), 12-13 Décembre 2006.

COUPEAU C.
Phénomènes d'endommagement des films minces : des structures de cloquage aux propriétés mécaniques locales.
Keynote lecture, Matériaux 2006 – Colloque 9 « Fonctionnalisation des surfaces – interfaces », Dijon, 13-17 Novembre 2006.

GOUDEAU P., COUPEAU C., FOUCHER F., GEANDIER G., TAMURA N.
Etude des propriétés mécaniques locales de films minces et revêtements par diffraction X en micro faisceaux (μ - DRX)
Matériaux 2006 – Colloque 9 « Fonctionnalisation des surfaces – interfaces », Dijon, 13-17 Novembre 2006.

GOUDEAU P., RENAULT P.-O., COLIN J., COUPEAU C., FOUCHER F., GEANDIER G., TAMURA N.
Thin film delamination study during in situ compressive testing by Scanning micro X-ray Diffraction
ALS User's Meeting, Berkeley (USA), 9-10 October 2006.