THE LAUE MICRODIFFRACTION SETUP

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The applications to micro and nano objects call for intense narrow X-ray beams available at latest generation synchrotron only. The strong demand for structural characterization of objects with ever decreasing sizes has driven the development of new methods to go beyond the average internal structure down to the local structure determination. Probing the local strain, shape (size, orientation) and composition of micro and nano materials are the main objectives of our Laue micro-diffraction setup, this setup being the first in Europe.

To reach its objectives, the Laue microdiffraction setup uses 2 curvature-adjustable KB mirrors to achieve a sub-micron 0.5x0.75 µm2 white beam. This white beam has X-ray energy ranging from 4 keV to continuously adjustable upper energy from 12 to 25 keV. White beam is so intense that collection of a sufficiently intense Laue diagram on any thick grain (>5µm) requires less than 0.5s exposure time (Fig 1).

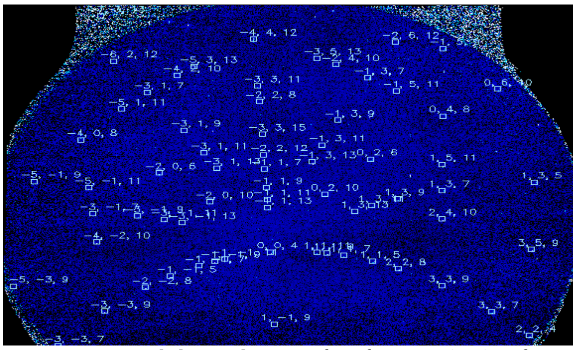

Fig 1: 0.15s recorded Laue diagram of a reference Si(001) wafer. More than 50 Laue diffraction peaks are visible and were indexed.

This sub-micrometer white beam is raster scanned on the sample to perform Laue scattering with sub-micrometer resolution. Laue scattering image are recorded by state-of-the-art large aperture CCD detector. Once indexed, a single Laue scattering diagram fully determines the grain orientation as well as the deviatoric part of the strain tensor (5 out of 6 components). If needed, a micrometric monochromatic beam may be energy-scanned to determine the hydrostatic strain, leading to a fully determined strain tensor. This allows for a grain-by-grain orientation (Fig 2) as well as strain determination with a 2 10^{-4} precision.

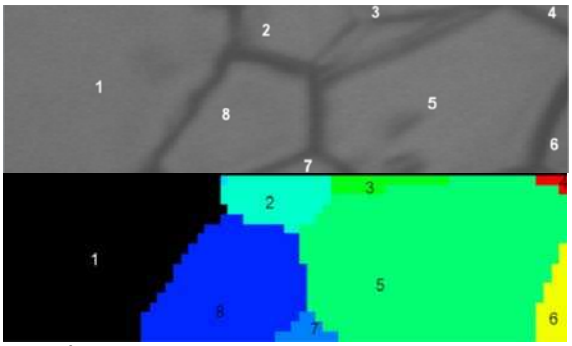

Fig 2: Comparison between our microscope image and grain size and orientation as determine by the white beam raster scan on a UO2 sample. A perfect correlation is clearly visible.

Chronologically, this setup was built along with the beamline refurbishment that started just after precedent BLRP. Given the high technicality and fine tuning necessary to achieve sub-micron white beam, the Laue microdiffraction setup became really operational to external user mid-2007. At that time, it was installed on top of GMT's multi-axis goniometer but we decide to move the setup onto a dedicated support still in used now (Fig 3). Once on this dedicated support, it appeared that vibrations were degrading the overall beam size. A very time-consuming struggle was engaged to find, diagnose and suppress or at least dampen vibration sources: cryogenically assisted pumping on adjacent ID01 beamline was suspended with elastomer sandwiches and the roof primary vacuum pump was anti-vibrated. Finally, we raised the self-resonance frequencies of the setup using reinforcement bars to avoid a very intense 14 Hz vibration frequency due to ESRF's experimental HVAC. This allowed us to achieve an overall 0.5x0.75 µm2 white beam where vibrations account for an estimated 0.25 µm enlarging in each direction.

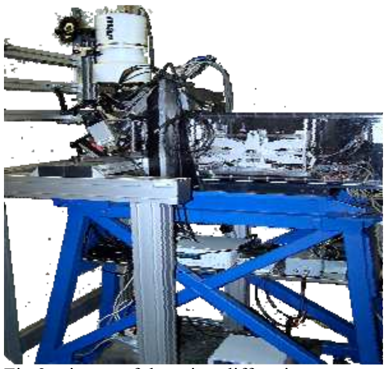

Fig 3: picture of the microdiffraction setup on its dedicated support in blue (cross reinforcement bars are clearly visible). KB mirrors are inside a transparent N2-filled chamber. On the top left, the MAR CCD camera is visible and at 40° our microscope.

The microdiffraction set-up has been fully functional for more than 3 years now and represents approximately 25% of distributed beamtime. Despite drastic spatial constraint, we were able to integrate the necessary specific sample environment for various thematics, namely an oven with temperature ranging from 20 to 400°C for Cu interconnects reliability studies, a precision tensile machine for slip plane studies and a high-precision rotation head for tomography studies

Selected publication(s): not relevant here	
Grant(s): ANR MiDiFaBl	Collaboration(s): not relevant here
Patent(s): not relevant here	Contact: xavier.biquard@cea.fr