ESRF	**Experiment title:** Interface sliding in FCC/BCC Cu/Nb nanolayers studied by in-situ X-Ray micro-diffraction	**Experiment number:** HC-4460
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Shifts: 12	**Local contact(s):** Nils Blanc and Gilbert Chahine	

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Report:

The goal of this experiment was the study of the mechanical properties and, in particular, the interface sliding in FCC/BCC nanolaminates. The aim is to measure the strain evolution and dislocation activities occurring in Cu/Nb nano-laminates during controlled in situ shear loading experiments, using monochromatic X-ray micro-diffraction. Micro-beam bending experiment, using samples prepared by accumulative roll bonding (ARB), have shown an anisotropic interface sliding behaviour linked to the strong in-plane texture. Similar behaviour is expected to be observed on macroscopic specimens, using in-situ tensile tests. Specific sample geometry has been used in order to promote shear loading parallel to the Cu/Nb interfaces.

FCC/BCC Cu/Nb nanolaminates with a nanolayer thickness of few tens of nanometers and a total thickness of 400 µm were prepared by ARB either in Singapore University of Technology and Design or at Harbin Technical University or at Erlangen University. Mechanical test specimens, about 1 cm long and 1-2 millimeters wide, are cut out of the multilayer plate along the rolling direction (RD) and along the transverse direction (TD) using electro-discharge machining (EDM). To promote shear loading, two notches with a width of 200 µm each are cut across the gauge width on both sides (see Fig. 1).

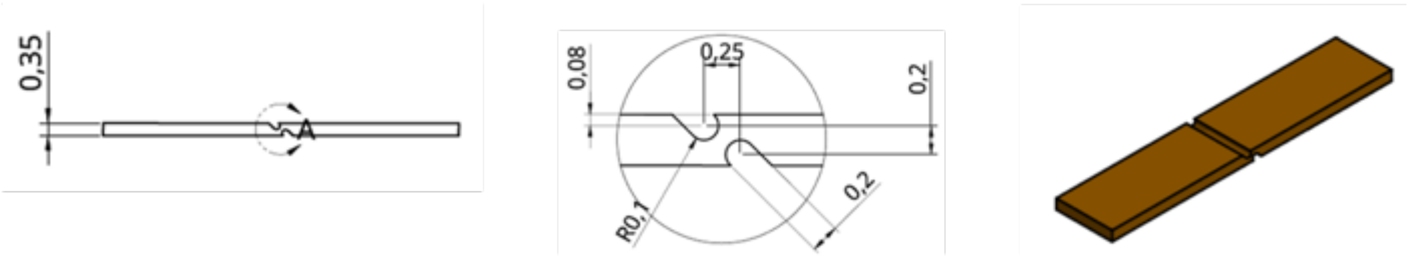

Figure 1 : Typical geometry of test specimens. Notches alow the central zone to evidence shear loading when the test specimen is loaded in tension.

For in situ mechanical testing, the displacement controlled tensile test machine Bulky (developed by Henry Proudhon at ENSMP) has been installed on the diffractometer at the BM02 beamline [1] (see Fig. 2).

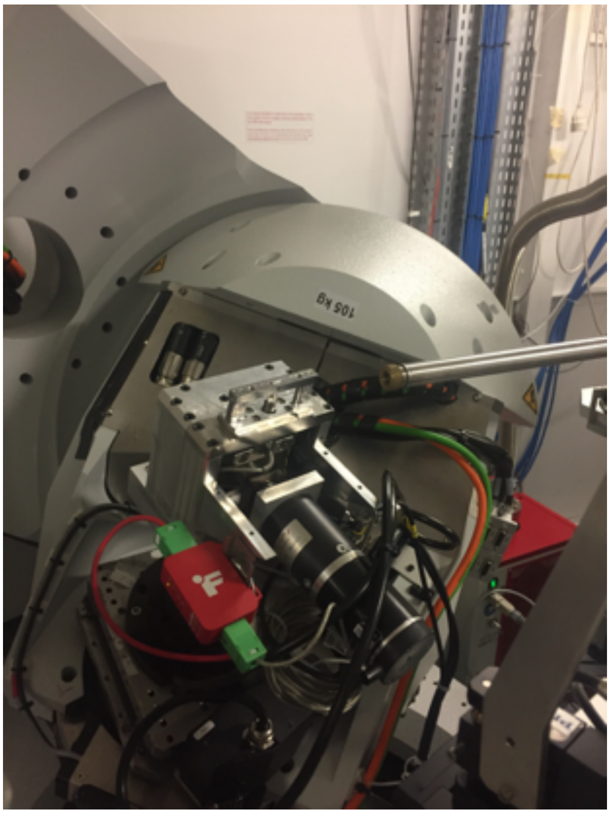

Figure 2 : Bulky tensile testing machine installed on the goniometer of BM02.

The energy of the 25x25 µm² (focused with KB mirrors) incident X-ray beam was fixed at 18 keV. The central part of the sample with an area of 800x400 μm^2 has been mapped in steps of 25 µm for two inclination angles (chi = 90° and chi = 70°) at increasing loading steps. In addition, optical microscopy images have been acquired at each loading step in order to estimate the displacement field on the sample surface from digital image correlation (DIC).
Nine samples in total have been investigated during the beam time (see table 1). Most samples broke at an applied force of about 80-90 N except the Erlangen samples, which exhibited a ductile behavior, as expected for large layer thickness where interfacial effects and confined layer slip are known to be negligible.

Test specimen	Loading direction	Cu thickness (nm)	Nb thickness (nm)
Harbin 1 RD	RD	27	54
LANL RD (1)	RD	16	16
Harbin 1 TD (1)	TD	27	54
Harbin 2 TD	TD	60	120
Harbin 2 RD	RD	60	120
Erlangen RD	RD	3800	3800
LANL RD (2)	RD	16	16
Erlangen TD	TD	3800	3800
Harbin 1 TD (2)	TD	27	54

Table 1 : List of tested specimens during the beamtime..

A typical loading curve force vs time is shown in figure 2 for sample LANL RD (2). The plateaus correspond to holding times at constant displacement during which the central part of the sample was mapped in diffraction. One notices some relaxation of the force during these long dwells.

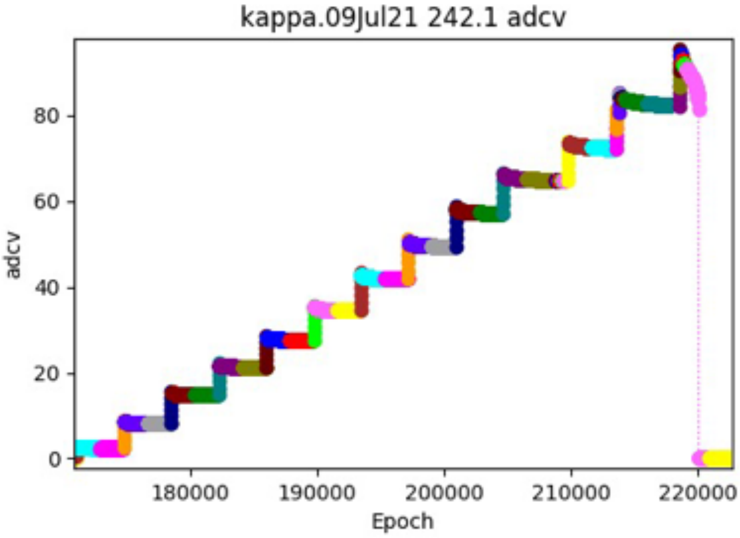

Figure 3 : Loading curve for sample LANL RD (2) that showed brittle failure at about 80 N.

Calibration of the detector has been performed with PyFAI using the diffraction pattern of a standard LaB_6 powder in a capillary placed on the tensile testing rig. Relying on this calibration, detector images are transformed into chi-two theta patterns, which are integrated in chi to yield 1D diffraction patterns displaying intensity vs two theta (see fig. 4).

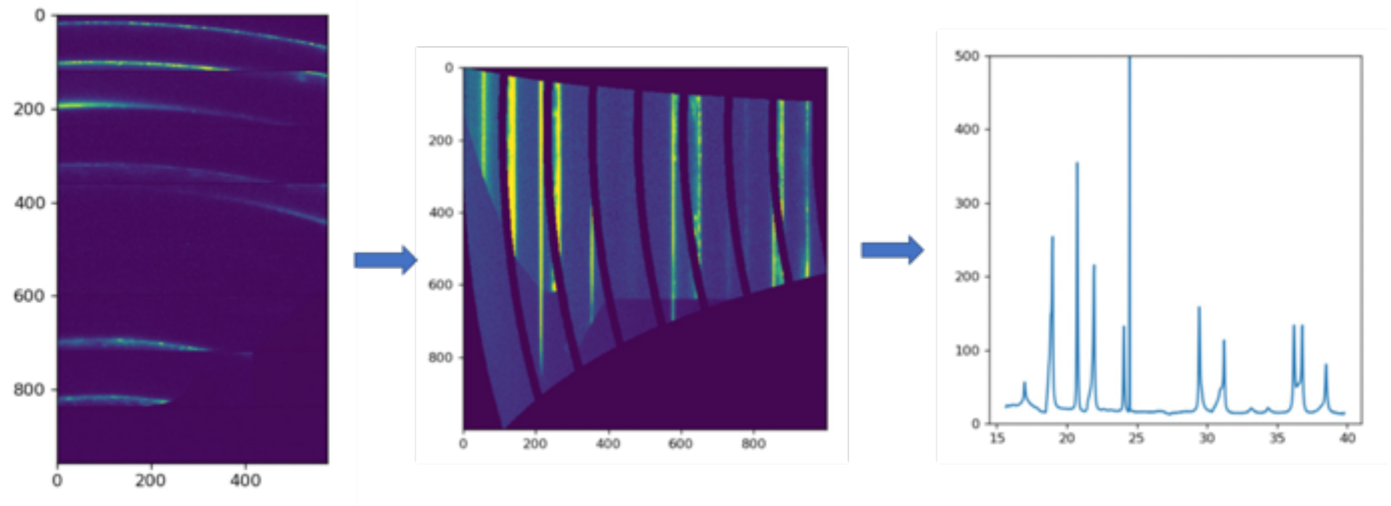

Figure 4 : From 2D detector image (left) to chi-two theta pattern (center) to 1D integrated I(two theta pattern).

From such patterns the diffraction peaks are fitted with a selected function to extract the integrated intensity, the peak position and the peak breadth (see fig. 5) for each pixel of a map.

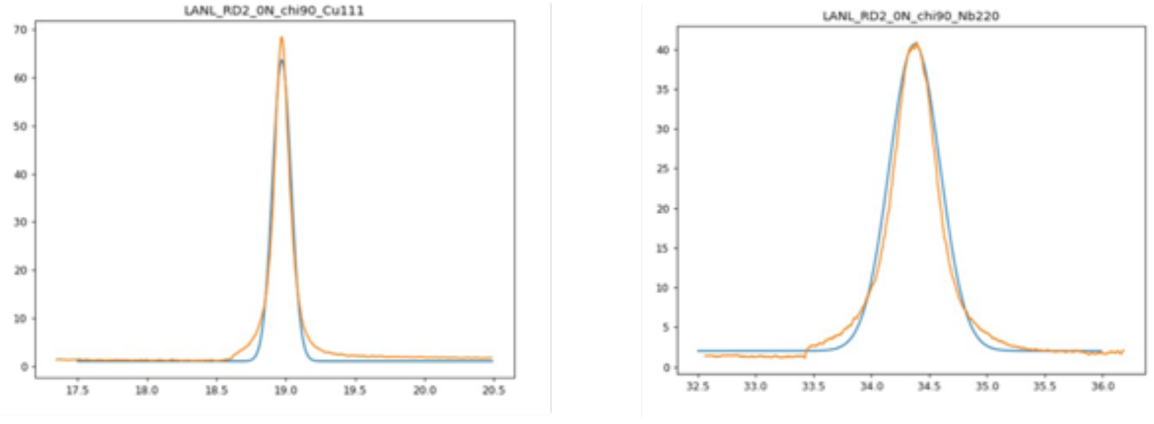

Figure 5 : Example of Gaussian peak fitting for Cu111 and Nb220 Bragg peaks.

The integrated intensity, the elastic strain and the peak width can then be plotted as a 2D map for each loading step (see fig. 6).

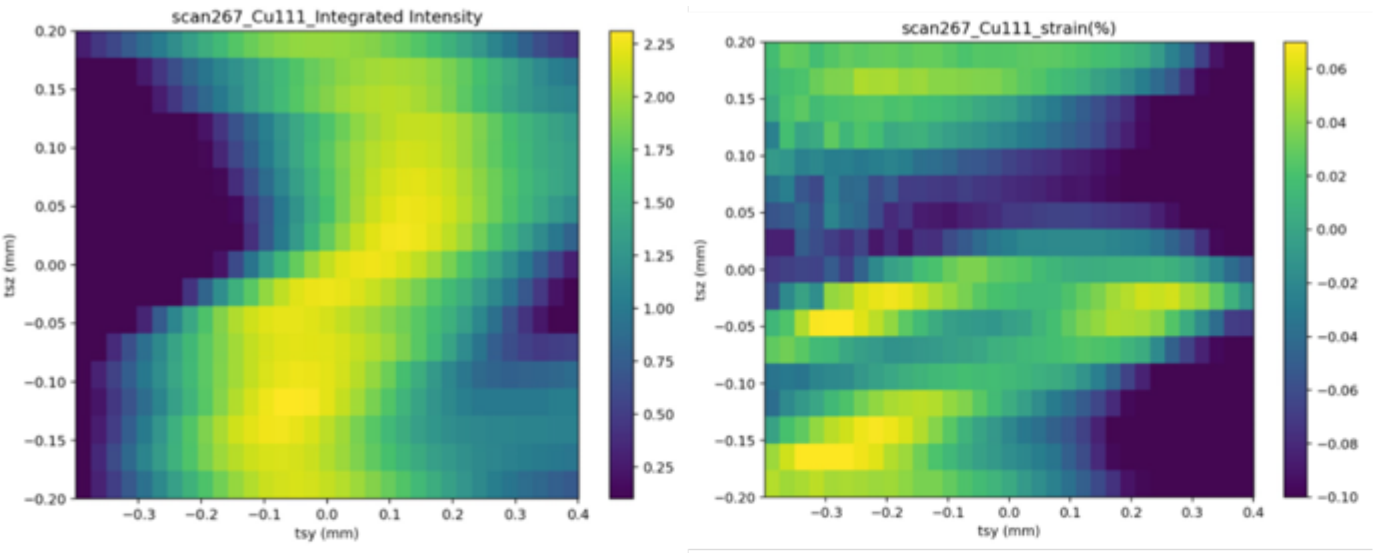

Figure 6 : Cu111 integrated intensity (left) and strain (right) for LANL RD (2), chi = 70°, F = 42 N.

The strain in the center of the sheared zone is shown for Cu in fig. 7 (sample LANL RD (2)). It shows an elastic behavior of the Cu layers for this particular sample. A more rigorous data treatment strategy is under development for determining the average strain in the same zone for each loading step.

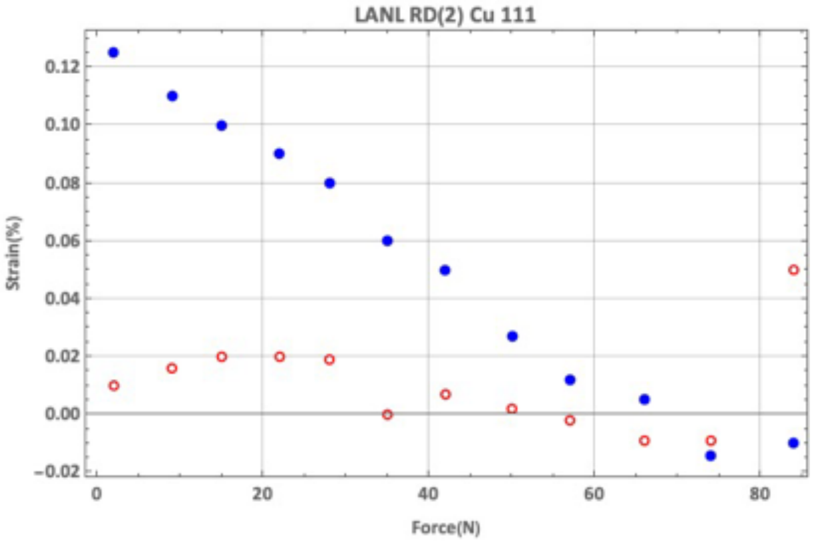

Figure 7 : Strain in the center of the sheared zone from Cu111. Filled symbols : chi = 90°. Open symbols : chi = 70°. Sample LANL RD (2)

Future work includes refining the data treatment strategy, treating all the 9 samples, compare XRD results with DIC results and perform elastic Finite Element Modeling in order to get a full picture of the stress and strain state in the central zone.

References:

[1] M. Pelerin et al., Integrating Materials and Manufacturing Innovation (2019)