ESRF	**Experiment title:** Strain heterogeneities in elastically deformed anisotropic polycrystals, at the micron scale, inferred from white-beam Laue microdiffraction	**Experiment number**: **MI-1035**
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Objective & expected results.

The long term aim of our research is to understand the way polycrystalline materials deform in the plastic regime, with emphasis on the link between microstructure (dislocation structure, crystallographic texture), activated deformation mechanisms at the grain scale (dislocation glide, dislocation climb, twinning), and overall behaviour. When polycrystals are deformed plastically, since individual grains exhibit an anisotropic plastic behaviour, they react differently to the prescribed load, and this gives rise to strong mechanical interactions between grains, with substantial redistribution of stress between "hard" and "soft" grains. Current theoretical efforts aim at predicting quantitatively the effect that these interactions have on the effective strength of a material, and on the evolution of the microstructure during mechanical testing. However there is still lack at present of precise analysis of the corresponding *stress* heterogeneities in the deformed grains, at the relevant (i.e. micron) scale. This is a severe limitation for a deeper understanding of many issues in Materials Sciences, e.g. resistance under fatigue loading, crack propagations, phase transformations, etc.

The goal of this experiment was to measure the stress field evolution during an in-situ tensile test, in a steel of industrial use, with a micrometer spatial resolution. Doing so, we hoped to analyse the mechanical interaction between deforming grains, resulting from the local anisotropy of elastic and plastic behaviors at the grain scale. We thus made use of the Laue microdiffraction technique recently installed at BM32. The other idea of this experiment was to test whether Laue microdiffraction, in its actual level of development, was adapted for application to industrial materials, for which grains can exhibit a large density of defects (in particular dislocations) that generate substantial lattice distorsions. In the field of Mechanics of Materials, there is an enormous field of application of such a characterization, for instance to capture effects of local plasticity, damage, phase transition, etc. in industrial materials. The choice for (stainless) duplex steel, containing ~50% BCC ferrite (alpha phase) and ~50% FCC austenite (gamme phase) was justified by the fact that (i) its microstructure is relatively simple, (ii) the mechanical behavior of each individual phase is relatively well known, and (iii) the different behavior of alpha and gamma phases (in terms of anisotropy, yield stress, etc) gives rise to a high mechanical contrast within the material, thus extending the overall elasto-plastic transition and generating a high level of stress heterogeneity between grains. This is thus a relatively simple material well adapted for our purpose.

The white-beam Laue microdiffraction setup was recently installed at beamline BM32 (ESRF). It allows 2D mapping of local stress in polycrystalline specimens with a (sub)micron spatial resolution. The white beam (energy range 5-30 keV) is focused down to a 0.8 x 1.5 μm^2 cross-section with Kirkpatrick-Baez (KB) mirrors. The specimen is mounted on an x-y-z translation stage at 40° from the incident microbeam, thus allowing scanning microdiffraction with micron spatial resolution. Laue diagrams are recorded on a 2D detector (Photonic science) positioned at 90° from the incident beam and about 7cm away from the specimen. After having indexed the Laue pattern and found the local grain orientation, deviatoric strain can in principle be obtained by a careful analysis of the distortions of the experimental Laue diagram. Local stress is then derived from lattice strain using the elastic constants of the scanned grain.

Results and the conclusions of the study

The experiment ran very well. We could make several 2-D xy Laue scans for about 10 different loading increments, in a region of the specimen selected in advance thanks to EBSD maps performed in the lab prior the ESRF experiment. As for the interpretation of Laue images, we have applied the standard method available in softwares XMAS (developed at the ALS-Berkeley) and LaueTools (developed at ESRF). But it turns out that accurate and trustful maps of local stress could not be obtained, the stress accuracy being generally too high for micromechanical applications. This result come from the fact that Laue spots generally exhibit complex shapes, indicating that grains contain a large density of Geometrically Necessary Dislocations (GND) generating large intragranular misorientations (see figure). Therefore, the fit of Laue spots with analytical function (such as gaussian), a step in the interpretation of Laue data, is not accurate enough. The obtained overall stress level was globally correct, but we could not trust the details of the obtained field.

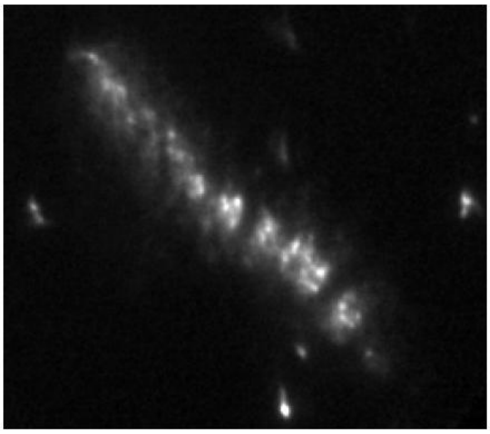

Figure. Zoom on a typical Laue spot. The complex discontinuous pattern indicates that the measured grain contains many sub-grains slightly misoriented with each other.

We believe that advanced image processing is needed to reach the desired accuracy of ~10^{-4} on the lattice parameters, which typically translates into ~0.1 pixel accuracy on the positions of the Laue spots. We are therefore engaged, since about 2 year, in the development of a new data treatment method, denoted Laue-DIC. The method is based on the use of image correlation techniques to get a very precise measurement of Laue spot displacement between two successive loading stages. The absolute stress map cannot be obtained yet with this new method, but rather the map of stress increment between the two stages. The method works as follow:

(i) First, it is necessary to superimpose, with a resolution of ~1 micron, two successive scans measured at two load levels. For this, a dedicated software has been developed and tested.

(ii) Next, an image correlation method, originally used to measure kinametic (displacement) field on the surface of deformed specimen, has been adapted to calculate the relative displacements of Laue spots on the camera, for increasing loading of the specimen.

The method has been tested on data obtained for W polycrystals in an earlier proposal, and more recently on a bent Si single crystal, an very promising results have been obtained (see details in the published papers listed below). However, although we feel confident that Laue-DIC will provide the accuracy adapted to micromechanical studies, with many potential applications on industrial materials, the method now requires a dedicated experiment, on several specimens exhibiting microstructures with increasing complexity (from Si or Ge monocrystals, to more complex multi-crystals) and deformed in very well controlled way. Doing so, we would like to compare results from Laue-DIC with available analytical solutions for the stress field, and get a very robust validation of the new method. We will be able to come back to the data from the present experiment after validation (and probably improvement) of Laue-DIC.

Based on results from this experiment, we have submitted a 4-years research project to the french funding agency ANR, with 6 partners (among them: EDF R&D and the BM32 staff from CEA) to develop Laue-DIC and let it work for plastically deformed polycrystals. The project has been funded, with 3 PhD and 2one-year post docs. It has started in january 2012.

Publication(s).

- Le Bourlot, C. (2012). PhD thesis, Université Paris Nord, France.
- J. Petit, M. Bornert, F. Hofmann, O. Robach, J.S. Micha, O. Ulrich, C. Le Bourlot, D. Faurie, A.M. Korsunsky, O. Castelnau, Combining Laue microdiffraction and digital image correlation for improved stress field measurements with micrometer spatial resolution, *Proc. Int. Union Theor. Appl. Mech. (IUTAM)*, in press.
- J. Petit, M. Bornert, O. Castelnau, D. Faurie, F. Hofmann, A.M. Korsunsky, C. Le Bourlot, J.S. Micha, O. Robach, O. Ulrich, F. Zhang, Laue-DIC: A new method combining Laue microdiffraction and digital image correlation for the improved analysis of stress fields at the micro scale, *J. Synchr. Rad.*, in preparation.