ESRF	**Experiment title:** Micro-structure - First Plastic Events Relationship in Gold Thin Films studied by Coherent X-Ray Diffraction	**Experiment number**: MA3324
Beamline: ID13	**Date of experiment**: from: 28/06/2017 to: 02/07/2017	**Date of report**: 22/09/2017
Shifts: 12	**Local contact(s)**: Manfred Burghammer, Andreas Johannes	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Pierre Godard, Julien Drieu la Rochelle, Pierre-Olivier Renault (also present: Tarik Sadat)

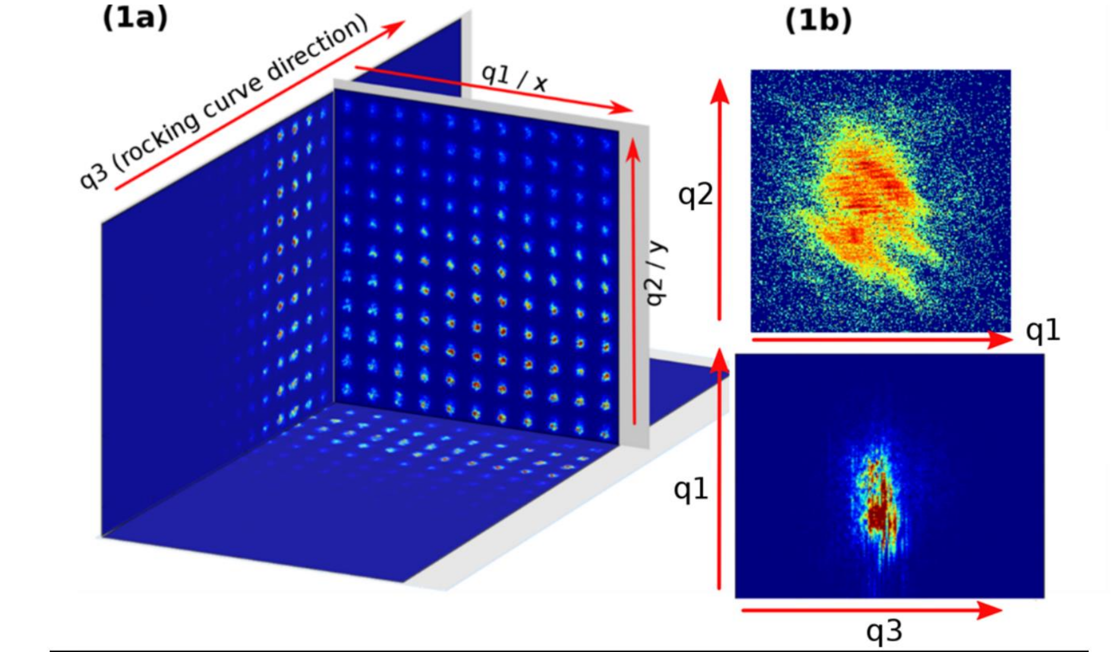
Laboratory Institut Pprime - Physique des Materiaux UPR 3346 CNRS-Université de Poitiers-ENSMA SP2MI Bd. M. et P. Curie, Téléport 2 Physique et Mécanique des matériaux FR - 86962 POITIERS

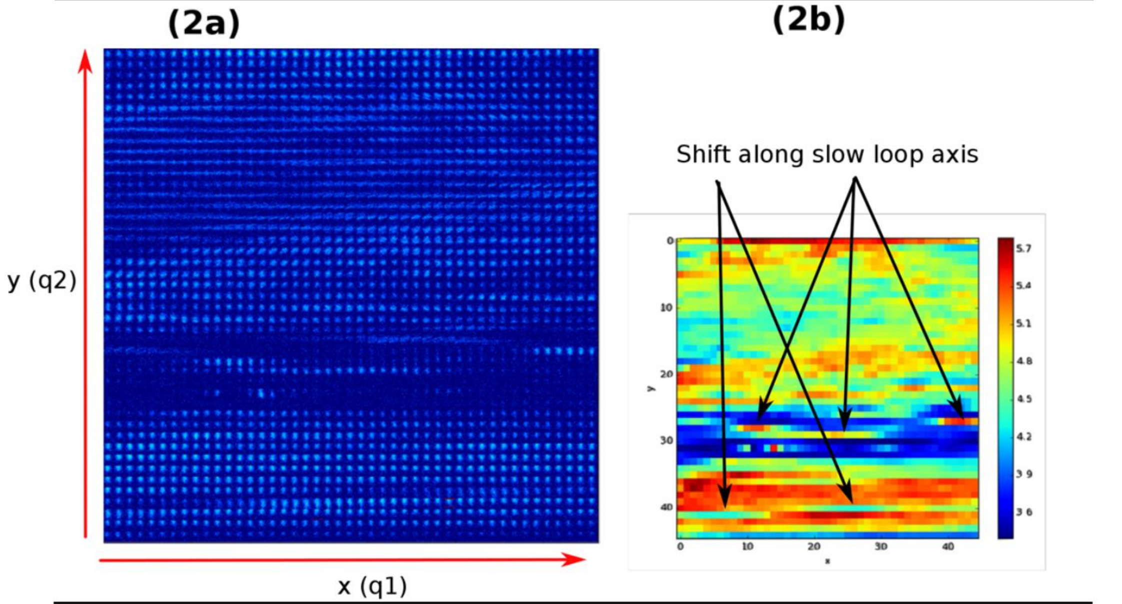
Report:

The MA3324 experiment (12 shifts) was to observe with the Bragg ptychography technic the first plastic events during deformation of twinned gold thin films. Indeed, a better understanding of gold thin film plasticity is important for the creation of flexible electronic components (skin sensor, screen, etc.). The ptychography technique is interesting because it could permit to have a 3 dimensional cartography of defects in the sample. The interferences due to the Coherent X-rays are very sensitive to the geometric characteristics and the phase defects present in the crystal, so the ptychography can show their evolutions after mechanical solicitation.

During our experimental session at the ID13 beamline, we acquired x-ray Bragg ptychography data sets after several strainings of gold thin films on flexible substrates (silicone and polyimide). Our sample was composed of a major monocrystalline [002] orientation with twins generating <221> orientation. We used a monochromatic 14.8 keV X-ray beam, focused at a 300nm x 300nm spot on the sample. For the deformation we use a mechanical device which deforms by traction. This device was placed on an hexapod and the beam went through the sample and we observed the diffraction patterns with an Eiger 4M. We focused our study on the [200] Bragg peak, aligned with the traction direction.
At first we made 11x11 cartographies with 250 points on a rocking curve (Figure 1a) at each deformation step. This configuration gave us a resolution of 11x11x5 nm^3 in the sample space. After a pre-strain at 5N, where we acquired a first ptychographic data set to serve as a reference state, we strained the sample to 10N, went back at 5N and collected a second ptychographic data set. This way we can map the residual strain lying in the sample. The procedure was repeated after an applied force of 15N. The accumulation of defects is clearly visible in the measured diffraction patterns. To be sure that we measured the same part of the sample, X-ray absorption maps on an adjacent crack, located 10 µm from the region of interest, were realized. But at the end data along the rocking curve axis are fitful (Figure 1b).
Because of the rocking curve problem encountered with the first sample, we installed a second sample, and acquire another set of data, composed of a 45 x 45 mapping at the rocking curve center (Figure 2a). But we encountered sample drift during the scan (Figure 2b): the data are discontinuous along the slow loop axis during acquisition.

All our sets of data need a deep post experiment treatment to be correctly analyzed. We are presently simulating samples with twins to recreate some coherent Bragg peaks (Figure 3).





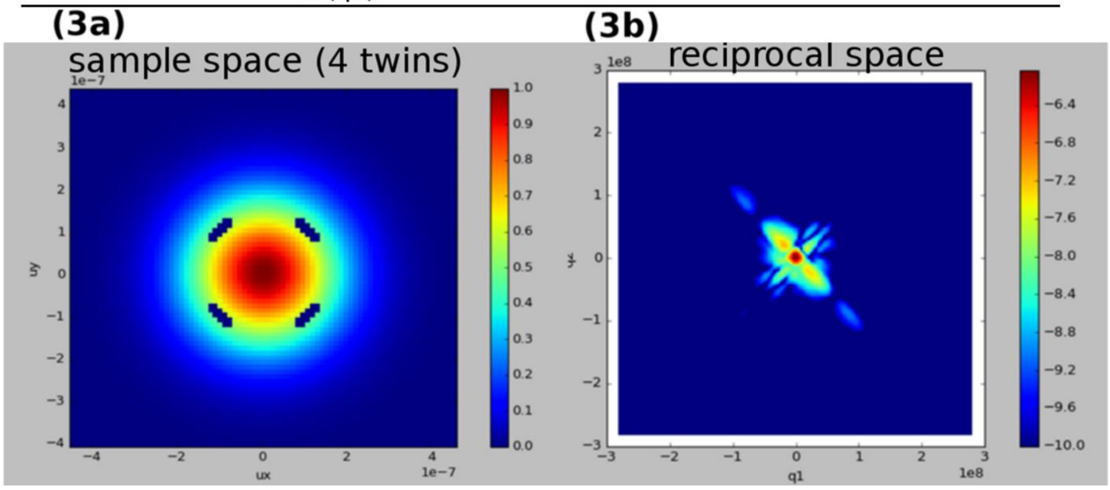


Figure 1 :(a) 5 dimensional matrix of data : q1.q2 (detectors pixels) . q3 (rocking curve direction) .x.y (sample cartography) ; (b) example of data along q1.q2 and q1.q3 axes.

Figure 2 : Cartography of the sample, 30nm step with a 300nm beam size, (a) q1.q2 map ; (b) global intensity map.

Figure 3 : (a) 4 twins illuminated by a 300nm x 300nm Gaussian beam ; (b) Simulation of the coherent Bragg peak (logarithm scale) of the sample (a).