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Microstructure and Texture Evolution During Annealing after Temper Rolling of a 2 wt.-% Silicon Electric Steel

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Abstract. The evolution of the microstructure, macrotexture, microtexture and mesotexture has been studied during the annealing at 760ºC after temper rolling (9% thickness reduction) of a non-oriented electrical steel sheet containing 2 wt. % Si. Results showed that the coarse grained microstructure, obtained on annealing, is produced through a recrystallization mechanism that advances from the surface to the interior of the sheet. However, starting of this process is delayed due to the presence of Si. The majority of experiments carried out in this work have been repeated for a low-carbon steel (C = 0.0385; Mn = 0.18%) containing only 0.03% Si and the results obtained were practically identical to those observed in the steel containing 2% Si. The main difference observed between both steels was that the process of formation of the exaggeratedly large grains was slower in the steel containing 2% Si.

Introduction

Non-oriented electrical steel is usually employed in the production of electrical motors. For this application, the ideal texture is the {001} fiber, i.e. the {100} family of planes that are parallel to the sheet surface and the <uvw> direction is isotropic. Under these conditions, magnetic losses due to hysteresis are reduced. In order to obtain the adequate grain size with consequent increase in electrical properties, the sheet in its final manufacturing stage receives a small (2% to 10%) reduction with further heat treatment. In this process, during annealing, large grains appear having a size range of 150 to 300 μm, with a consequent change in the texture of the end product [1,2]. The mechanism responsible for the appearance of the large grains (abnormal grain growth or primary recrystallization) and for the formation of the texture due to this process still is a subject of several studies and some points need further clarifying.

Experimental procedure

The material studied in this work was a non-oriented grain electrical steel sheet, with the following composition in wt. %: C = 0.0036%; Si = 2.03%; Mn = 0.124%; P = 0.006%; S = 0.0089%; Al = 0.137%; Cu = 0.018%; Cr = 0.09%; Ni = 0.034% and Mo = 0.012%. The steel was supplied in the form of a hot rolled coil with 2 mm thickness and 1200 mm width and subsequently semi-continuously pickled at 85°C in a 13% chloridric acid solution and then cold rolled with a 65% thickness reduction. Following, the material was annealed at 640°C for 8 hrs in an industrial high convection box furnace (95% N_2 and 5% H_2). Finally, it was cold rolled with a 9% thickness reduction. From this material samples were taken and heat treated in the laboratory for 5, 10 and 30 minutes at 760°C in a barium chloride bath. Several *in-situ* experiments have been carried out, were a region of the sample has been identified with the help of hardness indentations and the same region analyzed in a MEV/EBSD system after several successive annealing treatments. In these *in-situ* experiments, annealing was carried out also at 760ºC for 5, 10, 15 and 20 min., in a high purity argon atmosphere, in order to avoid sample surface damage. The macrotexture of the samples in the normal direction have been determined by X-ray diffraction in a Rigaku DMAX-2100 diffractometer, with a horizontal multipurpose texture goniometer, employing a Mo Kα radiation. Four incomplete pole figures {110}, {200}, {211} and {310} have ben measured on the surface of the samples. The microtexture and mesotexture have been carried out by back scaterred electron diffraction (EBSD) with an acquisition and identification system developed by TexSEM Laboratories Inc. that was attached to the XL-30 Philips scanning electron microscope, operating with a 20kV acceleration voltage.

Results and Discussion

After 65% cold working followed by annealing at 640ºC for 8 hours, the sample microstructure presented complete recrystallization with equiaxial grains having 30 μm average diameter. The 9% thickness reduction practically did not bring any microstructural modification at the optical microscopy level (Fig. 1a). Whether using optical microscopy or using microhardness measurements, it was not possible to detect any work hardening gradients through the sheet thickness. After 5 minutes annealing at 760ºC still no exaggeratedly large grains could be observed (Fig. 1b). After 10 minutes, large grains started to appear at the sample surface (left side of the micrographs) (Fig. 1c) and after 30 minutes (Fig. 1d) it was completed.

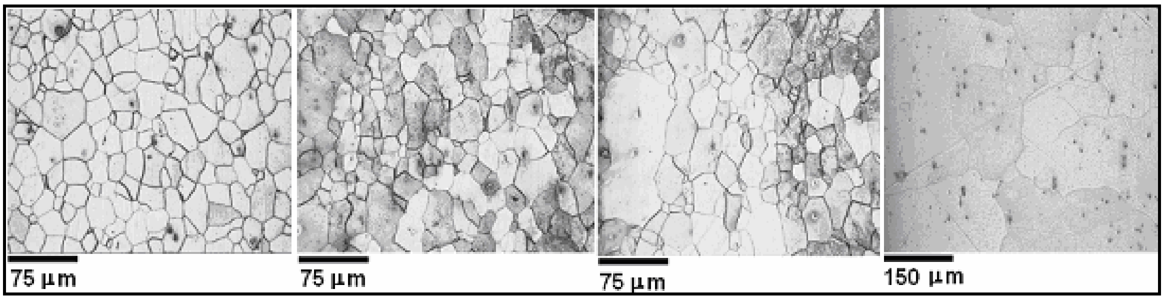

Figure 1: Optical microscopy of longitudinal section of the samples etched with 2% nital: a) cold rolled 9% thickness reduction; b) after cold reduction and annealing in a salt bath at 760ºC for 5 minutes; c) after cold reduction and annealing in a salt bath at 760ºC for 10 minutes; d) after cold reduction and annealing in a salt bath at 760ºC for 30 minutes. Sample surface lays on the left of the micrographs. Note that images are with different magnifications.

The analysis of X-ray diffraction textures has been performed at the sheet surface (rolling surface). The orientation distribution functions (ODF) for the 2 % Si steel 9 % cold worked, 9 % cold worked and annealed for 5 min in salt bath at 760ºC were determined, but the ODFs of the annealed samples for 10 and 30 minutes after cold working are not useful since the material presented very large grains (with an average diameter of about 300 µm, as shown in Fig. 1d), and, therefore, in a small number, which leads to pole figures that are statistically unreliable. This aspect can be easily visualized comparing the (110) pole figures of the 9% cold worked and the 9% cold worked and annealed for 10 min in salt bath at 760ºC, because pole figures for the 9% cold worked and annealed in salt bath for 10 min at 760ºC do not present such symmetry, since they are coarse grained materials. It has been observed the presence of preferential orientations typical of low carbon steels, deformed with a thickness reduction in the range of 65 to 70 %, i.e. the <111> low intensity fiber and the presence of the high intensity components (001)<110> and (110)<001>. These results agree, for instance, with those obtained by Park and Szpunar [3]. The ODF analysis, obtained by X-ray, do not show any significant preferential orientation due to annealing at 760ºC for 5 min, i.e., the low intensity <111> orientations and the high intensity (001)<110> and (110)<001> orientations are practically unaltered, being impossible to evaluate the orientations that suffered major deformation. The results clearly show the experimental difficulty related to the evaluation of the preferential orientation and its relation with the exaggeratedly grown grains using data coming from macrotexture, measured by X-ray diffraction. The possibility of analyzing automatically the Kikuchi lines, using scanning electron microscopy along with back-scattered electrons, opens a new perspective for studying the texture stemming from the growth of exaggeratedly large grains, fact that has been explored in the present work.

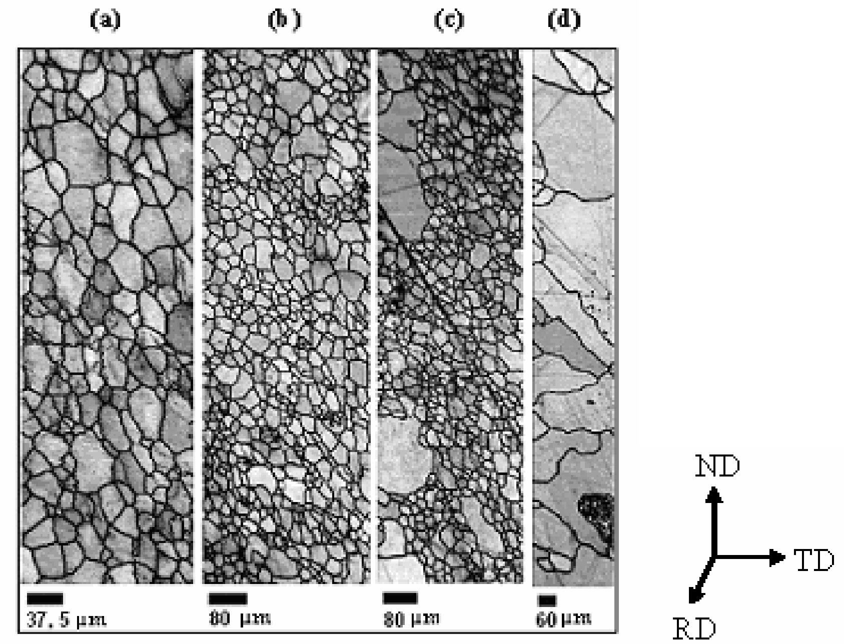

Figure 2: Orientation maps, section **transverse** to the rolling direction, obtained by OIM, for the conditions: (a) cold worked; (b) heat treated for 5 minutes; (c) heat treated for 10 minutes; (d) heat treated for 30 minutes.

Analysis performed using EBSD has been conducted in three sections of the sheet: normal ND (at the rolling surface), longitudinal RD (rolling direction) and transverse TD. Orientation maps have been determined, wherein each color represents the grain crystallographic orientation in relation to the surface normal where measurements have been performed. Orientation maps, which define the range of angle misorientation between grains, are obtained by orientation imaging microscopy (OIM), have also been performed, where colors, in this case, define if the boundaries are low, medium or high angle. Figures 2 (a), (b), (c) and (d) present the orientation maps of the samples in different conditions, where colors define the range of angle misorientation between grains, with **transverse** section to the rolling direction.

As it may be observed from Table I, the preferential orientation between grains is predominantly given by Σ3 and by Σ13b, i.e., a 60 ^{o} rotation around the <111> direction and of 27.79^{o} around the <111> direction. Histograms have been produced in order to evaluate the relationships between the coincidence sites in the lattice, that are special boundaries.

Table I: Frequency of occurrence of the between grains orientation relationship and lattice site coincidence for different heat treatments of the 2% Si steel. CW=cold worked, NS=normal section, LS=longitudinal section, TS=transverse section and HT=heat treated.

Sample	**Frequency of occurrence of the 60^{0} orientation**	**Frequency of occurrence of CSL Σ3**	**Frequency of occurrence of the 30^{0} orientation**	**Frequency of occurrence of CSL Σ13b**
CW NS	0.14	0.10	0.07	0.03
CW LS	0.12	0.12	0.075	0.03
CW TS	0.09	0.08	0.07	0.02
HT 5 min NS	0.12	0.09	0.07	0.01
HT 5 min LS	0.14	0.14	0.10	0.04
HT 5 min TS	0.04	0.03	0.08	0.04
HT 10 min NS	0.06	0.04	0.09	0.06
HT 10 min LS	0.14	0.14	0.07	0.02
HT 10 min TS	0.09	0.09	0.07	0.03
HT 30 min NS	0.42	0.37	0.05	0.08
HT 30 min LS	0.40	0.40	0.05	0.04
HT 30 min TS	0.35	0.33	0.01	0.005

In such a way, it has been possible to verify if the orientation relationship between grains was randomlike , i.e., if it followed the Mackenzie distribution [4,5]. The produced CSL histograms and the grain orientation between grains for the samples are those for the longitudinal section, here not reproduced for reasons of space. However, analysis of the CSL histograms showed that the Σ3 and Σ13b, i.e., 60^{o}<111> and 27.79^{o}<111> respectively, are the most frequent orientations. This suggests that this orientation relationship is a stable one and that when boundaries with such an orientation meet grain boundary their movement is hindered, making that other boundaries may be consumed first. If such a phenomenon occurs based on the oriented growth theory [6], the boundaries of these nuclei grow with an orientation relationship with the deformed matrix of 40^{o}<111>. These boundaries are forming a CSL of the Σ3-type. Supposing the oriented nucleation theory, the phenomenon would be the same, i.e., the nuclei have pre-defined orientation given by the deformed matrix, depending from the site where nucleation started, leading to preferential growth of the nuclei. In such a case, one could not affirm which would be the final orientation relationship between these nuclei, not knowing the site in which preferential nucleation occurred and the type of the orientation. Furthermore, the frequency of occurrence of these special boundaries, when compared to the between grain orientation relationship, they become more alike with increasing heat treatment time.

From these results it may be observed that the stable orientation relationships that hinder continuity in grain growth in the tested temperature are special $\Sigma3$ and $\Sigma13b$ boundaries. Relative to the distribution of the between grain orientation, it may be observed that the material in its different states does not present a random distribution. This effect becomes more accentuated as grains grow, pointing out the 30^{o} and 60^{o} orientation relationships.

Moreover, according to Hutchinson [7] there are different nucleation modes, as described in the following:

i) Nucleation can occur in the grain interior by subgrain growth. In the sequence, to form high angle boundaries-HABG, which have high mobility, it is necessary that subgrain boundary density and the misorientation between grains must be high, i.e., that the stored energy of the deformed grains, in which nucleation starts, must be high. Stored energy decreases following the orientation in the deformed state, i.e., ({111}<uvw>, {112}<110> and {001}<110>). This means that recrystallization should start in the {111}<uvw> oriented grains, whilst nucleation in the {001}<110> oriented grains should happen with smaller frequency. Furthermore, these grains may be consumed through the growth of other grains before nucleation starts. Nucleated grain growth, by this type of nucleation, favors the texture component with high stored energy in the worked state, at the expense of those with lower stored energy. In this case, there is a greater possibility of the occurrence of grains belonging to the recrystallized γ- fiber texture.

ii) Another mechanism that may occur is the nucleation at HAGB. If the grain boundary is located between grains with different stored energies, there is a displacement force that results in the movement of the boundary to the interior of the grain of higher stored energy, called strain induced grain boundary movement (SIBM), producing an opposite recrystallization behavior if compared to the previously mentioned mechanism. In such a case, nucleation starts with low stored energy orientations such as those of the {001}<110> and {112}<110> orientations. Due to the high cold rolling reductions, grain boundary density is high so that there is a considerable amount of SIBM grains.

iii) The third mechanism to be considered is the nucleation adjacent to second phase particles. High deformations around precipitates conduce to preferential nucleation in these regions, independently of orientation of the deformed matrix. In such cases the recrystallization texture is weak.

These mechanisms operate in a competitive way, so that, in principle, grains that nucleate first have more time to grow and dominate the recrystallization texture. In spite of this fact, none of the three mechanisms is suppressed. Consequently, despite nucleation mechanisms occur at distinctive times, temporal dependence of nucleation has little importance, once grain boundary mobility is sluggish due to the locking of the boundary movement. This effect may be caused by manganese or carbon in solid solution [8] and, in this case, also by silicon. In this way, the nuclei that start the primary nucleation, not necessarily will be the first ones to grow, consuming other nucleation sites with less favorable orientations.

In this work it has been shown that the material surface presents a higher defect density than the central part and that the exaggeratedly large grains occur from surface to the center of the sample. Therefore, it seems reasonable to suppose that the appearance of the exaggeratedly large grains occurs by recrystallization, starting in the more deformed grains and consuming the grains with orientation close to {011}<100>.

Finally, it should be mentioned that the majority of experiments carried out in this work have been repeated for a low-carbon steel (C = 0.0385; Mn = 0.18%; Fe = 99.692) containing only 0.03% Si and the results obtained were practically identical to those observed in the steel containing 2% Si. The main difference was that the process of formation of the exaggeratedly large grains was slower in the steel containing 2% Si, fact that allowed an easier follow up of the phenomenon and, for that reason, the higher Si containing steel has been chosen for this work.

Conclusions

Cold rolling with a low thickness reduction (9%) followed by an annealing heat treatment at 760ºC leads to the formation of exaggeratedly large grains in an electrical steel containing 2% Si. After annealing, a higher incidence of sub-grains at the surface as compared to the center of the samples has been observed, showing the presence of a strain gradient along the thickness.The appearance of exaggeratedly large grains occurs first at the surface and later at the center of the sample. Fast heating (molten salt bath) favors the appearance of exaggeratedly large grains, in comparison to slower heating rates (Ar).

The macrotexture analysis by X-ray diffraction was infeasible in the study of texture evolution of the growth of exaggeratedly large grains. The stable orientation relationship between large angle grain boundaries of the fully recrystallized structure is $\Sigma 3$ and $\Sigma 13b$.

Silicon delays the formation of the exaggeratedly large grains after temper rolling.

The EBSD technique has been shown to be adequate to evaluate the orientations of regions delimited by low angle grain boundaries.

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