

Measurement of High-resolution Recrystallization Textures in Nickel sheets using High-energy Synchrotron Radiation

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Abstract. The new developed “sweeping detector” techniques using high energy synchrotron radiation allow to measure textures and microstructures of materials and their change during heat treatment with high location and orientation resolution.

Here we show these new methods applied to cold rolled and subsequently annealed nickel samples. The grain-resolved measurements show, impressively, many details of the recrystallization process which can otherwise not be seen. The results of these measurements can be the base for comprehensive recrystallization theories.

Introduction

In the classical sense the texture is defined by the volume fraction of crystallites with the orientation \mathbf{g} . This definition does not include the microstructure, i.e. the position of the grains in the material. The complete description of a polycrystalline aggregate is given by the “orientation-location” function $\mathbf{g}(\mathbf{x})$ which specifies the orientation \mathbf{g} in any small volume element at the position \mathbf{x} . This quantity can be measured by location-resolved diffraction of high-energy x-rays (short wave) from synchrotron sources. The classical texture i.e. the orientation distribution function (ODF) of the crystallites does not distinguish individual grains, i.e. their sizes, shape and arrangement in the polycrystalline aggregate [1,2,9]. Hence, the classical texture is not sufficient in order to understand the formation mechanism of recrystallization textures, particularly the difficult interaction processes during dynamic recrystallization [2,7].

The six-dimensional orientation-location space must be imaged with the highest possible orientation- and location resolution. This can be done by location-resolved diffraction of high-energy synchrotron radiation [3,4]. In order to fully exploit the high resolving power of this radiation, the conventional step-scan technique had to be replaced by a continuous “sweeping” technique with a moving area detector [3,5,6].

For this purpose either a diffraction angle slit or a diffraction plane slit was introduced additionally between sample and detector in the diffractometer at the high-energy beam-line BW5 at HASYLAB at DESY, Hamburg, Germany [3,5]. During exposure the detector is being shifted continuously while, at the same time, the sample is being rotated, translated or annealed, continuously, too. This technique allows to measure four types of two-dimensional images which are sections and projections of the six-dimensional orientation-location space (fig. 1) [10].

Method	Orientation			Location			Environment
	ω	γ	ψ	X	Y	Z	Temperature
a	↔	↔	—	—	—	●	—
b	●	↔	—	—	↔	●	—
c	●	●	—	—	↔	↔	—
d	●	↔	—	—	●	●	↔

↔ continuous image
 ● integration over (projection)
 — fixed value (section)

Fig. 1 The imaging conditions of the four "detector sweeping" techniques in the six-dimensional orientation- location space.

The studied material

The four different methods were used to study orientation, orientation changes and local distributions of crystallites in cold rolled and subsequently recrystallized Ni sheets. The sample size of both materials was 50mm x 50mm, but larger sample sizes (150mm x 150mm) can be used in order to measure local texture distributions. The Ni was cold rolled with a deformation degree $\eta = 93.3\%$ to a thickness of 0.3mm and subsequently ex-situ annealed at 600°C with different times from 20min to 300min and annealed in-situ with a constant heating rates.

Experimental results

Nickel ex-situ measurements. In Figs.2 a and b the (111) pole figures of the Ni sheet are shown. Method "a" was used to measure the pole figures with an high angular resolving power. Starting texture was the typical cold rolling texture. After 45min heat treatment single peaks corresponding to individual recrystallized grains occur (fig.2a). Beside the individual grains there are areas of continuous intensity distribution which may be attributed to plastically deformed material. The spots of the recrystallized grains cumulate around the poles of the cube texture but they are also spread out over a wide range of the whole orientation space. In fact, there are virtually no empty regions after 300 min annealing (fig 2b).

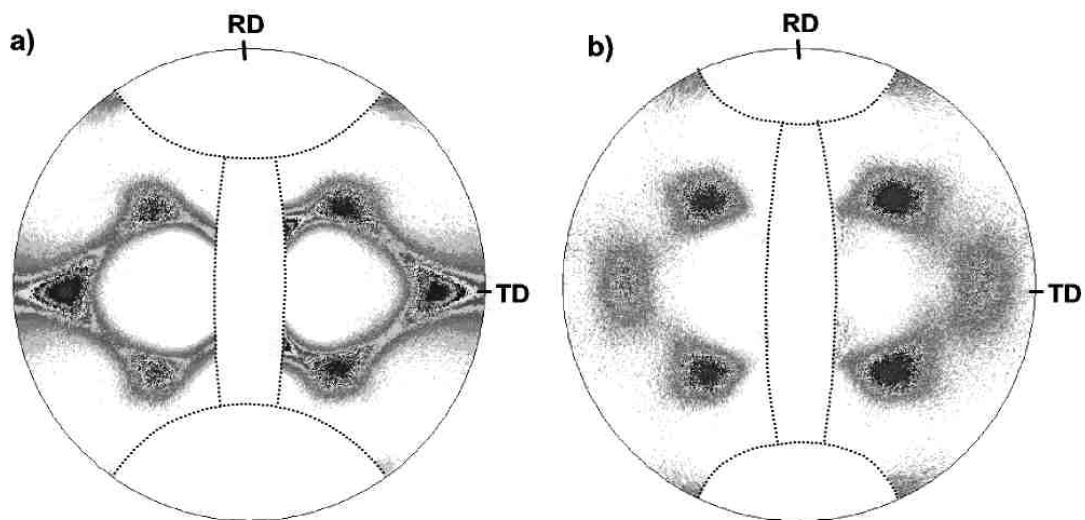


Fig. 2. High resolution (111) pole figures (method a) of a cold rolled Ni sheet annealed at 600°C at different annealing times: a) 45min and b) 300 min. Non-measured areas are marked by dotted lines.

Nickel in-situ measurements. These measurements were carried out in a vacuum furnace in the range from 22°C to max. 800°C with a constant heating rate. Method d (fig.1) with the Bragg-angle slits was used with different but constant orientation angles ω , the (111) reflection was chosen. So It is possible to follow the texture change in γ during the annealing in a fixed local position (Y,Z). In beam direction Z there is an integral measurement over the sample thickness. The fixed orientation angles ω were chosen to 55°, 18° and 0°. The starting cold rolling (111)-pole figure is shown in figure 3 in the detector co-ordinate system. In figure 3 the three ω positions are plotted as dotted lines.

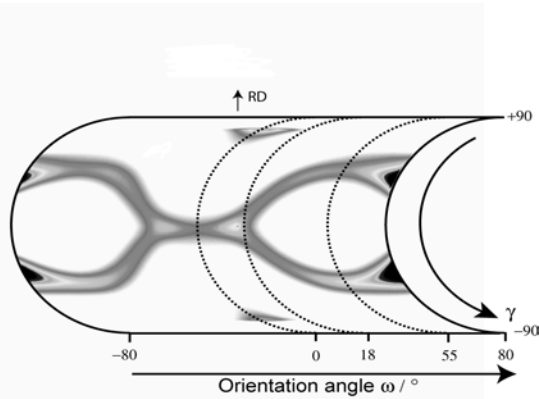


Fig.3. (111)-pole figure of a cold rolled Ni-sheet in the detector co-ordinate system. The positions at $\omega = 0^\circ$, 18° and 55° are marked by dotted lines.

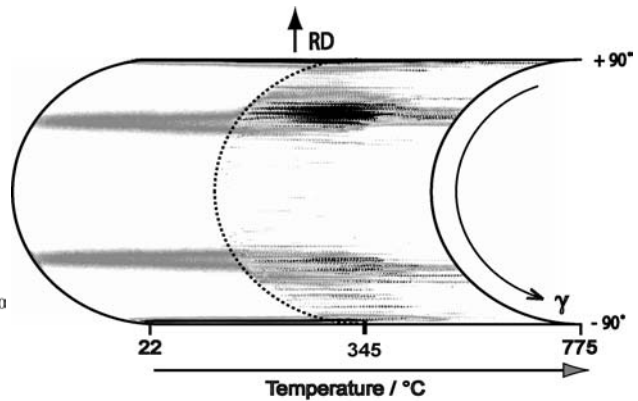


Fig.4. Measurement in (111) reflection of the texture change during annealing, temperature range 22°C to 778°C. The orientation angle ω is fixed at 55° (see Fig.3).

Figure 4 shows the orientation changes during a temperature change in the range from 22°C to 775°C. The annealing time was 26 min. The orientation angle ω was fixed at 55°. It can be clearly seen, that the first recrystallization components appear near 345°C. Close to the cube texture a lot of new grains appear, but there is also a spread over the whole orientation angle γ . After 345°C the deformation component is strongly reduced. In the regions of non-cube components some orientations vanish during annealing. This can be traced back to a consuming of the “wrong”

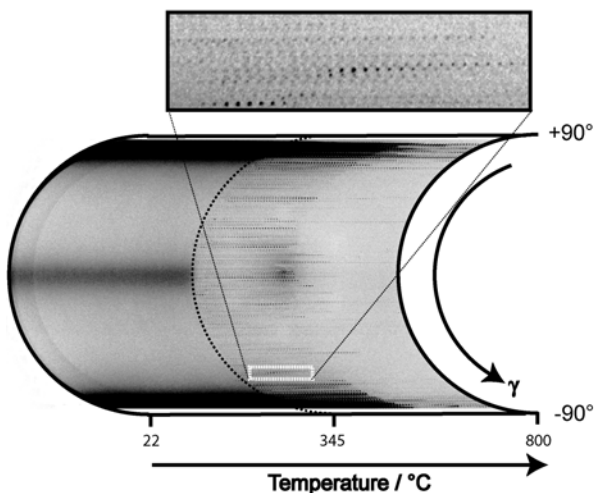


Fig.5. Measurement in (111) reflection of the texture change during annealing. The orientation angle ω is fixed at 0° (see Fig.3).

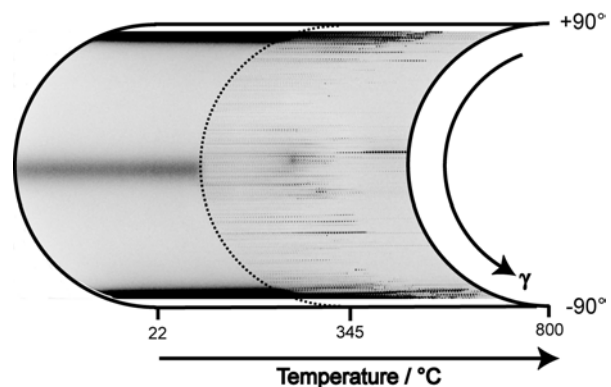
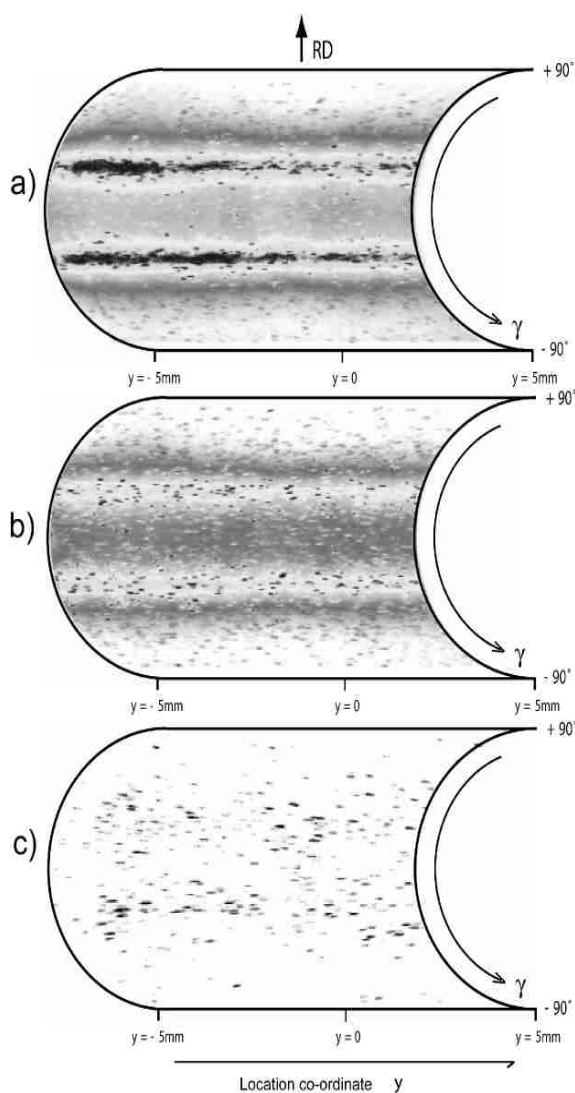


Fig.6. Measurement in (111) reflection of the texture change during annealing. The orientation angle ω is fixed at 18° (see Fig.3).

oriented grains or to a small orientation change. Because of the high angular resolving power both processes cannot be distinguished [5]. In order to get this information the sample was rotated during the measurement around the angle ω with an interval $\Delta\omega = \pm 1^\circ$ with an angular-speed of nearly $1^\circ/\text{sec}$. In the zoomed region of fig. 5 it can be clearly seen, that apparently both processes appear. Some components change their orientation slightly, this can be recognized as “jumps” in the orientation angle γ , some orientations vanish totally, maybe they are consumed. In figure 5 the orientation angle ω was fixed at 0° , the toggle angle $\Delta\omega$ was $\pm 1^\circ$. The recrystallization begins to start at 345°C . The picture shows, that the deformation component ends nearly abruptly at this temperature. Also in this image an wide orientation spread over γ is to be seen. In this large region some grains change their angular positions in γ or others are consumed and their intensities vanish. The measurement at a fixed orientation angle $\omega=18^\circ$ (fig. 6) shows that the recrystallization starts always at the same temperature (345°C). The deformation component does not change so fast as at $\omega=0^\circ$, because it is close to the cube twin component.



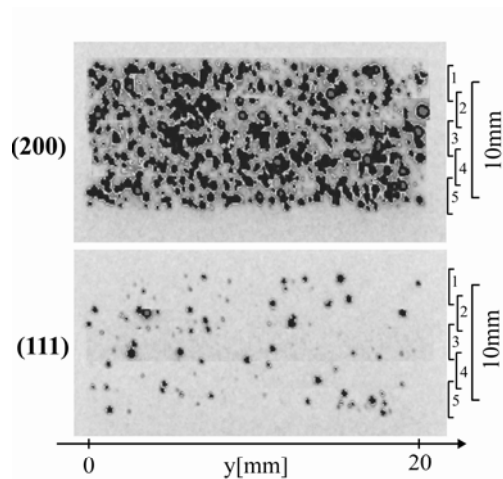
Nickel location / orientation measurements.

In fig.7 location scans of the a) 30 min, b) 45 min and c) 90 min annealed samples of the (111) reflection can be seen. The scans in x-direction were taken in the sample orientation $\omega=0^\circ$, i.e. with the incident beam perpendicular to the rolling plane. The slit was 0.1mm and very small grains are imaged. Because of the high angular resolution, the reflections are much narrower in γ direction than in y-direction. Hence, a crystallite is imaged by a streak rather than a point. Bigger grains are seen as longer streaks with the streak-length corresponding to the grain diameter in scanning direction (transverse direction). The scanning length in y-direction was 10mm.

Besides the streaks corresponding to grains with non disturbed crystal lattice, one also sees regions of continuous intensity distributions (figs. 7a and 7b) extended in the sample direction y as well as in the crystal orientation γ . These areas may be attributed to deformed material having a deformation texture which is distributed more or less homogeneously over the sample in y coordinates. One can clearly see that the amount of deformed material after 30 min annealing (fig.7a) is larger than after 45 min (fig.7b). After 90 min annealing a very weak continuous intensity distribution related to the deformed material (fig. 7c) can be seen.

Fig. 7 Location resolved scans (method “b”) of the recrystallized Ni sheet, (111) reflection. The scanning direction was the transverse direction, the scanning length was 10 mm. Annealing time: a) 30min, b) 45 min and c) 90 min.

Nickel location / orientation measurements. Fig. 8 shows the projected cross-sections of grains with selected orientations of a fully recrystallized nickel sheet obtained with method “c”. The grains correspond to one particular diffraction vector in each of the two partial diagrams i.e. they are near to the main texture component and far away from it, respectively. With this technique the development of individual grains during recrystallization and continuous grain coarsening can be very well studied.



Discussion. The static recrystallization textures of nickel (fig.2b) (93,3% cold rolled and subsequently 300 min annealed) is a sharp cube texture which shows, beside the cube orientation, also orientations of the recrystallization twins. The texture after an annealing time of 45 min (fig.2a) shows the main recrystallization components overlapping rests of the deformation texture. In both pole figures a relatively large spread of single peaks growing around the components. This mainly becomes visible due to the high angular resolution.

The presence of deformed nickel can be better seen in the location scans (fig. 7). With increasing annealing time the continuous intensity, which can be expected from a cold rolling texture component becomes, weaker and more clearly defined streaks attributed to individual grains appear (fig. 7c).

The in-situ recrystallization experiments (figs. 4, 5, 6) show at the appearance of the first recrystallization components virtually no empty regions. The deformation components vanish more or less at a specific temperature independent of the chosen orientation ω .

The present in-situ results must be considered as preliminary. They need to be corroborated by further investigations. Nevertheless, it can be concluded that the “Sweeping-Detector”-technique used here, is especially suited for the study of recrystallization processes, including particularly dynamic recrystallization [7,8].

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