DESY SUMMER STUDENT PROJECT

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Texture analysis of low-carbon packing sheets with high-energy synchrotron radiation

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Photon Physics, Material Science

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Introduction

Packing high-quality steel belong to the progressive types of steel. From other commonly manufactured steel products differ only by their low thickness, the characteristic mechanical properties, types of plating, but also their specific industry and market requirements such as high production speed requirements for the design and so on. Modern packing sheets can be used by manufacturers for the purpose of low-carbon aluminum-killed steel, effervescent. They are also subject of this work.

X-ray diffraction experiments at high photon energies are characterized by relatively small Bragg angles of the order of only a few degrees and the use of synchrotron radiation provides high intensities in the diffraction signal. The use of two-dimensional detectors is of interest for simultaneous registration of many diffraction lines. Hence, the two-dimensional image plate area detector provides a sufficient angular resolving power for quantitative texture analysis and a convenient digital intensity measurement. The detector output data file allows the application of more sophisticated mathematical methods for the analysis of diffraction data including profile analysis [1-5].

Crystal orientation and texture

Each grain in a polycrystalline aggregate has normally a crystallographic orientation different from that of its neighbors. Considered as a whole, the orientations of all the grains may be randomly distributed in relation to some selected frame of reference, or they may tend to cluster, to a greater or lesser degree, about some particular orientation or orientations. Any aggregate chracterized by the latter condition is said to have a preferred orientation, or texture, which may be defined simply as a condition in which the distribution of crystal orientations is nonrandom. Preferred orientation can have a profound effect on diffracted intensities measured by diffractometry.

In a case of the packing sheet it is desirable to achieve the situation that grains are aligned so that the normal of the (111) planes coincides with the normal of the sheet. Such texture ensures ideal conditions for further processing, typically in cases of pressing or deep drawing. On the other hand the worst features of the other metal processing occurs when the type of texture (100) is formed. The preferred orientation that is produced by the forming process (sheet rolling) is called a deformation texture. During the cold-rolled microstructure has been breached due to the high plastic deformation. Microstucture of deformed grains is characterized by flattened grain dragging. Cold rolling should be usually followed by recrystallization annealing which yields specif microstructure showing desired properties.

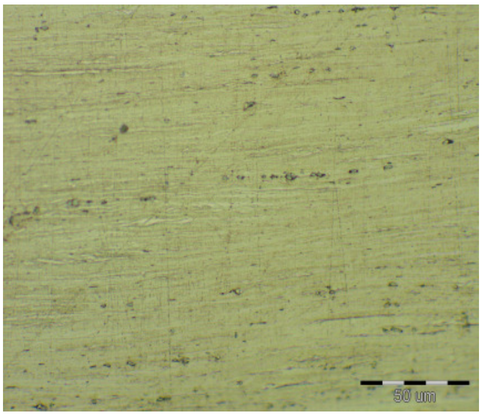

Figure 1: Microstructure of deformed sheet.

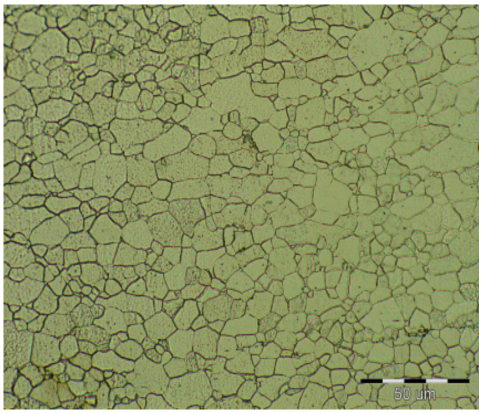

Figure 2: Microstructure of recrystallizated sheet.

Preparation of samples

Samples used in this study were prepared cold rolling of low carbon Al-killed steel, whose chemical composition is given in Table 1. Slabs were rolled to the wide-track, consisting of five counties perfect fourth pre-order and seven counties finish order. Finishing temperature was (T_{fin} = 870 ˚ C) and coiling temperature was ($T_{coiling}$= 720 ˚ C). The finishing temperature and coiling temperature, are relatively high. Definitely we can say that the high temperature causes exclusion of AlN during the coiling process. Rolling was followed by pickling the sheets. Pickled hot bands were cold rolled to five stand tandem with the overall reduction of more than 90%.

Chemical composition [wt %]						
C	*Mn*	*Si*	*P*	*S*	*Al*	*N*
0.0250	0.253	0.008	0.008	0.0069	0.043	0.0034

Tab. 1: Chemical composition

Experimental samples were taken in the direction identical with the direction of rolling. Samples were taken with the scissors table and its dimensions were 40 x 20 x 0.2 mm. Before annealing the samples were subjected to defatting given the aggressive nature of the annealing salt mixtures.

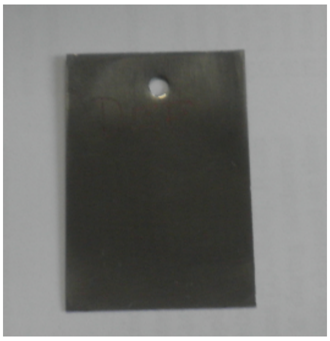

Figure 3: Typical sample geometry.

Recrystallization annealing was carried out under laboratory conditions in a resistive crucible furnace brand ESA Prague type K59. As the heat carrier was used for non-toxic salt mixture-type annealing NETOX SZ 600. It is a non-toxic mixture, working in the form of melt in the temperature range 540 to 800 ˚C with a protective effect against scaled surface. The course of the process of recrystallization has been studied in isothermal conditions (600˚C, 700˚C, 800˚C) and different annealing time intervals. Prepared samples were suspended on wires and ongoing input into the work area furnace heated to the desired temperature for selected time intervals, immediately after calcined, the samples were tarnish in water to stabilize and fix a microstructure. The texture measurement experiments were carried on six sheets which were annealed at different temperatures and times. As a reference cold rolled sheet was used.

High-energy X-ray diffraction at BW5

Wriggler beamline BW5 can produce photon beams with energies ranging between 60 and 150 keV. This facility is dedicated to X-ray scattering experiments. Due to high penetration depth of high energy photons, bulk samples having thickness several mm (depending on material composition) can be investigated in transmission mode. Synchrotron radiation at the BW5 station is produced by wiggler, which emits high-energy photons in a relatively wide horizontal fan. Incoming beam is partly diffracted on sample. Diffracted photons are collected using two-dimensional image plate detector MAR345. This detector has resolution 2300x2300 pixels and each pixel has a size of 150x150 μm^2. During experiments the beam energy was set to 80 keV, which equals to the wavelength λ = 0.155 Å. During measurement the samples to detector distance was fixed to 747 mm. The sample was clamped as shown in Fig.4. In order to determine the texture of rolled sheets X-ray diffraction patterns were collected upon rotation of the sample around vertical axis which denotes its rolling direction (see Fig. 5). The ω was scanned in the range between -80° and +80° with the step of 2°. The ω = 0° refers to the situation when beam hits the sheet perpendicularly. Since we used two-dimensional detector there is no need to rotate the sample around the beam as denoted by angle φ in Fig.5.

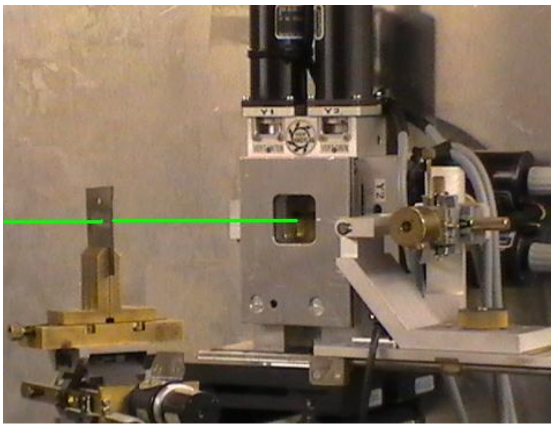

Figure 4: Detailed view on the clamped sample.

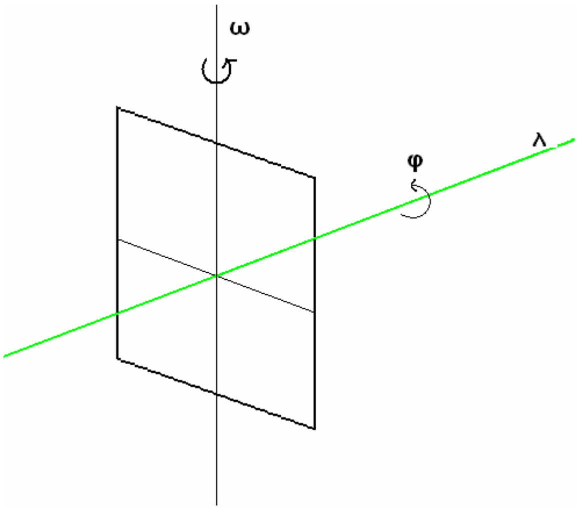

Figure 5: Sketch of the sample and coordinate system.

Data treatment

The texture analysis using high-energy x-ray involved several steps. On each sample we acquired 81 two-dimensional XRD patterns. Altogether six samples were measured. Evaluation of the texture was done by two apporaches: i) first by combination of Fit2D [6] and own macros written in Octave [7]; ii) and by MAUD (Materials Analysis Using Diffraction) program [8]. The first apporach was more illustrative so I could actually learn how the determine the pole figures step by step. The second approach relied completely on the work with the MAUD program which is a kind of standard program used for the texture analysis.

In the first approach two-dimensional XRD patterns were caked using the program FIT2D in 72 pieces each having the opening of 5° (see Fig. 6). Figure 7 shows resulting intensities obtained by radial integration of corresponding cakes. This procedure was repeated for each ω position so in total series of 5832 (= 72 x 81) curves (similar to the ones presented in Fig.7) were obtained. Further we analysed the evolution of the integral intensities for selected Bragg reflections (110), (200), (211) and (220) with respect to φ and ω. Figure 8 shows Evolution of the integrated intensity with φ and ω for the (110) reflection belonging to bcc-Fe in a case of sample annealed 600 °C for 5 min.

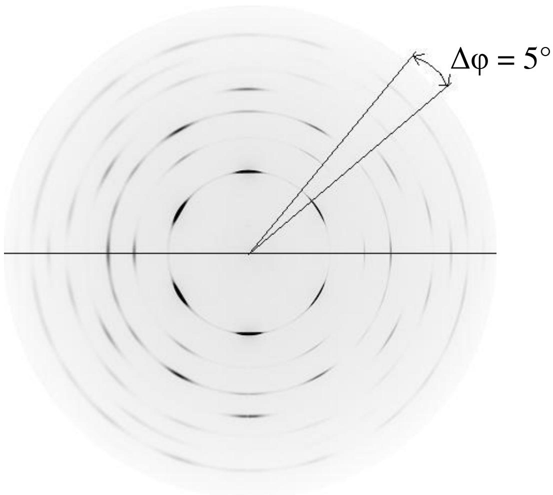

Figure 6: Typical 2D XRD pattern exhibiting relatively strong texture.

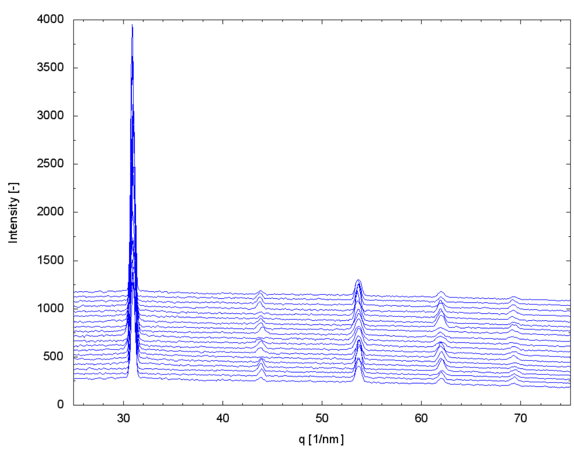

Figure 7: Series of integrated intensities along different azimuth directions φ at fixed ω.

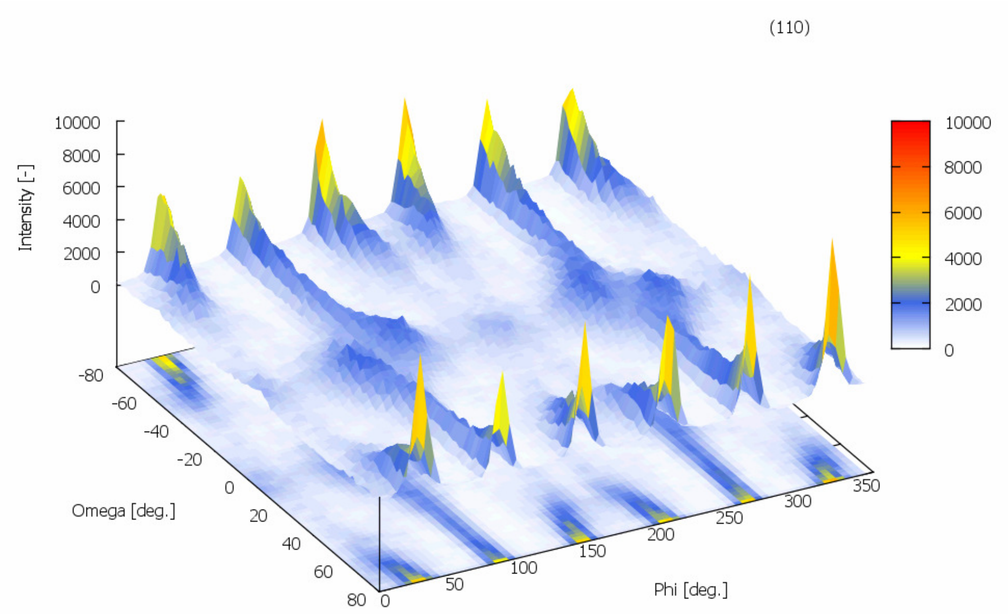

Figure 8: Evolution of the integrated intensity with φ and ω for the (110) reflection belonging to bcc-Fe in a case of sample annealed 600 ° C for 5 min.

Results and Discussion

X-ray diffraction using high-energy photons was used to study the texture of rolled sheets. All samples reveal relativley large extent of texture which is manisfested by intensity variations along the Debye-Scherrer rings. As an example Figures 9 and 10 shows pole figures of selected Bragg reflection belonging to bcc-Fe phase in a case of rolled sheet without additionl annealing and after annealing at 600 °C for 5 min, respectively. Analysing the pole figures indicates that the final texture is strongly affected by additional heat treatment.

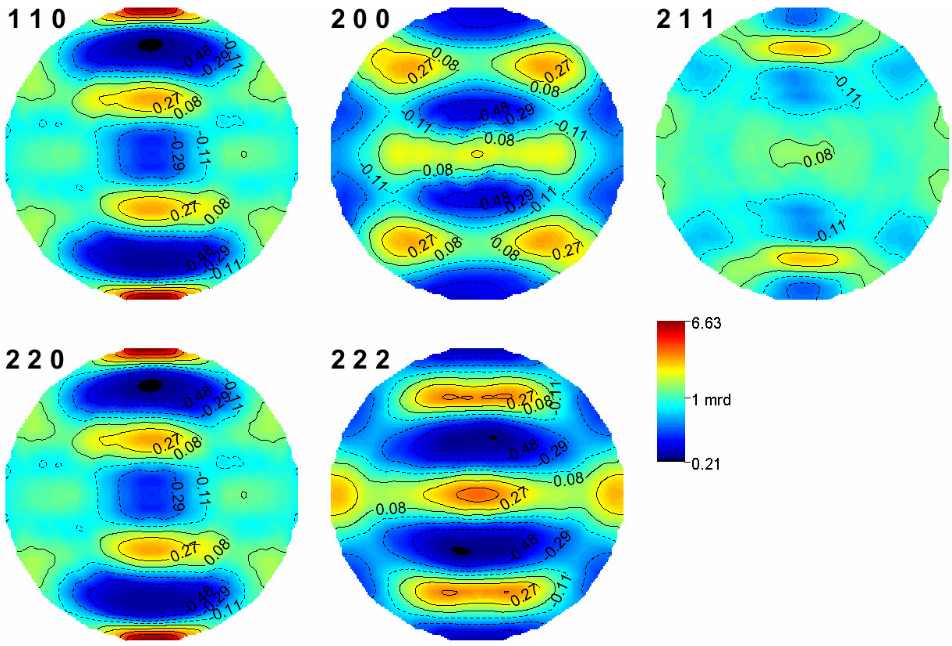

Figure 9: Pole figures for selected Bragg reflection of bcc-Fe in case of rolled sheet.

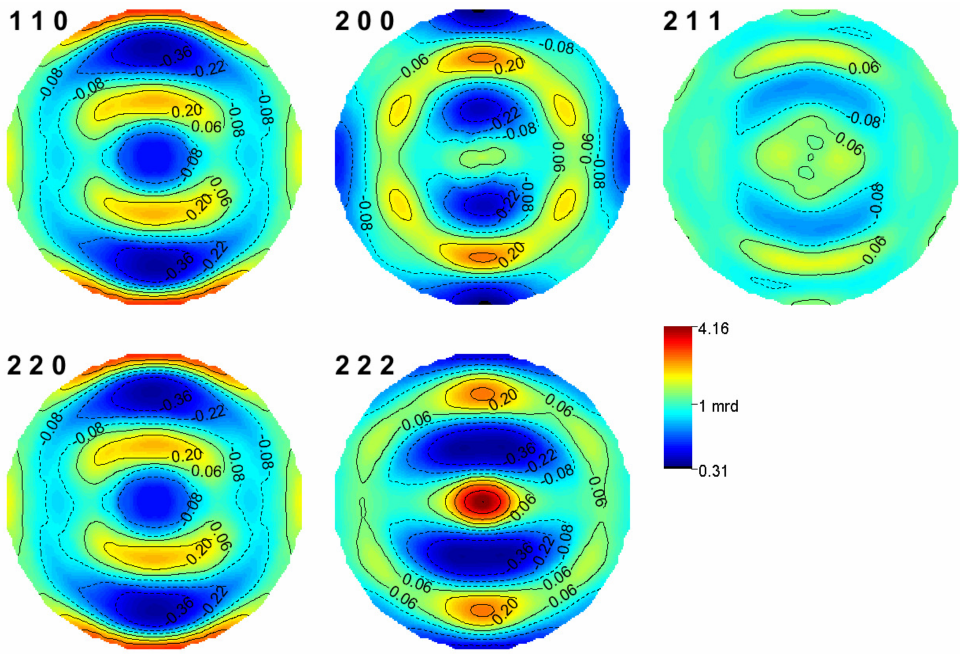

Figure 10: Pole figures for selected Bragg reflections of bcc-Fe in case of rolled after annealing at 600 ° C for 5 min.

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