Formation of the Goss Texture in a Thin Foil Experiment on Fe–3.2%Si

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Fe–3.2%Si steel from a commercial source supplied in the form of fully recrystallized sheet was selectively thinned to produce foils of different thicknesses which separated the subsurface layers in which the {110} texture dominated, from the central layer of {111} texture. Goss oriented grains were more frequent in the subsurface layers, but were also present in the central portion of the material. Annealing these foils at \( \sim 1000^\circ C \) produced Goss texture in the surface foil, and \( \{111\}(hkl) \) in the central foil. From these experiments it is clear that Goss secondaries grow easily in the \( \eta \) layer, but not in the \( \gamma \) layer and this was proved in a sequential heating experiment. A search of the misorientations between Goss oriented primary recrystallised grains in the \( \eta \) oriented volumes, for the most likely CSLs, \( \{SS\} \) and \( \{S13a\} \), proved to be unsuccessful. Some were found, but not in sufficient numbers to provide a satisfactory explanation for the formation of Goss secondaries. It is suggested however, that if CSLs are important in the selection of Goss secondaries, that Si segregation has also to be considered, for this will be less in special boundaries and thus provide less solute drag.

KEY WORDS: Goss texture; secondary recrystallization; orientation imaging microscopy (OIM); silicon segregation to grain boundaries.

1. Introduction

The only large scale exploitation of the secondary recrystallization process is in the production of Fe–3%Si for laminations in large power transformers. The Si increases the resistivity of the material, thus reducing the core loss associated with induced currents, and the secondary recrystallization texture is \{110\}/H20855\{100\}, the Goss texture. This texture allows magnetic saturation at the lowest field strength,\(^1\) and the total core loss in the highest quality steels is of the order of 10% of that measured in randomly oriented low carbon steel, which has a core loss of \( \sim 15 \) watts/kg.

The oldest production route of grain oriented Fe–Si involves two rolling and annealing cycles, the second annealing temperature being high enough to dissolve MnS particles, thus freeing previously Zener pinned grains for secondary recrystallization, which produces Goss oriented crystals of millimeters diameter running through the sheet thickness which is typically \( 0.2–0.3 \) mm.\(^2\) A more modern process uses AlN particles as pinning agents and a single rolling and annealing cycle.\(^3\)

Theories of texture formation in recrystallization are usually described as belonging to the Oriented Nucleation (ON) or Oriented Growth (OG) schools. In the case of the Goss texture \{110\}/H20855\{100\} formed in Fe–3%Si during secondary recrystallization, both of these ideas have been used by various groups to explain the extraordinary sharpness of the texture, but as yet, there is no widely accepted view as to its origin. For oriented nucleation the nuclei have to be special in some way, and an early suggestion was that Goss crystals should be larger than the matrix of recrystallized grains containing them.\(^4\) This size advantage, leading to curvature driven growth was accepted until Pease et al.\(^5\) showed that Goss oriented grains were not larger than the grains which surrounded them. With the acceptance that Goss grains were indeed of similar size to the matrix surrounding them, it was nonetheless possible to gain a size advantage for Goss grains by postulating that they could coalesce to form a larger volume.\(^6\) The details of such a grain coalescence theory had been given earlier by Nielsen.\(^7\) As far as the authors of this paper are concerned, no credible alternative explanation of what makes Goss grains special in terms of nucleation potential has emerged from the mass of literature now extant. Instead there are two broad theories based on OG which are currently popular.

The first is based on the suggestion that Goss oriented grains have many more neighbours which have special misorientation relationships with them.\(^5,9\) These special misorientation relationships are usually described as Coincident Site Lattices (CSLs) and it is commonly expected that boundaries between such grains will have a low energy. This low energy boundary is expected to be pinned by smaller precipitates compared with larger precipitates formed in high energy/high angle boundaries after primary
recrystallization. During the second anneal, in which precipitates are dissolved, the boundaries which are unpinned first will be those with the smaller precipitates and so Goss grains with a CSL boundary might be expected to have a growth advantage. This would be especially so in the case of slow heating to the secondary recrystallization temperature of >1 000°C. There is nothing in this theory to explain why Goss grains are not themselves consumed by the Secondary Recrystallization process.

A second variant of OG is based on the notion that higher mobility is a characteristic of high energy (HE) boundaries.10,11) Such boundaries are expected to have larger precipitates associated with them which coarsen more quickly during the heating stage, but before dissolution. Thus, at a critical stage they will be subject to less pinning, giving a growth advantage.

The basic problem with both OG theories is that the purported successful secondaries depend on grain misorientations of their neighbours. Now the orientation spread of the texture in which the Goss and near Goss grains are located is very large, much larger in fact than the spread of the final Goss texture, which can be as low as 7°.12) There has to be another mechanism which naturally favours the more precisely oriented Goss grains.

Finally, a theory of a different kind has been recently proposed by Park et al.,13) based on solid state wetting and differential interfacial energy between grains. They showed isolated grains trapped in growing secondary grains to be in the process of being consumed by penetration of the growth front, the penetration being a function of the wetting angle.

It has been known for many years that the Goss nuclei are located in the sub-surface layers of the sheet following the first anneal in the two stage process.31) In X-ray work Mishra et al.15) showed that the sub-surface material had a texture (100)/ND and designated the η fibre, while the central portion of the sheet had a texture (111)/ND, designated as the γ fibre. Since we are dealing with the primary recrystallisation textures there is no sharp interface between the different texture components in the layers, but connectivity and interpenetration between the layers exists. The present work reports the results of an experiment in which the differently textured layers were separated by selective etching and then annealed under conditions known to produce Goss secondary grains. The central idea is that of breaking the connectivity between layers of material having different textures and of following texture development in the isolated layers to see to what extent secondary recrystallisation depends on both layers.

2. Experimental Procedure

Commercially produced Fe−3.2%Si steel of the composition shown in Table 1 was made by casting, hot rolling and pickling the material and then subjecting it to a two stage rolling and annealing cycle. The first cold rolling was from the hot band to 0.7 mm followed by annealing at 950°C under a reducing atmosphere (a mixture of wet H2/N2) for 30–40 s. The second cold rolling was from 0.7 to 0.27 mm followed by a decarburizing anneal at 820–830°C for 4–5 min under a wet reducing atmosphere similar to the first atmosphere. This resulted in a fully recrystallized sheet stabilised by MnS with an average equiaxed grain size of ~18 μm measured by the line intercept method.

Secondary recrystallization was achieved by annealing the primary recrystallized sheet in a vacuum furnace at 1 000°C or in a salt bath at 970°C. Thin foils were prepared by controlled electrolytic removal of material to produce foils of the mid plane and subsurface layers of the sheet using a 10% perchloric/acetic acid solution at ~10°C and a polishing voltage of 24 V. Some of the material was subjected to multiple heating cycles in a vacuum furnace. The crystallographic and microscopic changes were monitored during the interval between two consecutive cycles. The microstructural and textural developments were followed using a LEO 1530 FEG SEM (field emission gun scanning electron microscope) which was equipped with EBSP (electron back scattered pattern) detectors to measure crystallographic information. HKL Channel 5TM software was used to analyze the data. Information was gathered from the rolling plane and from the cross-sections perpendicular to the rolling direction (RD) and transverse direction (TD).

3. Experimental Results

Large montages of channelling contrast (CC) images many hundreds of square microns were prepared from the rolling plane, TD and RD cross-sections of the as-received sheet. The largest grain diameters were of the order of 40 μm and the smallest ~5 μm in a continuous distribution, and the average intercept length was 18 μm. No particular texture was associated with the largest grain sizes and the grain size distribution was the same on the surface as in the cross-sections.

Textures from the cross-sections were determined by EBSP and the results are shown in the {100} pole figures in Fig. 1. As expected for material processed in the two step route, the textures comprise two fibres which are described here as the γ fibre, (111)/ND and the η fibre, (100)/RD. Figures 2(a) and 2(b) shows orientation imaging microscopy (OIM)15) images of representative RD and TD cross-sections shaded to show the main γ fibre components {111}(112) and {111}(110), and Figs. 2(c) and 2(d) show the η fibre oriented grains, i.e., (100)/RD. Montages in Figs. 2(a) and 2(c) are from the same area, as are (b) and (d), and the orientations are within a 15° spread. It is clear that η fibre material is contained within the first quarter thickness from the sheet surfaces, this being especially clear in the RD cross-section, Fig. 2(c). The γ fibre material is more generally distributed with a slight tendency to cluster in the central half layer, Figs. 2(a) and 2(b). This is a well known spatial distribution of orientations in material processed by the double rolling processing route. Figure 3

| C   | Mn  | S   | Si | Cu | Fe   |
|-----|-----|-----|----|----|------|
| 0.04| 0.06| 0.02| 3.2| 0.16|96.52 |
shows the same areas, only this time the Goss orientations are shaded. The Goss grains are located in the \( \eta \) fibre distribution and in this the Goss grains are concentrated. It is plain from Figs. 2 and 3 that the \( \eta \) fibre is more intense in some regions than in others, i.e., it occurs in patches across the sheet plane and it is most concentrated in the sub-surface quarter thickness layers.

Figure 4 shows the effects of heating at 970°C in a salt bath for 1, 5 and 10 min. Secondary recrystallization has begun in the top surface after 1 min, Fig. 4(a), and that it originates at both surfaces is the most probable explanation for the centre-line effect after 5 min annealing, Fig. 4(b). After 10 min secondary grains have grown to impinge both surfaces, and the process is now almost complete, Fig. 4(c). The orientation of the obvious secondary grains are widely scattered around the Goss orientation after 1 min, Figs. 4(d)–4(e) and the texture quite obviously becomes much more closely aligned with Goss as time at 970°C continues,
This texture sharpening is also well-known. In order to check the effects of the as-processed surface on subsequent secondary recrystallization, about 20–25 μm of material was removed from both sides before annealing. This is indicated schematically in Fig. 5(a), and the texture results after complete secondary recrystallization i.e., when secondaries were of sheet thickness, are shown in Fig. 5(b). Comparing Figs. 4(f) with 5(b) shows that any effects associated with the surfaces are not significant.

A sub-surface foil of 80 μm in thickness was carefully electropolished from the as-received sheet, and another foil of this thickness was prepared from the centre of the sheet. The geometry is shown in Fig. 6(a). It was found that secondary recrystallization occurred rapidly in both foils, but growth stagnated after 570 min at 1 000°C in the vacuum furnace. In the sub-surface foil grains ranged in size from 100 to 1 300 μm, while in the central foil the range was smaller, i.e., from 100 to 900 μm. In all cases the largest grains occupied the whole thickness of the foils. Fig. 6(b) shows a {100} pole figure of all the grains of size greater than 100 μm, and Fig. 6(c) those larger than 600 μm found in the sub-surface foil. It is plain that the largest grains shown in Fig. 6(b) contain a subset clustered around the Goss orientation, Fig. 6(c). When the data from the central foil is similarly processed, the texture is mostly {111}/hkl, as is the subset from the largest grains, Fig. 6(e).

This set of results indicate that Goss oriented grains, which exist throughout the thickness of the sheet, grow preferentially in the subsurface η fibre layer Fig. 2. That it can and does grow preferentially in the η layer is proved by the thin foil experimental results shown in Figs. 6(b) and 6(c), and that Goss is not particularly favoured in the γ texture is proved by Figs. 6(d) and 6(e).

During annealing in the vacuum furnace the “jerky” growth of secondaries deduced and observed by Hutchinson and Homma is presumed to happen in the foils. In the “stopped” phase of the process thermal grooving will occur if the grooving kinetics are fast enough. In any case of this kind, the boundary will advance leaving behind the traces of the grains which have been consumed. Figure 7 shows such a situation. Grains X and Y shown in CC in Fig. 7(a) are actually close to Goss, Figs. 7(b) and 7(c), and inspection of the CC image shows the grooves of grain boundaries of grains which have been consumed by the migrating front. The OIM and CC images provide direct evidence that Goss grows in η oriented material.

In order to restrict or prevent significant growth in the sheet plane normal direction a subsurface foil of 30 μm in thickness was prepared as in Fig. 8(a). This was heated to 1 000°C until stagnation, and examined. Figs. 8(b) and 8(c) show large grains in a matrix of much smaller grains, and Fig. 8(d) is a pole figure showing the orientation of 19 such
grains. It is clear that the few large grains which developed in the foil, all belong to the η fibre, with some close to the Goss orientation.

Figure 9 is from a 30 μm foil heated to 1000°C in a vacuum furnace, cooled, examined, reheated and examined again. This shows a growing grain Z located in a matrix of generally growing grains belonging to both the η and γ fibres. Clearly grain Z is Goss oriented (Figs. 9(c) and 9(e)) in a matrix of η fibre grains, which are not shaded, plus grains of orientation {111}{112} and {114}{148}. On reheating, grain Z grows into the material outlined in the figure, and inspection of the grooves in Figs. 9(a) and 9(b) shows that a small amount of grain growth was all that occurred in this part of the matrix before it was consumed. The orientations {411}{148} and {111}{112} are important in the OG theory of texture development for they have a S9 relationship with Goss. Interestingly, however, the lower magnification OIM shown in Fig. 9(d), shows that while significant growth had occurred in the neighboring η fibre material the material outside the envelope of orientations {111}{112} and {411}{148} had not been consumed by Z. It is therefore safe to conclude from results such as these in the 30 μm foils, that growth of grains to large sizes compared with the average grain size, is concentrated in the η fibre material, even when there is volume of γ and {411}{148} material present contained in the η layer in which Goss is growing.

A foil of approximately one grain in thickness was prepared from the subsurface layer, i.e., ~18 μm, and heated to 1000°C in the vacuum furnace. No secondary recrystallization occurred, even after extended periods. However there was significant general grain growth, which presumably was allowed to occur because there were no invading secondary grains.

General grain growth was observed in all of the sections examined, and the OIMs allowed Fig. 10 to be constructed. The data specifically excludes obvious secondaries and is gathered at the time when obvious secondaries had formed, and the graphs show that the spread of grain size widens and average grain size increases as the specimen thickness reduces. It is clear that Goss secondaries appear before much growth has occurred in the full thickness and 30 μm
foils. The 18 µm foil shows general grain coarsening with no secondaries observed.

4. Discussion

The Goss texture formed during hot rolling is inherited through the subsequent double cold rolling and annealing and lies at the centre of the texture spread (100)//RD, i.e., the η fibre which dominates the sub-surface annealed texture, Figs. 2(c) and 2(d). The inheritance process is necessarily complex but is initiated at the hot band stage when surface shear at high temperatures dominate and dynamic recrystallization or recovery allow Goss to become a prominent feature of the hot rolled texture. On the other hand, the centre of the sheet under these hot rolling conditions will experience plane strain leading to the conventional bcc annealing texture centered on {111}. This is also inherited, most probably through the mechanism of deformation banding of the γ deformed material, leading to the central layer after final annealing at 850°C having a {111}⟨hkl⟩ texture. Thus Fig. 1 comprises two fibre recrystallization textures, η and γ which are more concentrated in the surface and interior layers respectively, as shown in Fig. 2.

At 1000°C in the vacuum furnace or at 970°C in the salt bath, secondaries grew parallel to the surface in a pancaked and interior layers respectively, as shown in Fig. 2. The central line between grains nucleated near the surface indicates the symmetry of the processing, Fig. 4(b). Both this observation that growth invades towards the centre of the sheet after nucleation from both surfaces, and the texture sharpening shown as secondary recrystallization proceeds, cf. Figs. 4(d), 4(e) and 4(f) are well-known.

Inspection of the grain sizes shown in the cross-sections in Fig. 2 show that they are the same through the thickness of the sheet, which is interesting because of the texture gradients revealed by the OIM. This surprising result indicates that nucleation, growth and any grain growth which occurred at the latter stages of the final low temperature primary recrystallization process produced identical results even though the crystallographic relationships and dislocation storage in the different layers were probably dissimilar. It might also mean that solute and precipitation effects were far more important than is usually considered in determining the final state. Whatever the truth of this matter, it is plain that Goss grains are not larger in either the central or secondaries grown parallel to the surface in a pancaked and interior layers respectively, as shown in Fig. 2.

Analysis of the grain sizes shown in the cross-sections in Fig. 2 show that they are the same through the thickness of the sheet, which is interesting because of the texture gradients revealed by the OIM. This surprising result indicates that nucleation, growth and any grain growth which occurred at the latter stages of the final low temperature primary recrystallization process produced identical results even though the crystallographic relationships and dislocation storage in the different layers were probably dissimilar. It might also mean that solute and precipitation effects were far more important than is usually considered in determining the final state. Whatever the truth of this matter, it is plain that Goss grains are not larger in either the central or

The 80 µm foil experiments show clearly that when primary Goss grains are located in the subsurface foil, they grow preferentially to a large size, but when they are located in the central layer, certain grains grow but with orientations belonging to the original γ fibre texture spread, cf. Figs. 6(c) and 6(e) and certainly Goss is not favoured. It is thus plain that the growth selection for Goss grains occurs when they are located in η material, but not when they are located in the γ material, and growth restricted to this lamellar volume determines the pancake structure observed in Fig. 4. Texture sharpening is associated with the stage when growth produces through-thickness grains, as shown by comparing Figs. 4(c) and 4(f) and noting that in the 80 µm subsurface foil that the texture remains a Goss centered spread around (100)//RD, Fig. 6(c).

The role of precipitates is considered to be of vital importance because precipitates of MnS pin the migrating grain boundaries at the end of primary recrystallization. The size of these precipitates will be a function of the energy of the grain boundary on which they are located. Thus, it is to be expected that lower energy boundaries have smaller particles, and as these dissolve these boundaries are freed to migrate. It is likely that Σ5 and Σ13a relationships are frequent in the η fibre, for these CSLs to share a common (100), which precisely describes the η fibre layer. Some of these boundary types will be low energy. The more conventional view that Goss grains have a common Σ9 boundary is likely to be true in the γ fibre material, but Goss does not develop preferentially in this layer in this experiment. It is therefore to be expected that the shared (100) direction between the η fibre material, and the Σ5 and Σ13a CSLs, where these are of low energy, could be crucial in the production of the secondary recrystallized Goss texture. However careful measurement of such areas, as are shown in Figs. 2(c) and 2(d), allowed these special misorientations to be detected, and the results are shown in Fig. 12. There is a peak for Goss grains which subsequently consumed their neighbours between 30° and 40° misorientation which could be due to Σ5 relationships but attempts to prove that they were, in fact Σ5, were not very successful. Thus the results, while encouraging, are not entirely
convincing. It must be recognized, however that the results are from a 2D section, there might well be a significant bounding of Goss grains with these special orientation relations in 3D. Thus, from these results, the best that can be said about the $\Sigma$ theory is that it is a contributory factor in the formation of the Goss texture. A further point can be made, and that is that $\Sigma\beta$ boundaries, of which there must be many more in the $\gamma$ layer than in the $\eta$ layer, are not essential for the formation of Goss secondaries, otherwise Goss secondaries would form preferentially in the central layer.

The high energy (HE) theory is not considered in detail because of space limitations but is thought to be incapable of explaining our results because Goss grains in this material are located in both the $\eta$ and $\gamma$ fibre material. Since both textures are equally sharp, it is to be expected that the frequency of high angle boundaries would be roughly similar in both cases. The fact that Goss secondaries did not form in the central layer indicates that this theory, on its own, is also not capable of explaining the results shown. Only a special subset, located in the $\eta$ fibre material, could contribute to the formation of Goss texture, but there is no evidence for this.

In modern literature the role of Si is not mentioned, even though it must be important because ultra low carbon and IF steels do not produce intense Goss textures when they go through a process which produces secondary recrystallization, and MnS is usually present in such material, to mitigate problems caused by S segregation. We begin by noting that high angle boundaries can be distinguished in alloys by the degree of solute enrichment, Hondros et al.\cite{18} Two broad categories have been found, i.e., general high angle boundaries with relatively high values of enrichment, and a few special grain boundaries distinguished by low enrichment. To characterize special grain boundaries, various bulk geometrical parameters are used, the most widely being the coincidence site lattice (CSL) description, $\Sigma$. It must be noted that Sutton and Balluffi\cite{19} in their overview could find no correlation between a geometrical descrip-

![Fig. 12. Misorientation distribution between grains in areas where secondary growth of primary recrystallized grains were tracked during subsequent heating (at 1000°C in a vacuum furnace) in a 30 µm thin subsurface foil.](image_url)
between neighbouring primary recrystallization grains was not very successful. Thus any theory of the Goss formation which depends on CSL relationships remains problematic.

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