EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

Experiment Report Form

ESRF	**Experiment title: Stress localization in polycrystalline grains and damage effects studied using synchrotron diffraction and self-consistent model**	**Experiment number**: **MA-1611**
Beamline: ID15B	**Date of experiment**: from: 14.11.2012 to: 17.11.2012	**Date of report**: 11.06.2013
Shifts: 6	**Local contact**: **Thomas Buslaps**	*Received at ESRF:*

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Introduction

Information concerning small and large deformation of polycrystalline materials can be found by using diffraction to measure lattice strains during tensile test [1-3]. During yielding, plastic deformation gradually begins for different grains leading to load transfer between different grains. In this project the lattice strains will be measured using monochromatic synchrotron radiation during in situ tensile test and the lattice strains for groups of grains selected by different hkl reflections will be measured. The aim of the work is to determine differences in stress localisation for these groups of grains for one or two phase materials. To do this a new interpretation of experimental data has been proposed and already tested using TOF neutron diffraction data [1,2]. The experimental results obtained at ID15B (ESRF) will be applied to verify localization scheme and slip system activation in elastoplastic models. Using synchrotron high energy radiation, due to short acquisition time, the diffraction pattern can be measured directly during tensile test, without breaks and sample stabilisation. The second aim is the study of damage process by scanning lattice strains in the deformation neck with a small sampling gauge.

Report:

Mechanical behaviour of following types of steels: low carbon ferrite (one sample), two phase duplex stainless steel, pearlitic steel consisting of hard cementite within soft ferritic matrix, was studied. The "dog bone" shape samples having dimensions suitable for TOMO stress rig (2 mm width x 1.5 mm thickness = 2.25 mm^2 cross section) were measured at ID15B beamline. In situ tensile test was performed with beam size of 100x100 μm in transmission mode (average through the sample thickness) with monochromatic 87keV radiation. The diffraction pattern was recorded using detector PIXIUM4700 2D detector (2Θ range: 0° – 8° covered the main reflections from the measured steels) in order to measure lattice strains in the direction of loading and in transverse direction.

<u>The following experiments were performed:</u>

- For each material first the initial residual stresses were measured and after that the measurements were done in situ during tensile test. The first experiment was performed for the perlitic steel specimen. The tensile tests with the load control were carried until fracture of the specimen and also up to large deformation but before fracture. The same test was performed for the specimen made of two phase duplex steel for two different sample orientations with respect to the scattering vector.
- The experiment of the second type, carried out at ID15B, was the measurement of residual stresses in broken (after fracture) and not broken specimens. It was performed for all tested specimens. In case of duplex steel deformation neck was scanned in two perpendicular directions.
- Third type of experiment, performed for pearlitic steel and for the duplex steel was the position control test with different displacement speed. After this test the significant necking with heterogeneous strain was observed for duplex steel specimens.
- The last experiment was the load position tensile test with brakes allowing relaxation of the stress state of the specimen. Measurement were done both during the loading and relaxation process.

The example detector images for initial state of specimen and after the tensile test are presented in the Fig.1.

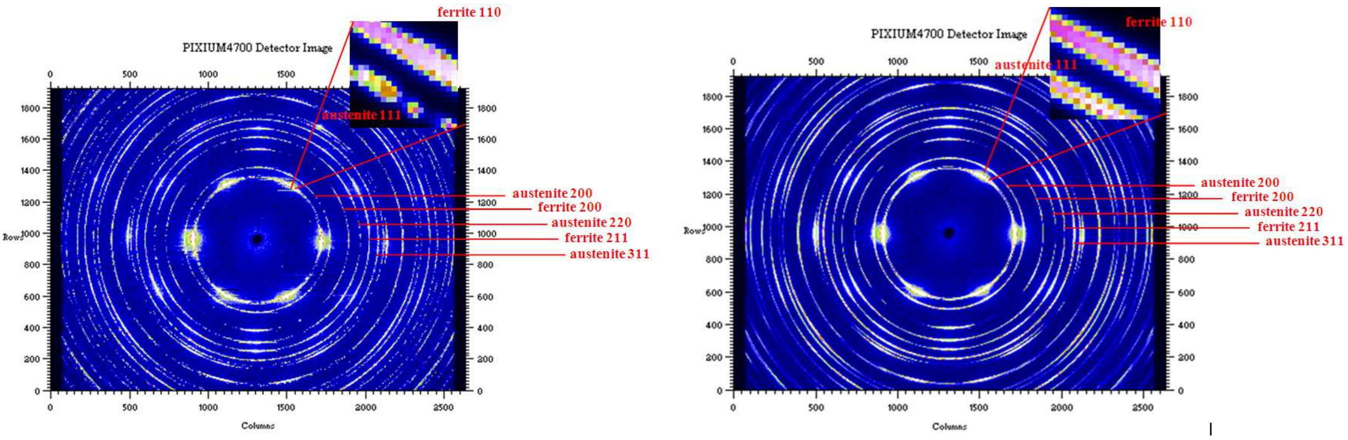
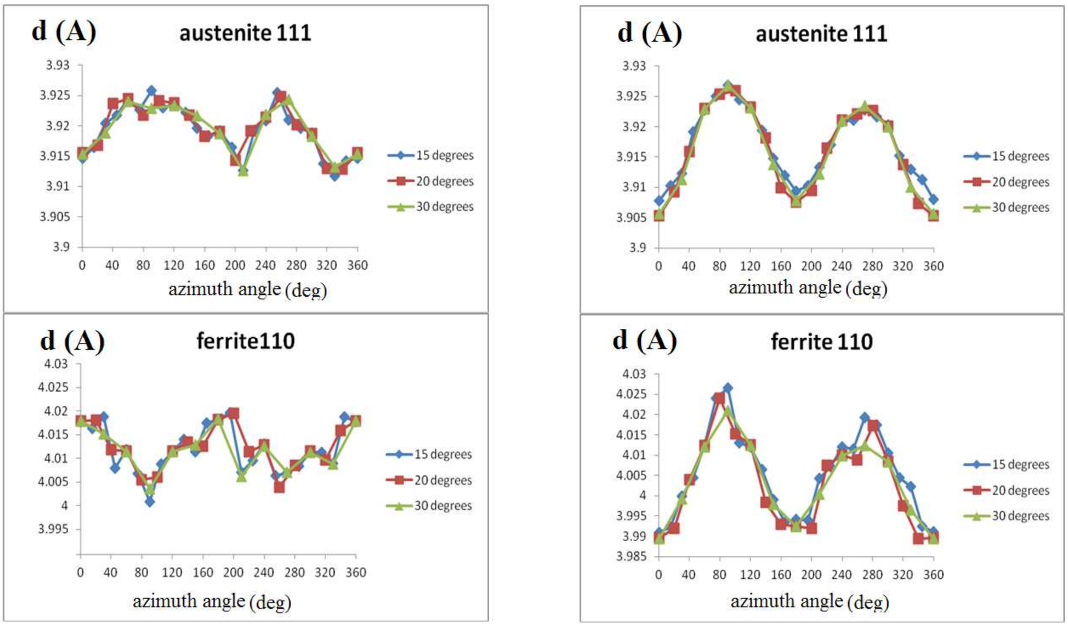


Fig. 1 The detector images for duplex steel a) before the tensile test and b) after the tensile test.

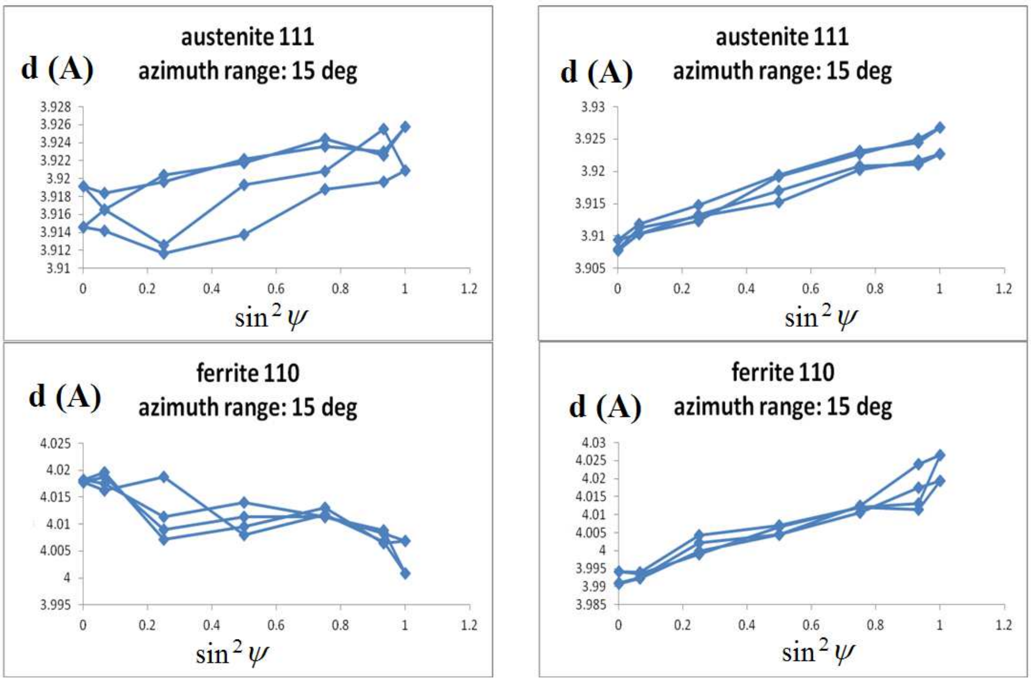
The first aspect of data treatment was the choice of the optimal integration range for azimuth angle in the images shown in Fig.1. Three integration ranges: 15, 20 and 30 degrees were tested. The comparison of interplanar spacings for four states of the specimen are presented below, i.e. before the tensile test in Fig. 2a and after tensile in Fig. 2b. It was decided that for the further analyze the integration range equal to 15 degrees will be used, but it can be concluded that the choice of even twice larger range does not change significantly the results.



a) **b)**

Fig. 2. Interplanar spacing d vs. azimuth angle integrated over different ranges (15^o, $20^{\ o}$ and $30^{\ o}$): a) specimen before the tensile test b) specimen after the tensile test.

Next, the comparison of the interplanar spacing vs. $\sin^2\psi$ for unstressed specimen (Fig 3a) and for the same specimen under applied load (Fig. 3b) was done.



a) **b)**

Fig. 3 . Interplanar spacing d vs. $\sin^2\psi$ for a) specimen before the tensile test b) specimen during the tensile test.

The presented plots show that before the tensile test the stress values in both phases is not significant, and during test the tensile stress appears. The measurements are accurate enough to estimate residual stresses in both stages.

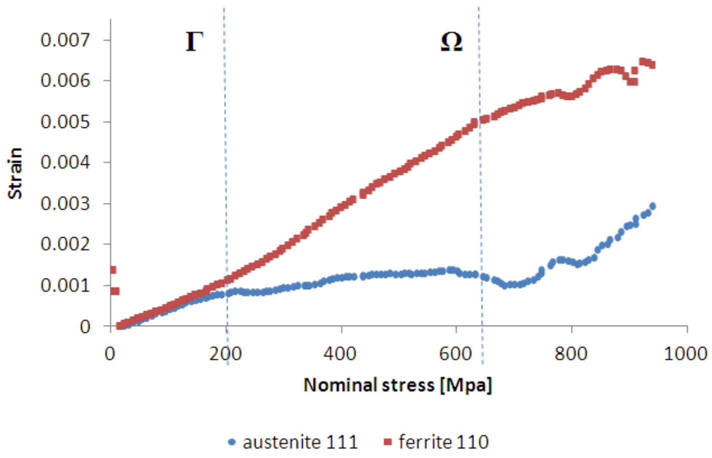


Fig. 4 a) Lattice strains measured using 111 reflection in austenite and 110 reflection in ferrite vs. nominal stress for duplex steel (ESRF, ID15B)

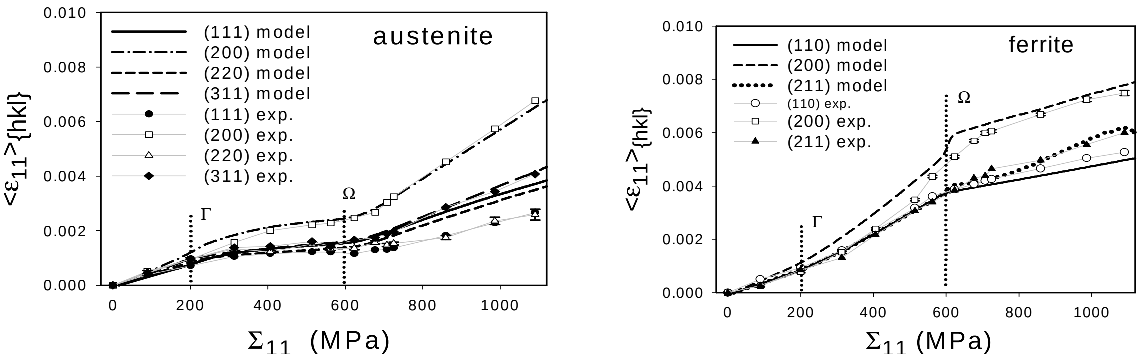


Fig. 4. The elastic lattice strains $<\varepsilon_{11}>_{\{hkl\}}$ parallel to the load direction versus macrostress Σ_{11} for both phases of the duplex steel (the ISIS experiment performed on ENGIN X [1]).

The aim of the experiment is to determine the stress localization tensor and to study elastoplastic deformation in different phases of the studied materials. The ‘in situ’ tensile test performed for duplex steel specimen shows that initially both phases (until the value Σ_{11} < 200MPa) were below the yield point – only the elastic deformation took place. It can be seen that the yield point for austenite is about 200MPa (marked by Γ). Above this value deformation of the austenite is plastic while the deformation for the ferrite is still elastic. The next characteristic threshold is the Ω point (Σ_{11} > 650 MPa). It can be identified as a yield point for ferrite. Above this point both phases are deformed plastically. The measurements performed in ESRF confirm the results obtained in ISIS (Fig. 4b).

The data obtained for different reflections during tensile tests performed for pearlitic, ferritic and duplex steels are currently treated. The quality of the results are enough to see evolution of lattice strains during elastoplastic deformation. Next step of the analysis is to determine the localization tensor for different phases in the studied duplex steel and pearlitic steel. Also, the effects of damage occurring in broken samples is studied. Additionally, during experiment also preliminary diffraction tests for such materials as metal matrix composite Al/SiC, single phase Ti were done in order to prepare further experiments concerning stress localisation

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