



	<b>Experiment title:</b> Orientation dependent onset of microplasticity and subsequent intragranular hardening in a duplex steel	<b>Experiment number:</b> <b>MA-990</b>
<b>Beamline:</b> ID15B	<b>Date of experiment:</b> from: june 10, 2010 to: june 15, 2010	<b>Date of report:</b> sept 20, 2011
<b>Shifts:</b> 15	<b>Local contact(s):</b> Veijo Honkimäki	<i>Received at ESRF:</i>
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## Report:

### Objective & expected results.

This experiment was dedicated to the investigation of the mechanical response of a duplex steel specimen containing two phases, 50% body-centred cubic (alpha) ferrite and 50% face-centred cubic (gamma) austenite. Duplex steel is of interest for many reasons: (i) the high macroscopic overall yield stress (700MPa) gives rise to large elastic strain and strain heterogeneities, (ii) the composite nature leads to a spreading of the elasto-plastic transition (BCC ferrite and FCC austenite exhibit different yield stress and plastic behaviour), and (iii) there is a relatively good knowledge of deformation mechanisms (dislocation glide and hardening) of both ferrite and austenite grains. Special attention was paid to the stress and strain intragranular heterogeneities at small strain, in particular during the elastic-plastic transition.

### Results and the conclusions of the study

Diffraction-based techniques are ideally suited for such a study since they allow characterizing of the average elastic strains (shift of Bragg peaks) and the strain heterogeneities (peak broadening). A 10.000 N Kammerth tensile rig was installed on phi rotation mounted on top of xyz translations and omega rotation at beamline ID15B. Diffraction signals were recorded simultaneously on the 2D detector (Pixium) placed at 932.36 mm from the specimen. The energy was set at 86.835 keV so that more than 10 *hkl* lattice planes could be recorded simultaneously in transmission geometry (thanks to a hole in the base plate of Kammerth rig). To increase the number of scanned grains and get good statistics for each individual measurement, i.e. for the diffraction volume approaching the size of a Representative Volume Element, the specimen was continuously rocked about omega and phi with an amplitude of typically +/-3 deg. Acquisition time was 5 to 10 sec.

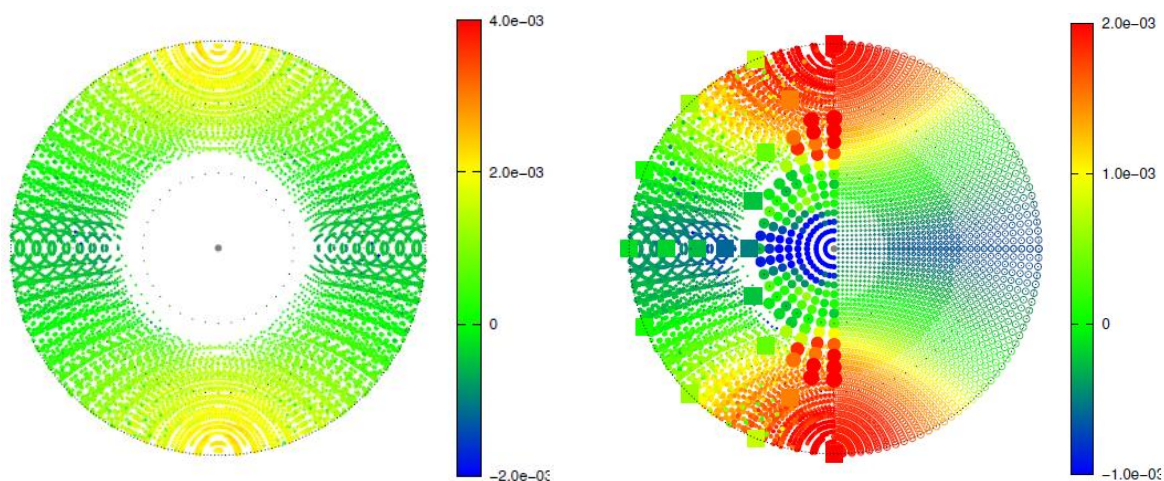
Two kinds of experiments were carried out.

(i) First, in order to characterize the time (and thus strain) dependence of the local material response, data images were obtained during a continuous sample straining. The sample orientation was kept fixed during the experiment, and scattered signal originating from all families of diffracting planes could be measured simultaneously. Diffraction patterns were recorded every 10 s. Considering the signal-to-noise ratio of the data, the maximum intensity of the Bragg peaks, and the photon noise based on Poisson statistics, the variances of the first and second moments of the Bragg peaks (providing the uncertainties for peak position and peak width) could be estimated (Le Bourlot, 2012). We could reach a strain resolution of  $10^{-4}$ , corresponding to a shift or broadening of the Bragg peaks as small as 0.2 pixels. This setup allows a precise investigation of lattice strain evolution during the elasto-plastic transition. An important result for this part was the measurement of a clear decreasing of the FWHM of some Bragg peaks at the beginning of the mechanical loading. By using a micro-mechanical model, we have then shown that this behavior, which was also observed in different contexts (e.g. in thin films) but not really explained, is due to a coupling between the initial residual stresses present in the material and originating from the elaboration process, and the applied stress.

(ii) Second, in order to characterize the orientation dependence of the local stresses, we did interrupted mechanical tests, with about 10 to 15 loading stages for each specimen. After having waiting for ~15-30 minutes to allow the specimen to relax, the specimen was rotated in all possible orientations in order to scan the Euler space, and  $hkl$  planes were measured on the 2-D detector. Doing so, we could calculate strain pole figures, providing the orientation dependence of lattice strain, for various lattice planes, and at various overall deformation. An example of result is shown in the figure below.

The obtained data were then compared to predictions based on mean-field homogenization models (self-consistent scheme) for thermo-elastic and elasto-plastic local behaviors, considering the specimen crystallographic texture, the elastic anisotropy of both phases, and dislocation glide on specific slip systems. Excellent agreement between the model and experimental results could be obtained. It is also worth noting that data have been compared to other diffraction experiments on similar specimens, carried out in transmission (neutrons diffraction) and reflection (12. keV X-rays) geometries, again with a very good match between all data (see figure below).

In summary, this experiment was very successful. It must be stressed that it was a very central experiment for the PhD of Christophe Le Bourlot, defended in jan. 2012. We are actually preparing papers to be submitted for publication in international journals.



**Figure.** Pole figures of mean elastic strain in the diffraction volume, corresponding to the shift of diffraction lines. (left) Data obtained during the present experiment. (right) Data from ID15B combined with neutrons diffraction (large squares) and 12. keV X-ray diffraction (medium dots in the pole figure centre). Data on the right hand side correspond to predictions obtained with a thermo-elastic self-consistent scheme. Peak {211} of the ferrite (alpha) phase, for an overall loading of 400 MPa. Macroscopic tensile direction is vertical.

#### Publication(s).

Le Bourlot, C. (2012). PhD thesis, Université Paris Nord, France.