GRAIN BOUNDARY CHARACTER DISTRIBUTION IN MAGNETICALLY ANNEALED ELECTRICAL STEEL

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Abstract. *Magnetic annealing at 800°C for 10 minutes and at five different magnitudes of field was conducted with the purpose of investigating how magnetic field applied during primary annealing affects grain boundary misorientation and CSL of GNO Fe-0.75%Si steel samples. In order to evaluate the effect of the field, ordinary annealing was conducted in a second set of specimens, under the same conditions. High magnetic fields have shown to increase the volume fraction of special boundaries and high mobility Goss-grains.*

Keywords*: Grain boundary, Magnetic Annealing, Electrical Steel*

1. INTRODUCTION

Studies have reveled that grain boundaries are not structureless but have a wide variety of atomic structures which generate structure-dependent properties and therefore are known to affect mechanical, physical and chemical properties. Five macroscopic parameters are needed to completely describe a grain boundary: three terms for the orientation relationship between two adjacent grains, such as the three Euler angles (ψ, θ, ϕ), and two terms for the spatial orientation of the grain boundary plane normal **n** with respect to one of the adjacent crystals. The minimum rotation angle required to bring two lattices into coincidence is called misorienattion angle. Misorientation is similar to orientation, but instead of bringing the crystal lattice into coincidence with the sample axes, it brings the crystal lattice of one grain into coincidence with another grain. For any two crystal lattices of different orientations there exists an axis common to both crystal lattices. Grain boundary misorientation angle has been associated to high or low grain boundary mobility (Doherty, 1997). Cotrell, 1953, noted that the mobility of grain boundary and its velocity under unit drive pressure were very much lower for sub-grain boundaries with a low-angle of misorientation than for high-angle grain boundaries. As a result of this mobility difference, only sub-grains that are highly misoriented, typically by more than 15°, can grow quickly and become recrystallized grains. Viswanathan and Bauer (1973) found that the relative boundary mobility in Cu increases as the misorientation angle increases from 10 to 20° and then becomes constant for higher misorientation angles. One of the theories involving the growth of Goss grains in Fe-Si (Hayakawa and Szpunar, 1997, Rajmohan *et. al*, 1999) is based on the assumption that boundaries with misorientation angle between 20 and 45° have high energy and are therefore more mobile than boundaries with misorientation angle lower than 20° or higher than 45°. The categorization of grain boundary misorientations as angle/axis pairs is often supplemented by further classification according to the coincidence site lattice (CSL) model, especially for cubic materials (Randle and Engler, 2000). The existence of a CSL indicates that the pattern formed by lattice points of both crystal lattices is periodic with the periodicity of the CSL (Grimmer, 1974). The parameter Σ is described as the reciprocal density of coinciding sites or in other words, Σ5, for example, is a relationship equivalent to a coincidence of 1 in 5 lattice sites. The population of grain boundaries is divided into 3 categories: low-angle boundaries (Σ1), special boundaries, (Σ3-29) and random boundaries, which can be assigned Σ values larger than 29. Boundaries associated with CSL parameters lower than Σ29, are of interest from the data processing point of view (Shvindlerman.and Straumal, 1985, Sutton and. Balluffi, 1987, Randle *et al.*, 1996). A qualitative explanation of the behavior of special boundaries is based on the differences in impurity segregation at different types of boundaries. Because coincident-site special boundaries are "good-fit" boundaries, they should accommodate fewer solute atoms than other "bad-fit" or random boundaries, reducing the solute drag effect. According to some authors (Abbruzzese *et al.*, 1992, Gangli and Szpunar, 1994, Ushigami *et al.*, 2002) CSL boundaries are responsible for the growth of Goss grains in Fe-Si steels. Grain-non-oriented electrical steel has its main technological application in rotating machines, where isotropy of magnetic properties on the plane of the sheet is required. Magnetic annealing is a term regularly used to indicate the application of magnetic field while a material is being annealed. It has been noticed that magnetic annealing affects microstructure evolution in ferrous as well as in non-ferrous alloys (Watanabe *et al.*, 1990, Sheikh-Ali *et al.* 2002, Bacaltchuk *et al.*, 2005).

2. EXPERIMENTAL PROCEDURE

Table 1 presents the chemical composition of the grain-non-oriented (GNO) electrical steel used in this research. The samples were hot and cold rolled at ACESITA- Brazil. Coupon specimens 5 mm wide, 8 mm long and 0.5 mm thick were sampled from the *"as received"* cold rolled sheet with their longitudinal axis parallel to the rolling direction of the sheet.

Table 1. Chemical composition

Material	%Si	%C	%Mn	%S	%N	%Al
GNO Fe-Si	0.75	0.003	0.50	0.002	0.003	0.002

The annealing treatments were performed at 800°C, for 10 minutes using a 95% argon-5% hydrogen inert atmosphere. Five different magnitudes of field were used during the annealing, 3, 5, 10, 15 and 19Tesla. The magnetic annealing was carried out in a cylindrical furnace inserted into a 20Tesla resistive magnet. An alumina sample holder, containing the samples, was inserted inside the furnace and placed at the center of the magnetic field with the silicon steel samples positioned with their rolling direction (RD) parallel to the direction of the field (H). In order to evaluate the effect of the field, ordinary annealing was conducted in a second set of specimens, with the same temperature, time and atmosphere used during magnetic annealing. All OIM measurements were made using an ESEM Model Geol E3, operating at 20kV. The electron gun source was LaB6 with a resolution of about 4 nm. The vapor pressure in the ESEM chamber was maintained during the measurements at about 0.2T (~ 267 bar). Table 2 shows the nomenclature of the samples.

Table 2. Nomenclature of the samples

Sample	Magnetic Annealing Field (Tesla)
O810	0
03M8	3
05M8	5
10M8	10
15M8	15
19M8	19

3. RESULTS

According to the graphics and the numerical data, in Tab. 3, not much change in the frequency of low, middle and high-misoriented grain boundaries was observed increasing magnitude of field as well as between the magnetically annealed samples and the sample annealed without field (O810). Small variations (2 to 3%) in the frequency of grain boundary misorientation from one sample to another did not have a trend, what turned difficult to correlate it with the amount of applied magnetic field.

After annealing, the percentage of low misoriented Goss grains did not vary significantly up to 5T when it started decreasing as the field increased in magnitude. The percentage of this type of boundary in Goss-grains was higher than the percentage shown by the grains with orientations different from the Goss ("other grains"), principally up to 10T. From 0 to 10T, the frequency of high-energy Goss-grain boundaries did not vary significantly with field and was equivalent to the frequency shown by the other grains. Afterwards, as the field increased to 15 and 19T the frequency of theses boundaries started increasing reaching 58.6% after 19T annealing.

Table 3. Grain boundary misorientation after annealing without and with magnetic field. Values of boundary misorientation for the entire set of measured grains ("all grains") and for the Goss-oriented grains.

	Grain misorientation (all grains) - %			Grain misorientation (Goss grains) - %		
Sample	< 20°	20° - 45°	> 45°	< 20°	20° - 45°	> 45°
O810	19.5	48.9	31.6	34.1	49.6	16.3
03M8	19.2	50.8	30	36.1	52.3	11.6
05M8	21.6	50.3	28.1	37.6	49.1	13.3
10M8	18.7	49.4	31.9	31.8	50.9	17.3
15M8	19.9	48.7	31.4	25.2	56.2	18.6
19M8	18.3	50.2	31.5	23.6	58.6	17.8

According to Tab. 4, the percentage of special boundaries, Σ3-29, slightly increased with increasing in the magnitude of magnetic field and the samples annealed at 3 and 5T were the ones that showed percentage of special boundaries closest to the ordinarily annealed sample. The percentage of low-angle boundaries was the same for the sample annealed without field and the sample annealed at 3T. The fraction of these types of boundaries changed about ± 1% among the samples, without any trend as the magnitude of the field was increased.

Table 4. Distribution of grain boundary structure after annealing without and with magnetic field.

Sample	Low-angle (%)	CSL ($\Sigma = 3 - 29$) (%)	Random (%)
O810	12.2 (5.3)	11.2 (0.99)	76.6
03M8	12.2 (5.3)	10.5 (0.93)	77.3
05M8	13.4 (5.8)	11.0 (0.97)	75.6
10M8	12.0 (5.2)	12.2 (1.08)	75.8
15M8	11.1 (4.8)	12.6 (1.11)	76.3
19M8	11.4 (4.9)	13.1 (1.16)	75.5

Table 5 presents the frequency of the three categories of boundaries for the recrystalized Goss-oriented-grains. For the magnetically annealed samples, the percentage of low-angle boundaries increased with field up to 10T, reaching a maximum of 26.5%, which wais higher than the percentage found for the sample annealed without field. After annealing at higher magnitudes of field, the percentage of low-angle boundaries decreased reaching 18.8% after annealing at 19T. The opposite trend was observed with respect to special boundaries where the percentage of CSLs decreased as the magnitude of field increased up to 10T. As the field continued to be raised, the percentage of these boundaries started increasing up to a maximum of 12.5% (sample 19M8). The lowest amount of special boundaries, 5,4%, was found in sample O810. This percentage corresponded to more than half of the amount found in sample 19M8.

Table 5. Distribution of Goss-grain boundary structure of samples annealed without and with magnetic field.

Sample	Low-angle (%)	CSL ($\Sigma = 3 - 29$) (%)	Random (%)
O810	24,1	5,4	70,5
03M8	21.4	9.3	69.3
05M8	25.4	9.0	65.6
10M8	26.5	8.3	65.2
15M8	19.6	10.5	69.9
19M8	18.8	12.5	68.7

Results of average grain diameter and grain size distribution for all grains in the matrix and particularly for the Goss-grains are being shown below. Grains that have diameter lower than 21μm will be called small, grains with diameter between 21 and 42μm will be called medium and finally, grains with diameter higher than 42μm will be named large grains.

According to the results (Fig. 1 and Tab. 6), most part of the recrystallized microstructure was formed by medium grains, regardless the application of field. The average grain diameter of sample O810 was 34.9μ and almost half of its grain population was formed by medium grains with only a small amount of grains below 21μm. As the 3T magnetic field was applied, the amount of small grains increased but the medium grains were still the majority inside the microstructure. Magnetic annealing at 5T and above affected grain size by decreasing the percentage of small grains and increasing the percentage of medium and, principally, large grains.

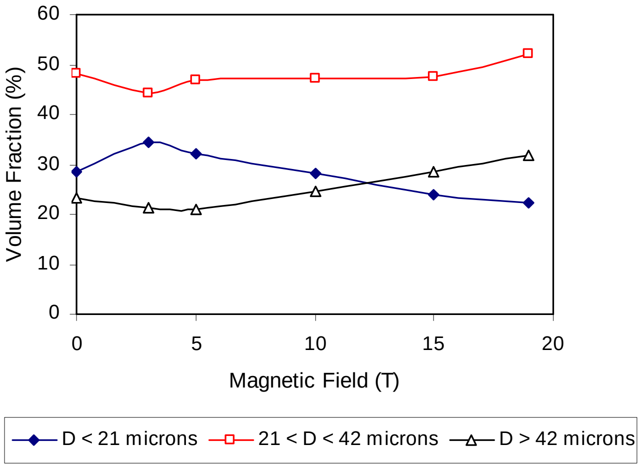

Figure 1. Percentage of small (D < 21μ), medium (21μ < D <42μ) and large (D > 42μ) grains after annealing at 800°C without and with field for 10 minutes.

According to Tab. 6, among all annealed samples, only the one annealed without field had the average Goss-grain size smaller than the average size of the other measured grains. For the magnetically annealed samples, the average Goss-grain diameter increased progressively with magnitude of field and was a little higher than the diameter of the other grains. For the general set of grains, the average grain size of samples 03M8, 05M8 and 10M8 was very close to the grain size of the sample O810. After annealing at 15T and 19T, despite the presence of a few very large grains the average grain size of these samples did not increase significantly in comparison to the other samples annealed at lower fields.

Table 6. Values average grain diameter for the entire grain population (all grains) and for the Goss-oriented grains for the samples annealed without and with magnetic fields

	All grains	Goss grains
Sample	Average grain size (μm)	Average grain size (μm)
O810	34.9	30.1
03M8	33.3	34
05M8	34.1	35.1
10M8	34.3	36.2
15M8	39.3	42.4
19M8	40.3	43.6

Figure 2 shows the effect of the magnitude of magnetic field on grain size distribution of Goss-oriented grains. The percentage of small grains increased when the 3T field was applied and started decreasing as the field was raised until reaching 27.8% at 19T. The fraction of medium grains varied from 34.4%, at sample O810, to 45.5% at sample 03M8 and after that, as the magnetic field was raised, no significant variation in the percentage of these grains was observed. The large grains seemed to be the group of grains that were more affected by the variation in magnitude of field. A considerable drop from 24.3% to 9% in the percentage of these grains was observed as the field was applied. This percentage remained almost constant until 10T when the amount of large grains started increasing with field.

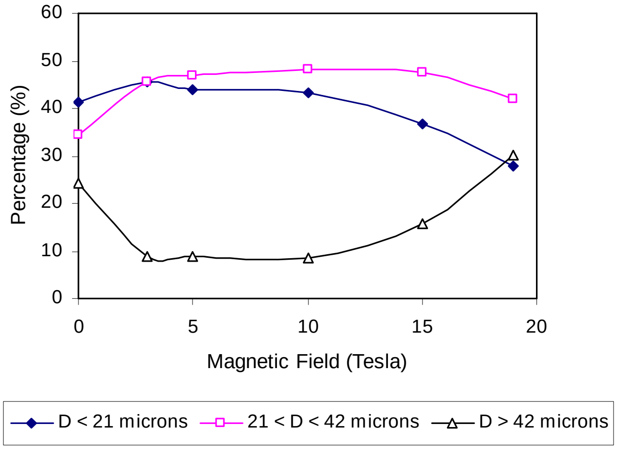

Figure 2. Percentage of small (D < 21μm), medium (21μm < D <42μm) and large (D > 42μm) Goss-oriented grains after annealing at 800°C without and with field for 10 minutes.

4. DISCUSSION

During annealing the percentages of low, middle and high misoriented boundaries for the whole set of grains (all grains) practically did not vary from sample to sample. High magnetic fields (15 and 19T) seemed to have affected the grain boundary misorientation of Goss-grains by decreasing the amount of grains with misorientation angle lower than 20° and by increasing the amount of high mobility grains. It can be considered a positive result since electrical steels with elevated percentage of high energy Goss-grains after primary recrystallization tend to develop a strong Goss texture after the secondary annealing of these materials (Hayakawa and Szpunar, 1997).

According to the results of grain-boundary structure (Tab. 4) for the general set of grains, not much difference was observed among the samples annealed with field and without field. The change in the percentage of the three types of boundaries was very small when compared to the relative increase in the magnitude of applied field.

The percentage of Goss-grains with low angle boundaries in the samples annealed up to 10T was similar to the ordinarily annealed sample. At higher fields (>10T), magnetic annealing started decreasing the amount of these type of boundaries. The rising in the percentage of special boundaries in Goss-grains, on the other hand, could be seem since the lowest applied field. Magnetic field had no effect on random boundaries. Some authors have shown the importance of special boundaries on the development of strong final Goss-texture (Harase, 1992, Gangli, and Szpunar, 1994).

The values of average grain size of the samples magnetically annealed up to 10T did not differ from the average grain size value of the sample annealed without field (O810). As the magnetic field increased to 15 and 19T, an increase was observed but the grain size difference between these two samples and sample O810 was not higher than 15%. As far as grain size distribution is concerned, increasing the magnitude of magnetic field results in a decrease in the percentage of small grains and an increase in the percentage of medium and mainly large grains. From these results it can be observed that the field driving force might have compensated a possible retardation effect caused by the application of magnetic field. It could be noticed since low magnetic fields (3 to 5T) but only high magnetic fields were able to affected average grain size by increasing the amount of medium and large grains in the matrix. The distribution of Goss grains was not so different from the distribution of “all grains”. The number of small Goss grains increased after application of the 3T magnetic field and after that decreased monotonically as the field was raised. The percentage of medium grains increased after application of field remaining about constant until 19T when this percentage slightly decreased. The amount of Goss-large grains decreased significantly with the field and only after 10T the percentage of large Goss- grains started increasing again, reaching a maximum of 30% at 19T. The amount of Goss-grains and grains with orientations different than that was evaluated and the ratio between them was not affected by the magnitude of field, indicating that magnetic annealing did not collaborate for the nucleation of Goss-grains.

5. CONCLUSION

The effect of magnitude of magnetic field was more pronounced in the Goss grains. High magnetic fields did not show to be very effective to increase the volume fraction of Goss grains but it has shown to be able to affect their grain boundary structure by: a) generating more Goss-grains with special boundaries (CSL boundaries) and b) decreasing the volume fraction of grains with misorientation angle lower than 20° and increasing the volume fraction of high mobility grains (misorientation between 20° and 45°).

High magnetic fields (>5T) decreased the percentage of small grains and increased the percentage of medium and, principally, large grains.

6. ACKNOWLEDGEMENTS

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