Fast and simple method for Goss texture evaluation by neutron diffraction

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Abstract. Requirement of low power losses is one of the crucial demands laid on properties of electric steel sheets used in construction of various magnetic circuits. For cold-rolled grain-oriented (CRGO) Fe-3%Si sheets used in majority of power distribution transformers, the Goss texture {110}<001> is known to provide the best utility properties (low power loses, high magnetic permeability). Due to the coarse grain size of CRGO steel, neutron diffraction (ND) is dominantly used to characterize the sheets' texture in order to achieve statistically significant data. In this paper, we present a fast and simple method for characterization of Goss texture perfection level in CRGO steel sheets based on monochromatic ND. The method is tested on 8 samples differing in fabrication technology and magnetic properties. Satisfactory performance of the method and its suitability for a detail texture analyses is tested by juxtaposition of the obtained textural and the magnetic characteristics measured by Barkhausen method.

1. Introduction

Highly perfect (100) [001] crystallographic preferential orientation of grains (Goss texture) in cold-rolled grain-oriented (CRGO) Fe-3%Si steel sheets is of crucial importance for achievement of the extraordinary magnetic properties characterized nowadays by typical magnetic losses in range of several tens of W/kg and saturation magnetisation in the easy direction 1.91-1.92 T [1-3]. Still, even for the best materials produced today, there is some level of the Goss texture imperfection that can be reduced in order to achieve even better figures of the resulting magnetic properties. To do this, fine and detail characterization of the Goss texture sharpness must be performed with the angular resolution exceeding the usual limits used in standard texture analyses.

In this paper, a simple neutron diffraction method suitable for fast and highly sensitive evaluation of the ideal Goss texture perfection in large sample series is proposed and tested on 8 samples of CRGO Fe-3%Si steel sheets differing in their fabrication procedures and, consequently, resulting magnetic properties. The later were characterized by Barkhausen noise (BN) measurements, compared with the obtained texture characteristics obtained for the analysed samples and the level of correlation between both types of parameters analysed in order to verify applicability of the proposed method.

2. Experimental

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2.1. Neutron diffraction texture analysis

Samples were stacked from 6 sheets of square shape, the side being 30 mm long. Total thickness of the resulting specimens was between 1.2 - 2 mm.

The diffraction patterns were collected on KSN-2 neutron diffractometer situated at one of the horizontal channels of the nuclear research reactor LVR-15 at the Nuclear Research Institute, plc. Rez, Czech Republic. The specimen was mounted in the Huber Eulerian cradle 511.5 goniometer and irradiated by the parallel beam of monochromatic neutrons with the wavelength 0.1052 nm obtained by reflection on Cu(200) single crystal. In such setting, the KSN-2 diffraction device offers good intensity and the best resolution value (δd/d) ca. 0.007 within the interval of inter-planar distances d ~ 1.0 ÷ 0.1 nm.

For every specimen, distribution of integral intensity of the (002) reflection (I^0) was recorded at nodes of the angular network (ω, χ) scanned in the range ±10° with the steps Δ ω= Δ χ=1° around the rolling direction (RD). The raw data were then corrected for background, absorption and irradiated volume changes and normalized on the (002) integral intensity of a specimen with randomly oriented crystallites

$$I^{sc}(\chi,\omega) = \frac{1}{I^{r}(\omega=0)}(I^{0}(\chi,\omega) - B^{0}(\chi,\omega))K(\omega) \tag{1}$$

Here $I^{sc}(\chi,\omega)$ is the normalized intensity distribution, $B^o(\chi,\omega)$ is the background intensity, $I^r(\omega = 0)$ is integral intensity of the (002) reflection for the randomly oriented powder sample measured in a symmetrical transmission position (ω = 0) and corrected for the background. The correction factor $K(\omega)$ must be applied used to unify the geometric and material conditions of the measurements performed on the textured specimens and on the reference powder sample

$$K(\omega) = \left(\frac{\rho_p}{\rho_s}\right)\left(\frac{t_p}{t_s}\right)\frac{\cos(\theta+\omega)}{\cos\theta}\exp\left[\frac{\mu_s t_s}{\cos(\theta+\omega)} - \frac{\mu_p t_p}{\cos(\theta)}\right] \tag{2}$$

Rel. (2) is derived for specimens of a cuboid shape, irradiated by the incident beam through only one of the faces. The latter assumption can be easily fulfilled in our case of the small (ω, χ) scanning range. The quantities ρ and t denote the mass density and thickness of the textured (subscript s) and the powder sample (p) respectively; Θ is the Bragg angle of the (002) reflection maximum.

The relative percentile volume of crystallites with the [001] crystallographic direction within the angular surroundings Ω of RD is then

$$V^{\%}_{[001]}(\Omega) = j(001)\frac{100}{4\pi}\oint_{(\Omega)} I^{sc}(\alpha,\beta)\sin\alpha\, d\alpha\, d\beta \tag{3}$$

Here α is the tilt angle between *RD* and the scattering vector, β is the azimuthal angle corresponding to the rotation of *RD* around the scattering vector, and j(001) is the multiplicity of the {001} planes. Within sufficiently narrow angular surroundings of RD, Rel. (3) can be approximated by

$$V^{\%}_{[001]}(\Omega') \cong j(001)\frac{100}{4\pi}\oint_{(\Omega')} I^{sc}(\chi,\omega)\, d\chi\, d\omega \tag{4}$$

In case of $\alpha \leq 10^o$ and $0 \leq \beta \leq 360^o$, the difference between Ω and Ω' values is less than 6×10^{-4} rad. Rel. (4) was applied to calculate the volume fractions summarized in Table 1.

2.2. Barkhausen noise analysis

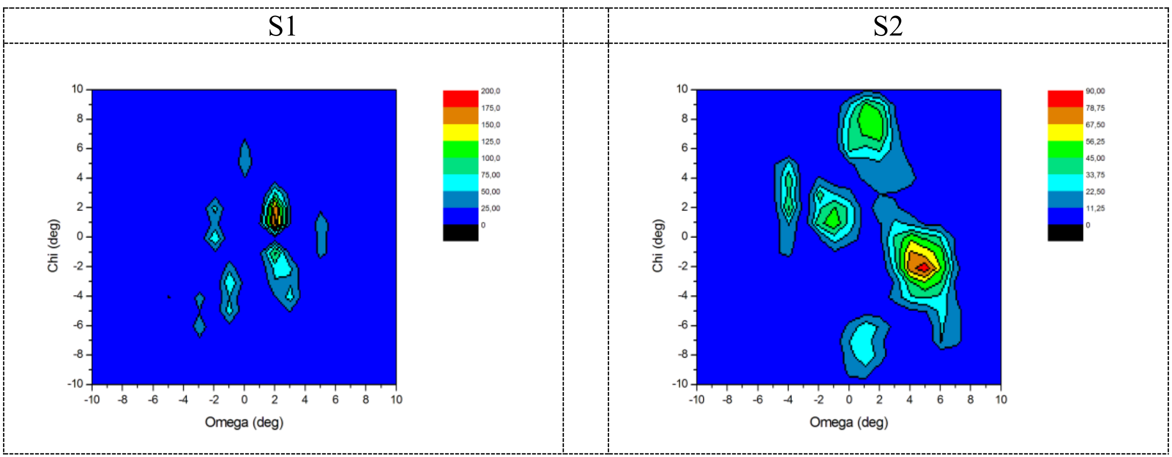
Measurements of the Barkhausen effect [4-7] and hysteresis curve characteristics were performed on single sheets with aid of the magneto-elastic analyser Rollscan 300 (Stresstech) equipped with the software MicroScan 600-1 and the standard probe S1-138-15-0. Coercive force Hc, remanence Br, relative permeability μ_r, in-plane angular distribution of the root mean square (rms) of the BN intensity and the angular average of the latter (further referred as RMS) were recorded at four randomly selected places on the tested sheet surface. The final data were obtained as the statistical average of the individual measurements. A sinusoidal modulation of the magnetization field intensity was applied with the frequency 45 Hz and the magnetic probe driving (peak-to-peak) voltage 12.5 V.

3. Results and discussion

The intensity distributions $I^{sc}(\chi, \omega)$ obtained from the experimental data in accordance with Rel. (1) are shown in Figure 1. It is apparent that significant differences exist in character of the distributions concerning both intensity/broadness of the present maxima as well as declination of these from the RD. Integral quantitative representation of the distributions (neglecting such important features as sharpness of the maxima) is provided by the volume fractions presented in the first row of Table 1.

Table 1. Percentile volume fractions $V^{\%}_{[001]}$ of the crystallites having [001] direction oriented within ±10° (χ, ω) angular surroundings of RD.

Sample	**S1**	**S2**	**S3**	**S4**	**S5**	**S6**	**S7**	**S8**
$V^{\%}_{[001]}$	49.8	45	38.7	16.3	39.2	27.8	29.2	24.6
Hc (kA/m)	119,5	71,5	71,5	47,4	100,9	70,1	52,8	58,1
Br (mT)	879	639,3	405,4	243,1	589,3	342,8	193,8	178,5
μ_r	8432	10156	6285	5349	6074	5288	4030	3277
RMS	161,5	222,1	136,9	127,8	138,4	111	95,4	81,3



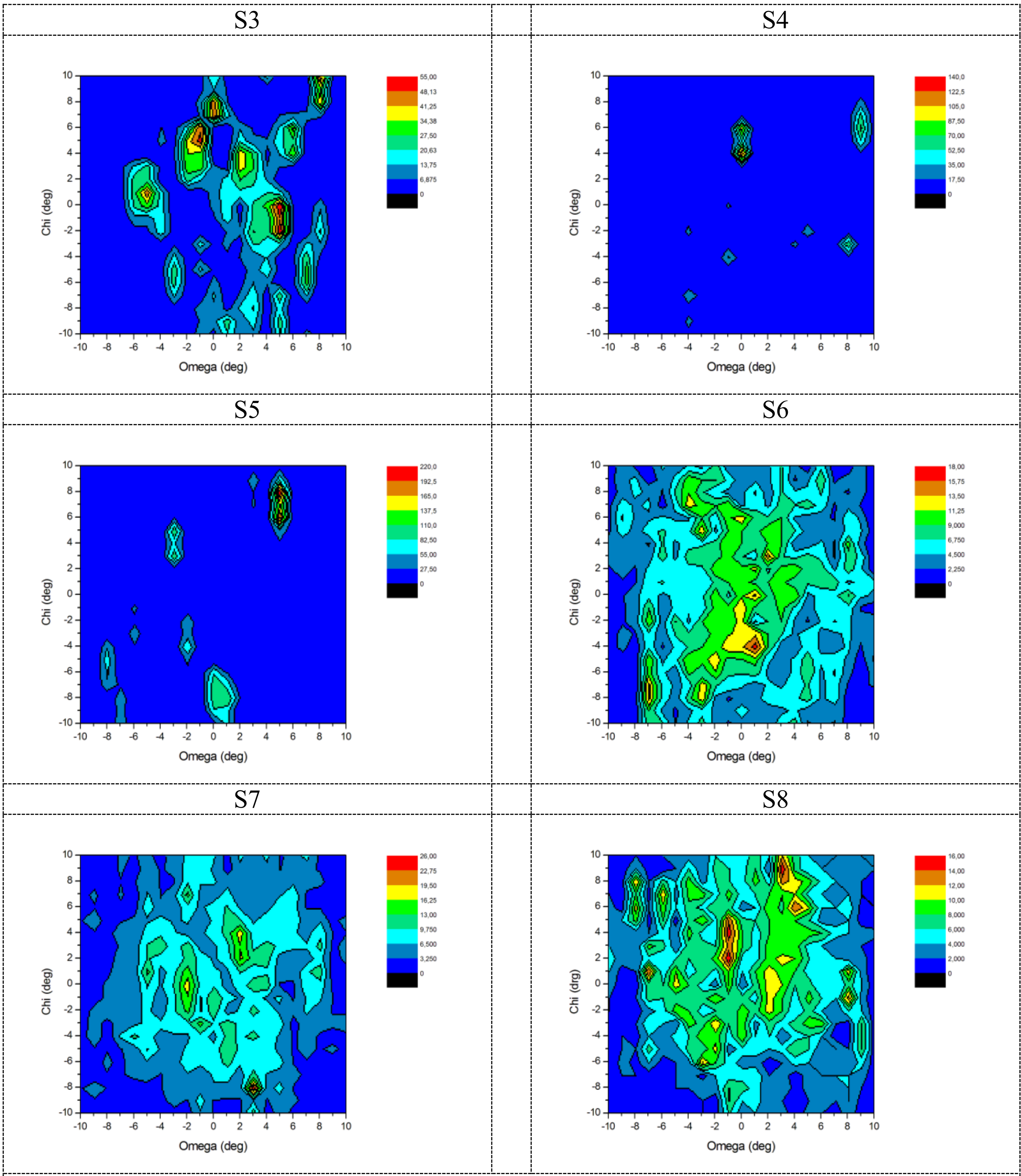


Figure 1. Distribution of [001] direction around rolling direction (RD).

Experimental values of the tested magnetic parameters (Hc, Br, μr, RMS) are summarized in the second part of Table 1. Figure 2 then shows the polar plot of the RMS intensity of BN obtained for the tested samples.

In order to discuss the relationship between the textural and magnetic characteristics, we started with determination of the correlation coefficients linking together the grain volume fractions $V^{\%}_{[001]}$ and the magnetic parameters. The value 0.81, 0.89, 0.77 and 0.68 was obtained for Hc, Br, μ_r and RMS, respectively. Thus, the remanence Br shows the most remarkable overall correlation with the texture

order of the samples. Moreover, some more delicate tendencies can be further noticed. The sample S1 shows the maximal $V^{\%}_{[001]}$ value as well as the highest Hc and Br figures. But, it is not superior in its magnetic permittivity and RMS. These are maximal for sample S2 characterized by a slightly lower $V^{\%}_{[001]}$, but higher sharpness of the texture (c.f. Figure 1). The latter findings seems to be in sound with the expectation that the Bloch walls jumps responsible for the BN signal are easier in case of when the crystallites orientation distribution is more uniform and narrow.
The minimal value of $V^{\%}_{[001]}$ volume fraction provides the sample S4. The latter possess the minimal coercive force value, but the values of Br, μr and RMS are still higher than those observed for samples S6-S8, showing all larger $V^{\%}_{[001]}$ values. Careful look on the detail intensity distributions in Figure 1 reveals that the grain distribution of S4 is apparently sharper than the distributions observed for S6-S8. Thus, the detail sharpness of the grain distribution seems to affect differently the different magnetic characteristics, promoting likely higher μ_r and RMS values and, following the curves in Figure 2 affecting in the same way the angular distributions of the RMS BN intensity, too.

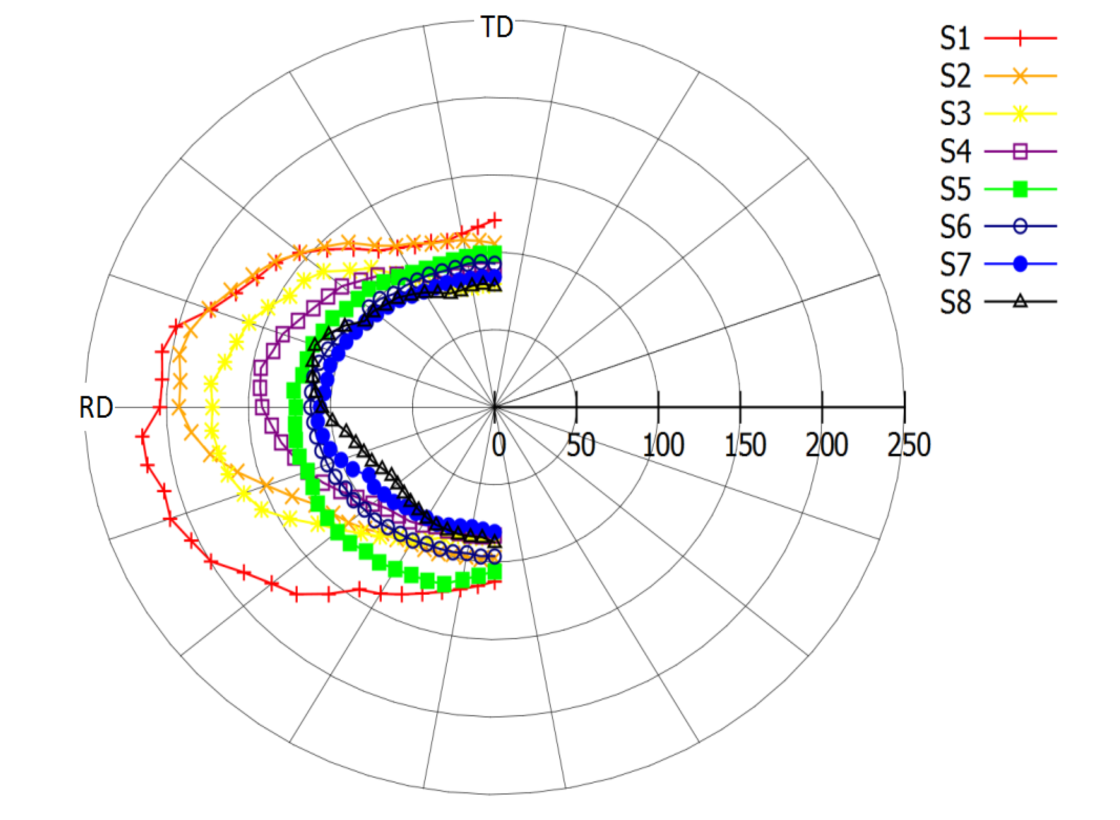


Figure 2. Polar plot of the RMS intensity of BN observed on the tested samples S1 - S8; only the angularly asymmetric part of the signal is shown.

4. Conclusions

Simple method suitable for detailed texture analyses of CRGO Fe3%Si steel sheets is proposed and tested on 8 samples significantly differing in the Goss texture perfection. Comparison of the texture results with the magnetic properties characterized by Barhausen method confirmed that the method provides results that can be well correlated with the main magnetic properties of CRGO sheets. Moreover, combination of the integral view of the investigated grain distribution with analysis of its local variations and sharpness seems to provide a valuable tool for investigation of special relations between different features of the texture distribution and the selected magnetic properties.

5. References

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