Quasi in-situ EBSD characterization of the evolution of microtexture and microstructure in non-oriented electrical steels during cold rolling and annealing

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Abstract. Non-oriented electrical steel sheets are extensively used to manufacture core laminations for electric motors. The microstructure and crystallographic texture of the final thin sheets have a significant effect on the magnetic quality of the lamination core. Controlling the development of microstructure and texture during thermomechanical processing of these steels is of great practical importance. Although the final microstructure and texture of the steel sheets are affected by all the processing procedures employed, cold rolling and final annealing have the most direct effect since these are usually the final steps to process the steel. Although much effort has been made to study the evolution of microstructure and texture in non-oriented electrical steels, the formation mechanisms of the final microstructure and texture during annealing (especially grain growth) are still not completely understood, which is partially due to the fact that it is very difficult to directly investigate these processes using conventional characterization techniques. In this research, a quasi in-situ electron backscatter diffraction (EBSD) technique was presented, which enabled the tracking of the changes of the morphology and orientation of individual grains during cold rolling and the subsequent annealing process. The experiments revealed the rotations of individual grains under plane strain compression, and tracked the formation of nuclei from the deformed matrix and the growth of new grains during the recrystallization process. The results provided valuable insights into the evolution of microstructure and microtexture during cold rolling and annealing of non-oriented electrical steels, and helped understand the mechanisms that govern the nucleation and grain growth during recrystallization.

1. Introduction
Non-oriented electrical steels are the most widely used soft magnetic material for the manufacturing of magnetic cores for electric motors and generators. Due to their practical importance, these steels have been investigated for decades to improve their magnetic and other properties [1-5]. The evolution of
microstructure and texture during thermomechanical processing of electrical steels has long been a topic of many researches [6-10], and the numerous experiments and simulations carried out in the past have greatly helped scientists and engineers understand the mechanisms governing the formation of the final microstructure and texture in the steel sheets. Based on these studies, the processing parameters have been optimized, and better microstructure and texture have been produced, which improved the magnetic properties of the final laminates. Nevertheless, there are still mechanisms that are not completely understood, which are of both scientific and practical importance. For example, although advanced crystal plasticity models [11, 12] have been developed to simulate the formation of texture during plastic deformation, it was very difficult to directly verify these simulations since the incremental deformation of the individual grains could rarely be traced experimentally. Furthermore, the mechanisms governing the nucleation and growth of new grains during recrystallization are still not completely understood, and there is still debate on whether it is oriented nucleation or oriented growth that determines the final texture [13].

The existence of these discrepancies was partially due to the lack of appropriate experimental techniques that can directly track the evolution of microtexture and microstructure during the deformation and annealing processes. Although advanced diffraction facilities (e.g. neutron and synchrotron) have been frequently employed to investigate the evolution of deformation texture in situ (usually only on tension, compression or torsion, though), it was very difficult to apply these techniques in a rolling mill to investigate the rolling process. Heating stages have also been integrated into conventional scanning electron microscopes (SEM) and transmission electron microscopes (TEM) to enable direct observation of the recovery and recrystallization of metals during annealing, but these facilities usually require large capital investment, and there are limitations as well. For example, the maximum temperature they could reach was usually relatively low, so only a few materials (e.g. those with relatively low melting points: aluminum, zinc, magnesium, etc.) could be studied. This paper presents a simple quasi in-situ technology based on conventional electron backscatter diffraction (EBSD) techniques, which can be utilized to track the morphology and orientation changes of individual crystals in electrical steels during conventional rolling and annealing. This can help us better understand the evolution of microtexture and microstructure during these thermomechanical processes.

2. Materials and Experimental Procedure

Two non-oriented electrical steels were utilized for this study, one containing 2.8 wt% of silicon (High Silicon) and the other containing 0.88 wt% silicon (Low Silicon). The chemical compositions of the two steels are listed in Table 1. The steels were melted in a vacuum induction furnace and cast into ingots with a 200 mm × 200 mm cross section. The ingots were reheated to 1038°C and hot rolled to a thickness of about 2.5 mm in a reversing rolling mill. The hot-rolled plates were then annealed at 840°C in a dry 100% hydrogen atmosphere for 60 hours before cold rolling.

| Material       | C    | Mn | S    | P    | Si | Al     | Fe   |
|----------------|------|----|------|------|----|--------|------|
| Low Silicon    | 0.002| 0.31| 0.001| 0.01 | 0.88| 0.46   | Balance |
| High Silicon   | 0.003| 0.30| 0.001| 0.01 | 2.80| 0.52   | Balance |

To track the deformation of individual grains under plane strain compression (rolling), a rectangular plate of 100 mm × 50 mm × 2.5 mm was cut from the hot-rolled and annealed steel plate (High Silicon) along the rolling direction, and a smaller rectangular sample of 25 mm × 10 mm × 2.5 mm was cut from the plate, as shown in Figure 1a. One surface of the sample was at the rolling direction-normal direction (RD-ND) plane on the center line, and this surface was polished using conventional metallography procedures. The surface was further polished using a colloidal silica suspension (0.05 µm), and the microstructure and microtexture of a designated area (marked by microhardness indents) were characterized by EBSD, which served as the initial microstructure and texture before rolling. The small
A rectangular sample was then inserted back to the steel plate and cold rolled to pre-defined thickness reduction rates. Because the small rectangular sample was constrained within the larger plate, bulging of the RD-ND plane at the center line was essentially prevented, and the deformation was assumed to be plane-strain compression. After each thickness reduction, the surface was slightly polished using a colloidal silicon suspension, and the same area was examined under EBSD to record the changes of microstructure and microtexture so that the rotations of individual grains can be tracked during each increment of deformation, which was otherwise very difficult to be realized using conventional methods.

To track the nucleation and grain growth of the deformed material during annealing, the Low Silicon steel (0.88 wt% Si) was inclined cold rolled at an angle of 45° to the hot rolling direction \([10]\) to a thickness of 0.5 mm. An area on the deformed sample was first marked using microhardness indents and examined under EBSD (Figure 1b), which recorded the microstructure and microtexture of the material before annealing (after cold rolling). The sample was then annealed at a fixed temperature (650°C) for different times to track the nucleation and grain growth of individual grains during recrystallization. After each annealing time, the sample was quenched in cold water to freeze the microstructure. The previously examined surface was slightly polished using a colloidal silicon suspension to prepare the surface for EBSD scan. Usually about 1-2 µm of the surface will be removed by this polishing step. To effectively track the start of recrystallization, the sample was examined at short intervals at the beginning, since the recrystallization occurs very fast from the deformed microstructure. The sample was annealed with extended holding times once all the grains are recrystallized, since the growth of the recrystallized grains within a matrix consisting of all new grains is much slower than the recrystallization from a deformed matrix. In this way both the initial nucleation and the grain growth processes can be traced.

![Figure 1. Schematic illustration of the quasi in-situ EBSD characterization technique: (a) tracking the cold rolling process, (b) tracking the annealing process.](image)
3. Results and Discussion

3.1. Tracking cold rolling

An example of quasi in-situ EBSD characterization of the cold rolling of a 2.8 wt% Si non-oriented electrical steel is shown in Figure 2. An initial EBSD scan of the hot-rolled and annealed sample was made to reveal the microstructure and microtexture before rolling. The scanned area consists of about 100 grains on the RD-ND section at the center line (Figure 2a). After 8% thickness reduction, essentially all these grains can still be identified, and the orientation change of each grain can be clearly noticed from the variations in color within the individual grains (Figure 2b).

![Figure 2](image-url)

Figure 2. An example of tracking the individual grains during plane-strain compression: (a) EBSD inverse pole figure (IPF) map before deformation, (b) IPF map of the same area after 8% thickness reduction. The textures shown to the right are $\phi_2=45^\circ$ sections of the orientation distribution functions (ODFs) based on Bunge’s notation.
Although with only a small thickness reduction (8%), shear bands are already visible in some of the grains (e.g. 2, 22 and 27). It is also noticed that grain subdivision occurred in most of the grains, as can be seen from the color differences within the deformed grains. However, some grains did not show apparent shape or color change, e.g. grains 10, 53 and 58. Apparently, the rotations of these grains are more difficult than the others under plane-strain compression conditions. Some originally separated grains (with distinguished grain boundaries) are merged after deformation, and the grain boundaries may disappear due to the merging. For example, grains 22/59 and 23/20 are merged into two single grains (without apparent boundaries) after deformation. Other examples include grains 37, 55 and 56, which also merge into a single grain after deformation. Thus it is seen that, by quasi in-situ EBSD tracking of the individual grains during rolling, it was able to observe some interesting phenomena that might have been overlooked during conventional experiments, where tracing the deformation of the same grains was not possible. The orientation data obtained from the tracing of individual grains can be conveniently used to verify the theories used to predict deformation texture [14]. The overall texture of the sample also shows apparent changes, i.e. the initially strong {113}<581>, {110}<557> and Goss textures are weakened and rotated to {115}<7 12 1> and {110}<115>, and the γ-fibre texture was considerably strengthened, even if the reduction rate is only 8%.

Another example of deformation tracking is shown in Figure 3, where a few grains with near \{111\}<121> orientations (γ-fibre) are isolated from the deformed matrix, and their deformation is tracked from 10% to 20% reductions. It is seen that even though the shapes of the grains are considerably changed, the orientations of these grains are essentially maintained the same as the undeformed ones. The γ-fibre grains have relatively high Taylor factors, and thus have high resistance to plane strain compression. During deformation, these grains tend to retain their orientations. However, apparent orientation variation was noticed in each grain, which contributes to the increase of the orientation spread and the rotation of the original \{111\}<121> orientations to other orientations (as indicated by arrows). This also results in the decrease of the intensity of the original grains.

![Figure 3](image)

Figure 3. An example to track the deformation of near \{111\}<121> grains during cold rolling: (a) before rolling, (b) 10% reduction, (c) 20% reduction. Textures are shown on \(\varphi_2 = 45^\circ\) sections of the ODFs (Bunge notation).

### 3.2. Tracking recrystallization

Figure 4 shows an example of tracking the recrystallization of a cold-rolled 0.88 wt% Si steel after inclined cold rolling. Figure 4a illustrates the early stages of annealing when new grains nucleate from the deformed matrix, and grow gradually while other nuclei are formed in the meantime. It is seen that,
After cold rolling, the steel is mainly composed of elongated [111]/ND and [001]/ND grains with numerous shear/deformation bands within these grains. At the beginning of recrystallization (3 minutes), nucleation preferably starts from the deformed [111]/ND grains, either at the grain boundaries (marked with rectangles) or within the deformed grain along the shear bands (marked with ovals). Only one grain (marked with a rhombus) is noticed to nucleate within a deformed [001]/ND grain. When the annealing time is increased to 6 minutes, some of the new grains grow considerably (e.g. regions 1, 4 and 3), while others only grow slightly (e.g. regions 2, 7 and 6). New grains are also seen nucleated from the deformed matrix, e.g. regions 8, 9 and 10. At this moment, the growing grains are usually constrained within the elongated grains from which they nucleate, and they do not cross the boundaries of those deformed grains. After 10 minutes, all the deformed grains were replaced by recrystallized grains, due to the continued growth of the already recrystallized grains, and due to the nucleation and growth of new grains from the remaining deformed matrix.

Once the recrystallization is complete, i.e. the deformed matrix has been completely consumed and replaced by new grains, further annealing will result in grain growth within the all-recrystallized microstructure, which is much slower than the nucleation and growth of grains from the deformed matrix. There is no nucleation after this, and the subsequent grain growth will determine the final microstructure and texture. Figure 4b shows an example of tracing grain growth during later stages of annealing, where the growth of some grains is tracked during annealing from 54 minutes to 329 minutes. At the lower left corner, the large, red “R” grain (with a “C” shape) is seen gradually embracing the smaller light-green “G” grain inside it, and finally consuming the “G” grain after 149 minutes, to form an equiaxed grain with an almost circular shape. The “B” grain near the top has experienced a different path: initially (54 minutes) a small grain (pink) is seen trapped inside this grain, which is gradually “eaten” by the “B” grain to form two small islands (74 minutes), and are completely consumed after 104 minutes. The size of the “B” grain itself essentially does not change, though, from 54 to 104 minutes, i.e. it does not grow except that the grain inside it has been consumed. In the meantime, all the neighboring small grains around the “B” grain are gradually consumed by the larger growing grains adjacent to them. After 149 minutes, the “B” grain becomes a small grain as all the neighboring grains have grown to much larger than it, and it starts to be consumed by other grains (209 minutes). After 329 minutes, it essentially disappears. These direct observations clearly confirmed that during grain growth, the larger grains had a size advantage, and will eventually grow by consuming their smaller neighbours.

The above observation directly illustrates the morphology changes of individual grains during later stages of annealing. It should be mentioned that, the orientation data obtained from the EBSD scans can be utilized to evaluate the misorientation and mobility of the grain boundaries of each growing grain, which can help unveil the mechanisms governing the growth process. A few examples using such data to elucidate the recrystallization mechanisms, especially during grain growth, have already been presented in the literature [15, 16].

It is thus seen that the quasi in-situ EBSD technique presented in this paper provides an effective experimental tool to directly observe the rolling and annealing of electrical steels, which can give insights into the mechanisms governing these processes. However, it should be noticed that, these tracking techniques require careful design of the experiments, and also need good sample preparation skills for EBSD characterization. For rolling tracking, the sample should be cut precisely, and be able to be inserted back into the sample holder so that bulging of the examined surface can be minimized. The sample holder should be the same material (with the same processing history) as the sample to avoid the difference in deformation between the sample and the sample holder. The amount of thickness reduction that can be reached before distorting the examined surface is highly dependent on how the experiments are conducted. Usually, a total of 25-30% thickness reduction can be readily achieved. With more than 30% reduction, the examined surface becomes largely elongated and the bulging of the plane may prevent further tracing of the same area.

For the tracking of recrystallization, there is no bulging issue of the plane to be examined. However, the heating and quenching of the sample during the experiment, i.e. interrupting the annealing process, may result in different behaviors from the real annealing process (continuous). Nevertheless, the results...
shown in this study did provide some insights into the nucleation and grain growth processes. This technique can be used as an effective experimental tool to investigate the recrystallization of non-oriented electrical steels.

4. Summary and Conclusions
In this paper, a quasi in-situ EBSD characterization technique was presented as an effective experimental tool to investigate the cold rolling and annealing of non-oriented electrical steels.

The changes of morphology and orientation of individual grains during plane-strain compression can be clearly followed at specific thickness reductions. The orientation data can be used to verify crystal plasticity simulations that involve the effect of grain boundaries. Some interesting phenomena, such as
the formation of shear bands, merging of separate grains, and the retention of some grain orientations during deformation can be directly observed.

The tracking of the annealing process can be used to investigate the preferred nucleation from the deformed matrix, and the growth of the nuclei while new grains are created in the meantime. The tracing of the later grain growth process clearly showed how the small grains were consumed by the large grains during extended annealing holding. The information on grain misorientations can be used to evaluate the mobility of the grain boundaries, and thus understand the formation mechanisms of the final texture.

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