

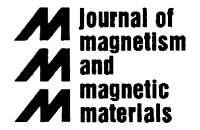


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Texture measurement of grain-oriented electrical steels after secondary recrystallization

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Abstract

The measurement of the final Goss texture sharpness in grain-oriented electrical steels is a challenging task due to the immense grain size ranging from millimeters to centimeters. Although, it is widely claimed in the literature that the orientation deviations from the ideal Goss orientation lie in the range of about 7° for conventional grain-oriented steel and in the range of about 3° for high permeability grades, no precise investigation with an appropriate statistical relevance is known to the authors.

In this work, X-ray diffraction and large-area EBSD-based orientation microscopy (EBSD: electron backscatter diffraction) were used for texture analysis and orientation determination in order to estimate the Goss orientation spread of different grades of grain-oriented steel. Two production routes for grain-oriented steel sheets are compared, the conventional route and a low heating route with lower inhibitor strength. The results of the texture measurement demonstrate that both routes deliver comparable values of orientation deviations. Furthermore, it can be shown that small differences of the magnetic properties can be correlated with the texture sharpness of the material.

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1. Introduction

Grain-oriented Fe–3% Si steels are used as cores in electrical transformers due to their soft magnetic properties. They are characterized by a sharp {011} <100> texture (Goss texture) and a very large grain size (in the range of millimeters to centimeters) developing at the end of a complex production process during discontinuous grain growth.

The texture sharpness is usually regarded as a measure of magnetic properties of grain-oriented steels (and vice versa) and misorientations 3° and 7° of the <100> axis from the rolling direction are generally accepted for high permeability (HGO) and conventional grades (CGO), respectively [1–3]. The analysis of the texture sharpness is not at all trivial due to the enormous grain size and it has been a neglected area in the recent years although powerful tools

with enhanced measurement speed and precision have been developed.

Increasing quality demands and the pressure to reduce manufacturing costs are the two main driving forces for research and development activities in industry. New production techniques have been recently developed, e.g., low-heating routes and thin-slab-casting methods [4]. Magnetic properties of these materials are of major interest to the steel producers and these properties can be measured with standardized methods. However, little is known about the texture sharpness of the final sheet produced by either of these process routes.

The present paper aims at presenting a couple of methods for measuring the texture of large-grained materials based on X-ray diffraction (XRD) and electron backscatter diffraction (EBSD) in the scanning electron microscope (SEM). In the authors' view, the XRD method has the potential of becoming a standard method for quality control of Goss-textured electrical steel sheets.

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2. Experimental technique

The material used in this investigation was the final sheet of Fe–3% Si grain-oriented steel of two different grades, HGO and CGO. Furthermore, two production routes were analyzed, the conventional route (route 1) and a low heating route (route 2) with lower inhibitor strength. All the materials were kindly provided by ThyssenKrupp Electrical Steel. In order to check the accuracy of the XRD setup a Goss-oriented Fe–3% Si single crystal, produced at the Max-Planck-Institut für Eisenforschung by zone melting, was also investigated.

XRD measurements were carried out in a goniometer equipped with a sample stage with 6 degrees of freedom: an Eulerian cradle with φ -, χ - and ω -rotations in combination with a translation sample stage with x -, y - and z -movement. At every pole figure measurement position, the sample was moved in x - and y -direction in order to cover a maximum area of the measured sample. For detection, an area detector with a high measurement speed (200 counts/pixels) was used. The setup further consisted of a cobalt X-ray tube and a collimator for the generation of a parallel beam with 1 mm spot size.

For covering large sample areas, the final sheet was cut by spark erosion into 100 slices of $10 \times 30 \text{ mm}^2$, stacked and mounted in a holder for sample preparation and texture analysis on the cross-section (Fig. 1). With this method, probing of grains from more than 400 cm^2 was realized, containing about 1400 grains in case of CGO (route 1) and about 130 grains in case of HGO (route 1), thus providing excellent statistical data.

XRD analysis was performed using a standard grid of 5° for the φ -rotation, measuring the (100) and (110) pole figures which takes a total time of less than 4 h. For determination of the texture sharpness, the peaks of the pole figures were fitted along the φ -circle by a Gaussian distribution: the value of the full-width at half-maximum

(FWHM) represents the directional spread about the normal direction and is a measure for the orientation spread of the Goss orientation.

In contrast to the calculated orientation distribution function (ODF) obtained by X-ray diffraction, the EBSD technique directly *measures* the ODF by means of individual crystal orientation measurements. Therefore, it avoids possible calculation errors. The use of a large-scan EBSD setup enables accurate analysis and rapid acquisition of the diffraction patterns. For the present investigation, a JEOL 840A SEM was utilized. Large-area scans on the sheet surface were realized in a combo scanning mode with a step size of $100 \mu\text{m}$, taking about 30 min for a single sample of $2 \times 3 \text{ mm}^2$ size, providing a total area of about 20 cm^2 for the display of the pole figures for each material. It should be mentioned that this area contains only about 70 grains in case of CGO (route 1) and about 10 grains in case of HGO (route 1).

3. Results and discussion

3.1. X-ray diffraction

The pole figures plotted in Fig. 2 show the (110) poles of CGO (a), of HGO (b), processed by the conventional route 1, and of a Goss-oriented single crystal (c). As expected, the peak width, i.e. the orientation spread, is distinctly larger in the case of CGO material compared to that of HGO. The calculation of FWHM values of the poles in the pole figures delivers orientation spreads of 9° and 6° for CGO and HGO, respectively. These results were obtained with a standard measurement grid of $\Delta\varphi = 5^\circ$. Furthermore, orientation deviations of 11° and 7° were obtained for CGO and HGO, respectively, processed via route 2. All results of the XRD analysis, as summarized in Table 1, show a correlation with the magnetic properties, even in the case of only slight differences with regard to core losses and permeability between the materials of the same grades processed by different routes.

The accuracy of the XRD setup was examined by an additional investigation of a single crystal with exact Goss orientation. For the same measurement grid ($\Delta\varphi = 5^\circ$), an orientation spread of 5° , which matches the magnitude of the grid, was found. A change to higher orientation resolution, i.e. to a smaller grid of $\Delta\varphi = 1^\circ$, led to considerable smaller orientation deviations, in the case of the single crystal to a FWHM value of 1.1° (Fig. 2c). A high-resolution measurement with the narrow grid was accomplished for the HGO material produced via route 1, and a value of 3.8° was found for the orientation spread which is markedly less than the measurement with a larger grid of $\Delta\varphi = 5^\circ$, as shown above. Consequently, for the exact analysis of small orientation deviations a high-orientation resolution, i.e. a fine grid, is necessary, which extends the measurement time by a factor of 3 to 11 h, approximately. However, the total time can be drastically

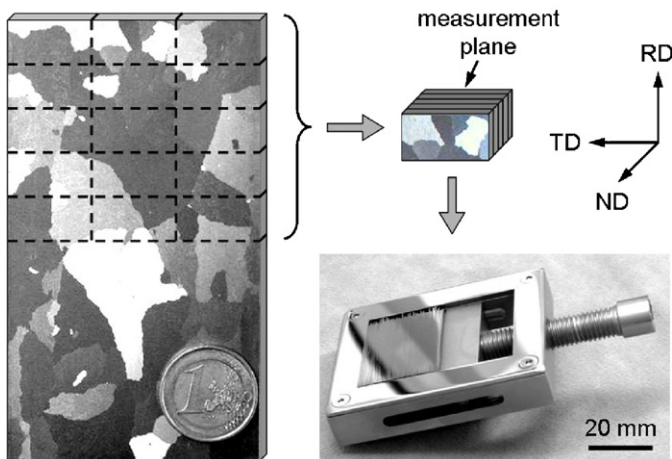


Fig. 1. Sample setup for XRD measurement: a stack of sheets is mounted in a holder for sample preparation and texture measurement on the cross-section.

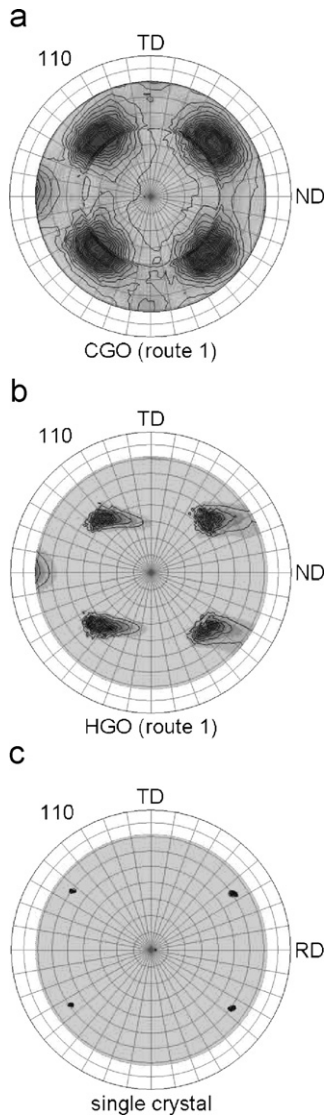


Fig. 2. XRD pole figures of (a) CGO and (b) HGO (both route 1) measured with a grid of $\Delta\phi = 5^\circ$ and (c) pole figure of a Goss-oriented single crystal measured with $\Delta\phi = 1^\circ$.

Table 1
Comparison of the texture sharpness measured with XRD and EBSD

Material	Goss orientation spread (deg)			Magnetic properties
	XRD (grid 5°)	XRD (grid 1°)	EBSD	
HGO (route 1)	6	3.8	3.2	
HGO (route 2)	7	–	3.1	
CGO (route 1)	9	–	5.9	
CGO (route 2)	11	–	5.3	

reduced (by a factor of 4 or more) if only one peak of the pole figure is measured which is sufficient for the analysis proposed above.

3.2. EBSD measurement

Examples of EBSD measurements obtained from single scans of HGO and CGO samples processed via route 1 are shown in Fig. 3. It is obvious that the grain size of HGO is drastically larger than that of CGO. Furthermore, the orientation deviation from the ideal Goss orientation is less in the case of HGO as can be seen from the coloring of the mappings.

In order to account for the inaccuracies due to sample positioning and minor statistical aberrations, the texture sharpness was not directly calculated as the deviation from the theoretical Goss orientation. Instead, a reference orientation well out of the Goss orientation spread was

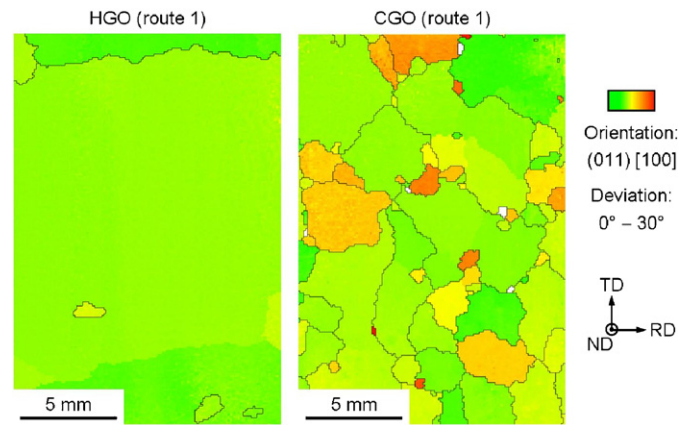


Fig. 3. EBSD images of the normal plane of HGO and CGO (both route 1); the color code indicates the deviation from the ideal Goss orientation.

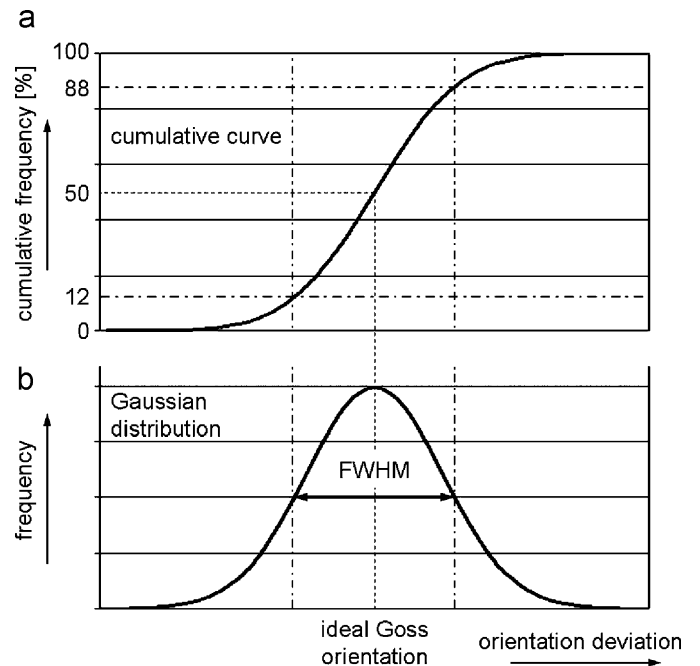


Fig. 4. Schematic diagram of the cumulative frequency of the deviation from the ideal Goss orientation and resulting Gauss distribution used for the evaluation of the EBSD data.

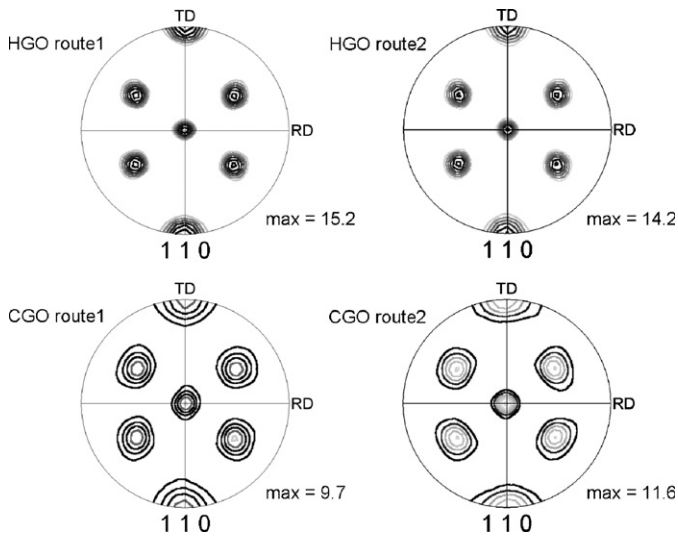


Fig. 5. EBSD pole figures of HGO and CGO for routes 1 and 2.

selected and the deviation from this theoretical orientation was measured and classified into intervals of 1° . From these data the cumulative frequency was calculated, shown in Fig. 4a. Subsequently, an orientation distribution was computed from the cumulative curves under the following assumptions:

1. The true major orientation is the Goss orientation.
2. The deviation from the ideal Goss orientation follows a Gaussian distribution.

The appropriate distribution is shown in Fig. 4b. The FWHM values determined from these curves are collected in Table 1. The texture sharpness tends to have smaller values—about 3° for HGO and 6° for CGO—compared to the XRD results measured with $\Delta\varphi = 5^\circ$. The same tendency is found concerning the difference between the two grades of electrical steel. Moreover, the result the high-resolution XRD analysis ($\Delta\varphi = 1^\circ$) performed with HGO

(route 1) is in good agreement with the result of the EBSD measurement.

For a better visualization, Fig. 5 displays the (110) pole figures for both grades of the material processed via routes 1 and 2. The texture data were obtained from the EBSD measurements after merging all the maps belonging to one sample. A comparison of the pole figures shows larger orientation spreads and lower intensities for CGO compare to those for HGO, but shows similar peak width for the same material processed via routes 1 and 2.

4. Conclusions

Both measurement techniques, X-ray diffraction and EBSD, deliver comparable values for the Goss orientation spreads and they lie in the range of about 3° for HGO and about 6° for CGO. Furthermore, it is shown that both production routes—the conventional and the low heating process—deliver materials of comparable texture sharpness. The setup proposed for X-ray measurement offers an efficient tool for the determination of the texture sharpness of large-grained materials due to excellent statistics and less experimental effort compared to EBSD measurements. The measurement time for the XRD analysis can be drastically reduced if only one peak of the pole figure is measured, which is sufficient for determination of texture sharpness.

Further experiments are currently being performed to complete the XRD measurements with high orientation resolution ($\Delta\varphi = 1^\circ$) in order to further correlate the texture sharpness with magnetic properties of materials processed by different production routes.

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