ESRF	**Experiment title:** Measurement of stored energy in cold-deformed iron	**Experiment number**: 02-02-666
Beamline: D2AM (BM02)	**Date of experiment**: from: 24/11/2005 to: 29/11/2005	**Date of report**: 17/04/2006
Shifts: 15	**Local contact(s)**: N. Boudet	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

A. Wauthier*, LPMTM – CNRS (Villetaneuse, France)

O. Castelnau*, LPMTM – CNRS (Villetaneuse, France)

T. Chauveau*, LPMTM – CNRS (Villetaneuse, France)

H. Réglé, Arcelor Research SA (Maizières-les-Metz, France)

J.-L. Uriarte*, Arcelor Research SA (Maizières-les-Metz, France)

Report:

During the deformation of low carbon steel by cold-rolling, the shape and the orientations of the grains change relative to the direction of the applied stress and some dislocations are continuously created. In the frame of the PhD of Aurélie Wauthier (2004-2007), which concerns the understanding and modelling of the recrystallization phenomena in ultra low carbon steels, samples have been deformed by cold-rolling with different thickness reductions (from 15% to 93%). These samples have been characterised in term of crystallographic texture, and their physical and mechanical behaviour during the recrystallization process has been investigated. During recrystallization the new grains form preferentially in the grains that are mostly deformed, i.e. the grains which have stored the highest amount of dislocations during deformation.
The purpose of this experiment is to measure the dislocation densities in some specific crystallographic orientations and for a number of selected cold-rolling levels. For a quantitative and statistically representative analysis, the analysis of the broadening of the X-ray diffraction lines, when obtained with a high resolution setup, is perfectly adapted as shown in [1].

Investigated samples

Six specimen of ultra low carbon steel have been measured:

- three cold-rolling levels are considered: a quite low reduction (29%), a severe one (93%) and a rate frequently used in industry (76%)
- two different states for each cold-rolling level: the deformed one (samples named 29D, 76D, 93D) and the restored one (10min at 600°C) (samples 29R, 76R, 93R).

Specimen have been polished at mid-plane thickness and the deformed samples have been previously investigated in the laboratory by diffraction of classical X-rays (Cobalt target) in order to get their global textures.

Experimental conditions and analyses of X-ray profiles

The samples were mounted in the 7-circles diffractometer of BM2. Diffraction in Bragg geometry has been carried out at an energy of 20keV. The corresponding attenuation length is 50µm for iron, so it is larger than the grain size even for the less deformed sample (about 15µm). Moreover, the beam size was selected at 1mm^2 in order to scan a large number of grains simultaneously.
First, a reference powder of CeO_2 was analyzed in order to characterize the resolution of the setup (FWHM of about 0.018° in the investigated 2θ range). Then, for each sample, 10 positions on the {110} pole figure and 11 on the {200} one were reached. The investigation of two orders of each pole figure is necessary to separate size and strain broadening effects, so the diffracting planes {110}, {220}, {200} and {400} were analyzed, what means 42 peak measurements per sample. For each measured θ-2θ profile 310 points were acquired in order to have 10 points describing the peak itself and 300 points describing the intensity decay in the peak tails, so 30 times the FWHM (2θ amplitude ranging between 0.9 and 2.9°). Counting 1s per point was necessary to acquire a good statistic on the {110} and {200} peaks (ratio signal/noise about 10^3). But for the {220} and {400} ones, 3s per point was needed for the less deformed samples and 9s for the others. The total acquisition time per sample was then between 12 and 17 hours.

Results

After the background treatment, each profile was analyzed on the basis of the Fourier transform. As an example, the evolution of integral breadths for the diffracting planes {200} and {400} of all samples and the powder is given in figure 1. Regarding the low values obtained for CeO_2 we do not take into account the instrumental part of the X-ray line profiles for the end of the analysis.
Line profiles are analyzed with the Modified Williamson-Hall and Modified Warren-Averbach methods proposed by Ungar and Borbely [2]. For the same position than previously, the results obtained with the Modified Williamson-Hall method in term of dislocation densities (ρ) are given in fig. 2. The grain size is decreasing while the dislocation density is increasing with the strain level and these two effects are less pronounced for the restored materials than for the deformed ones.

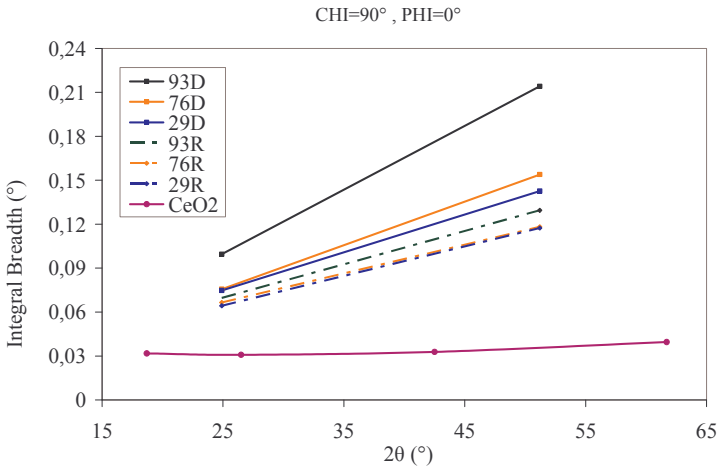

Fig. 1: Evolution of integral breadth with the bragg angle for the reference powder, the deformed samples and for the restored ones (in dashed lines). Measure in the center of the poles figures, corresponding to a maximum texture intensity for the samples.

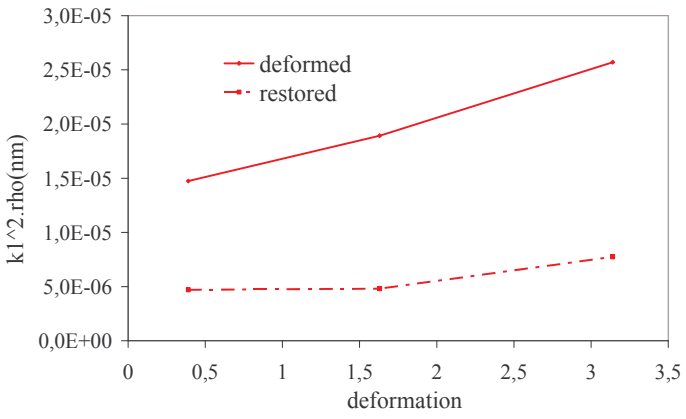

Fig. 2: Relative dislocation densities evolution with the strain level (Modified Williamson-Hall method).

The obtained data are of good quality and the beamline well matches the requirements of the study, these 15 shifts have given us the opportunity of analysing the six specimen chosen.
Unfortunately we didn't had time to investigate some specific orientations. This could be done in a next experiment.

The interpretation of the data is still not completed at present but final results will be published as soon as possible.

[1] I. Groma, Phys. Rev. B, 57, 13, pp 7535-7542, 1998
[2] T. Ungar, A. Borbely, Appl. Phys. Lett., 69, 21, 3173-3175, 1996