Correlation between the deformation microstructure after rolling and the recrystallization nucleation of a non-oriented electrical steel

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Abstract. The characteristics of the cold-rolled (CR) microstructure of a non-oriented electrical steel after 50-90% cold rolling were studied using electron backscatter diffraction. The local heterogeneity of the deformed microstructure was found to consist of high misorientation regions (HMRs) and low misorientation regions (LMRs) according to local misorientation and size. The overall orientation distribution of HMRs was obtained accordingly and was compared to that of the fully recrystallized sample. Moreover, the CR and partially recrystallized states of the same area were analyzed and compared. The position and orientation of each HMR can be identified individually, and be correlated with the recrystallization nuclei. The HMRs were distributed near the original high angle boundaries at 50% rolling reduction, but extended into the grain interiors with increasing rolling reductions to 70% and 90%. As a result, the recrystallized nuclei for the 50% cold rolled sample were mainly distributed at the original high angle boundaries and the triple junctions, whereas extended nucleation at the deformation bands was observed for the high rolling reduction samples. Good correlation of the orientation between the HMRs and the recrystallized nuclei were observed.

1. Introduction
Texture evolution of non-oriented electrical steels from a deformed state to a fully recrystallized (RX) state during heat treatment has been an important issue in the academia and industry [1-3]. The cold rolling (CR) texture contains mainly a $\alpha$-fiber, $\{hkl\}<110>$, which shows orientations from $\{001\}<110>$ through $\{112\}<110>$ to $\{111\}<110>$. Furthermore, a weak $\gamma$-fiber, $\{111\}<uvw>$ has also been identified in the CR texture. The strong $\alpha$-fiber develops during rolling has been noted to disappear after RX. On the contrary, the RX texture, comprising varying intensities of $\{001\}<uvw>$, $\{111\}<uvw>$ and $\{011\}<100>$ components, develops during annealing. It has been recognized experimentally that the RX nuclei evolve from fast growing subgrains surrounded by high angle boundaries (HABs) at high rolling reductions [4-7]. Additionally, Humphreys [8] proposed a unified theory determining whether RX is discontinuous during heat treatment according to the size and misorientation of the subgrains. Both experimental observations and theoretical calculations imply that the texture of the recrystallized steel evolve from the subgrains having high misorientation angles relative to the surrounding matrix in the deformed state.

The deformed microstructure is usually not homogeneous. For instance, extended planar dislocation boundaries forming cell blocks were observed in CR IF steel [3]. The presence of bands of localized...
micro-shearing was noticed to be stronger than in fcc aluminum and nickel [3]. RX nucleation sites are expected to closely depend on the heterogeneities in the deformed microstructure. It is therefore important to differentiate the structural heterogeneities and identify the subgrains, which have the potential to develop into RX nuclei, in the CR structure. Orientation image mapping (OIM) based on electron backscatter diffraction (EBSD) can give the spatial distribution of crystal orientations over large areas. Consequently, EBSD analyses are suitable for characterizing the heterogeneities in the deformed structures. HABs and the differences of stored energy across boundaries are related to the local misorientation in the deformed structure, which can be determined using EBSD with proper scan step sizes. Brewer et al. [9] showed that local misorientation distribution in CR structures can be illustrated using kernel average misorientation (KAM) method. Moreover, Mishin et al. [10-12] proved the use of EBSD to analyze local heterogeneity in deformed metals. The deformed microstructure can be considered as a two-phase mixture containing both high misorientation regions (HMRs) and low misorientation regions (LMRs). Subsequently, the LMR/HMR map can be constructed even when the specific characteristics of the low angle boundaries in the microstructure cannot be completely identified [10-12].

In the present work, the same areas in both the CR and RX states of an electrical steel were examined using EBSD. HMRs and LMRs can be distinguished in the CR state. It was revealed that the potential RX nucleation sites in the CR structure are the places located inside the HMRs and having high KAM values. Places satisfying the above criteria were the original grain boundaries in the 50% CR sample and extended to deformation bands in the 70% and 90% CR samples.

2. Experimental
A non-oriented electrical steel (ES) with the composition (wt%) of 0.0015 C, 0.86 Si, 0.31 Mn, 0.22 Al, 0.0021 N, 0.005 P and 0.005 S was used. The steel was hot rolled from 100 mm to 12 mm in thickness with a finishing temperature of 860°C and followed by annealing at 740°C for one hour. The hot rolled plate was subsequently cold rolled to 50%, 70% and 90% rolling reductions. RX temperatures of the three samples were determined from the microhardness value change as a function of the annealing temperature.

Texture analyses were carried out on the normal direction (ND) planes of the samples by X-ray diffraction (XRD) with Co-Kα radiation. The orientation distribution functions (ODFs) were calculated from three pole figures, (200), (220), and (211), using the series expansion method. Microtexture measurements were conducted on the transverse direction (TD) planes for the same areas of the CR states and partially RX states using EBSD. A step size of 0.25 μm was selected to scan four areas in each sample. The local misorientation distribution in the CR state was characterized using KAM approach with a selection of KAM > 10° as high-KAM. The heterogeneity of the CR structure was examined using LMRs and HMRs. It is essential to identify LMRs and the rest of the microstructures are HMRs. θ₀ is the maximum misorientation between adjacent pixels in a LMR. A_L defines the minimum size of LMR. In this work, θ₀ of 5° and A_L of 10 μm were chosen according to our previous analyses [13].

3. Results and Discussions
The ODF section at φ₂ = 45° for the CR and fully RX states are given in figure 1. It can be noted that the intensity of the α-fiber increases gradually, whereas that of the {111}<112> component decreases slightly, with increasing rolling reduction. The strongest orientation is close to {112}<110> along the α-fiber. These observations are consistent with the reports in the literature [1-4]. The fully RX states of the CR samples exhibit a considerably different texture. It can be detected that the intensity of the α-fiber diminishes drastically while the γ-fiber becomes the dominant texture. The intensity of the γ-fiber is noticed to become stronger with rolling reduction. The {111}<112> component along γ-fiber is the most intense orientation, while Goss orientation ({011}<100>) is also observed. However, the intensity of Goss is found to decrease with rolling reduction. The ODFs of the high-KAM sites inside the HMRs (denoted as K-H sites thereafter), which are extracted from the CR samples, are also
provided in figure 1. It is clear that the texture of the deformed substructures which fulfill both high-KAM and HMRs requirements correlates well with the texture of the RX grains. The intensity of the \{111\}<112> component increases with increasing the rolling reduction, whereas that of the Goss component decreases as the rolling reduction increases from 70% to 90%. The HMRs are regions containing high stored energy in which RX can proceed preferentially from the thermodynamic point of view. Moreover, the K-H sites exhibit high boundary mobility which allows a fast growth kinetically. The results therefore indicate that the potential nucleation sites in RX can be screened from the CR microstructure effectively.

The annealing temperatures for partial RX (~20% RX) were determined from the microhardness tests. 650°C, 630°C and 610°C were chosen for the 50%, 70% and 90% CR samples, respectively. It is reasonable that the RX temperature decreases with increasing deformation strain since a higher stored energy is retained in the more severely deformed structure [14]. The ND inverse pole figure (IPF) maps of the 50% CR sample and the partial RX sample are shown in figure 2a and 2b. It can be noted that the deformed grains become slightly elongated and align with the rolling direction (RD). The average long axes and short axes of the grains were measured to be over 100 μm and about 20 μm, respectively. The CR grains are observed to show some extent of heterogeneity. The grains with orientations along the \(\alpha\)-fiber are more elongated while the grains with \(\gamma\)-fiber orientations have smaller aspect ratio. Additionally, there are some deformation bands within the grain interiors. It can also be seen that some grains have more deformation bands subdividing them, which show multiple orientations while others (mostly the grains with orientations along \(\alpha\)-fiber) are only slightly deformed. This is an obvious evidence of deformation heterogeneity. The partially RX IPF map of the same area clearly indicates the locations of RX nuclei (figure 2b) and can be directly compared to the CR structure. These RX grains are seen to locate at the original grain boundaries. More apparent confirmation will be given later.

**Figure 1** ODF \(\varphi_2 = 45^\circ\) section for the (a) 50%, (b) 70% and (c) 90% CR samples. **CR**: XRD ODF for the as-rolled samples; **fully RX**: XRD ODF for the samples annealed at 710 °C for 60 s; **K-H**: EBSD ODF for the K-H sites screened from the CR samples.
As the ES sample is cold rolled to a 70% reduction, the grains become more elongated (figure 3a). The long axes of the grains are measured to be over 200 μm, while the short axes are about 15 μm. Similar to the 50% CR sample, many deformation bands with different orientations can be noticed within the original grains. Some grains are subdivided to a higher extent while others are not. An IPF map of the partial RX for the same area is presented in figure 3b, which can be used to make a direct comparison. The IPF maps for the K-H sites are revealed in figure 2c and 3c for the 50% and 70% CR samples, respectively. These figures indicate that the K-H areas are mainly distributed near and at the original grain boundaries and triple junctions with a small portion of them in the grain interiors in the 50% CR sample. Nevertheless, the K-H sites extend extensively to the interior of the γ-fiber grains in the 70% CR sample. The RX grains are extracted from figure 2b and 3b, and given in figure 2d and 3d. It is observed that the distribution of the RX grains coincides well with that of the K-H sites in both conditions. Moreover, a good correlation of texture between the K-H sites and the RX grains can also be observed. The use of HMR/LMR to identify the heterogeneity of the deformed microstructure was reported by Mishin et al. [10] in equal channel angular extrusion (ECAE) processed copper. These samples were kept at room temperature for eight years and RX occurred. It was found that RX grains appear typically within HMRs, which are located at shear bands, extended boundaries and the intersections of shear band and extended boundary. These observations are consistent with the present work.

Figure 2 (a) ND IPF map of the CR state, (b) ND IPF map of the partial RX state, (c) K-H sites, and (d) the extraction of RX grains for the 50% CR sample. (e) color key as a reference for the IPF maps.
Figure 3 (a) ND IPF map of the CR state, (b) ND IPF map of the partial RX state, (c) K-H sites, and (d) the extraction of RX grains for the 70% CR sample.

Figure 4 The intensity of $\{111\}<112>$ orientation in the CR and RX states as well as K-H sites changes as a function of CR reduction.

As mentioned in the early section, $\{111\}<112>$ is an important texture component observed in the RX state for the ES. However, the intensity of $\{111\}<112>$ is relatively weak in the CR states. Furthermore, the intensity of $\{111\}<112>$ is noticed to decrease with rolling reduction, as shown in figure 4. The decline is due to the formation of a strong $\alpha$-fiber with rolling reduction. It is remarkable that the weak $\{111\}<112>$ orientation becomes the dominant texture component in the RX state. The intensity of $\{111\}<112>$ in the RX state is found to increase substantially with rolling reduction (figure 4). It is interesting to observe the large difference of intensity for the CR and RX states, especially in the 90% CR sample. It means that the small populations of $\{111\}<112>$ orientation develop into the main component in the RX state. The intensities of $\{111\}<112>$ of the K-H sites
obtained from the CR structures are also given in figure 4. It shows the same trend as the RX one with increasing rolling reduction, except that its intensity in the RX state is stronger than that in the K-H sites. The difference is mainly originated from competitive growth of nuclei in RX. Consequently, the combination of high-KAM with HMR criteria is an efficient method to predict the locations of RX nuclei and their orientations in the deformed structure. This is due to the deformation heterogeneity and then inhomogeneous distribution of stored energy. The approach can be employed to materials design, in which RX is important.

4. Conclusions

A non-oriented electric steel was cold rolled to different rolling reductions of 50-90%. EBSD was employed to analyze the deformed and partially RX microstructure and texture. The texture of the K-H sites correlated well to that of the RX samples of three reductions. Furthermore, both the texture and the spatial distribution of the K-H sites showed a good one-on-one correspondence to the nuclei formed in the early stage of RX. Therefore, the combination of high-KAM and HMR criteria is an efficient approach to predict the locations of RX nuclei and their orientations in the deformed structure.

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