EBSD ANALYSIS ON MAGNETICALLY ANNEALED ELECTRICAL STEEL

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Abstract. *It has been noticed that the controlling factors of microstructure formation during annealing are different from those during conventional annealing. The objective of this work is to investigate the effect of magnetic field on grain size and grain boundary structure. Magnetic annealing at 17 Tesla was applied on Fe-3.25%Si samples, for 10 minutes at 800°C. Annealing without magnetic field was also carried out and the recrystallized microstructures, obtained after both types of annealing, were analyzed by means of optical microscopy, scanning electron microscope and electron back scattering diffraction. According to the results, although magnetic field may have caused retardation during recrystallization, it has also promoted grain growth of (001)[001] grains and has increased the fraction of high energy boundaries and the fraction of Σ3 and Σ5 types of boundaries.*

Keywords: *Electrical Steel, Magnetic Annealing, Texture*

1. INTRODUCTION

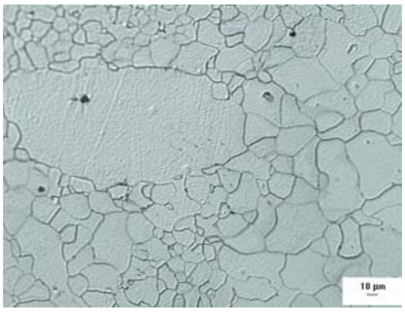
Soft magnetic materials cover a huge market of various products – about 7 x 10^6 tons- annually (Moses, 2003). Among the groups of soft magnetic materials, electrical steel occupies almost 80% of the market, where the grain non-oriented electrical steel occupies more than 50% of the annual value of world production of soft magnetic materials (Tumaski, 2010). Grain non-oriented (GNO) electrical steel has its main application in lamp reactors, power meters and in small and large electric motors where high permeability, low coersivity and isotropy of magnetic properties are required. Texture and grain size are among the main factors affecting magnetic properties and the control of these two microstructural parameters is crucial. Magnetic losses, which have a direct effect on the performance of electrical machines, are strongly affected by final grain size (Landgraf *et al.*, 1999). When the grain diameter *d* increases, the hysteresis loss decreases in proportion to 1/*d*. The total core loss is minimized at an optimum grain diameter in the range of 150 and 200μm (Stephenson and Marder, 1986). Grain boundaries are important elements of the microstructure of most engineering metallic and ceramic materials. The orientation relationship between two neighboring grains is a primary factor controlling their properties, for example, intergranular brittleness (Watanabe and Tsurekawa, 1999), diffusivity, energy and mobility (Lee and Szpunar, 1995 and Randle *et al*, 2001). Grain boundary misorientation angle has been associated to high or low grain boundary mobility (Doherty, 1997). The growth of Goss grains in electrical steel has been associated to grain boundary misorientation angles between 20 and 45°. According to evidences (Rajmohan *et al*, 1999) grains having these misorientation angles have high energy and are therefore more mobile than grains with misorientation angle lower than 20° and higher than 45°. Another structural classification frequently used in grain boundary characterization is the coincidence site lattice (CSL). This geometrical model consists of lattice sites that correspond to both mutually misoriented and interpenetrating lattices of adjacent grains (Lejcek *et al*, 2003). Boundaries associated with low values of CSL (i.e. high degree of coincidence between two grains), generally < Σ29, are of interest from the data processing point of view (Randle *et al*, 1996). According to some authors (Ushigami *et al*., 2002), CSL boundaries are responsible for the growth of Goss grains in electrical steels. The purpose of this work is to investigate how magnetic field applied during primary annealing affects grain size and grain boundary structure of GNO electrical steel.

2. EXPERIMENTAL PROCEDURE

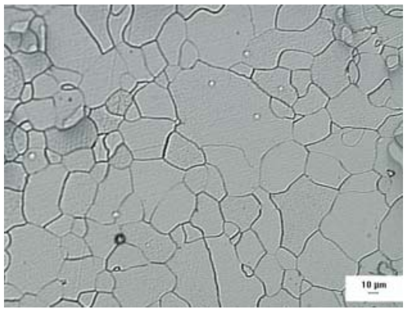
The starting material was a Fe-3.25%Si hot and cold rolled at ACESITA-Brasil, with chemical composition (wt.%): 3.25%Si, 0.003%C, 0.30%Mn, 0.001%S, 0.002%N and 0.60%Al. Specimens measuring 5 x 8 x 0.5mm were sampled from the cold rolled sheet and annealed at 800°C, for 10 minutes inside a 17 Tesla magnetic field and outside magnetic field. The sample annealed inside the field is named HM810 and the one annealed outside the field is the HO810. Magnetic annealing was carried out in a cylindrical furnace inserted into the 195mm bore of a 20Tesla resistive magnet. An alumina sample holder was placed inside the furnace at the center of the magnetic field with the electrical steel samples positioned with their rolling direction (RD) parallel to the direction of the field (H). In order to avoid oxidation a mixture of 95% argon and 5% hydrogen was used as an inert atmosphere. Annealing without magnetic field was performed under the same conditions (time, temperature, and atmosphere) as for the magnetic annealing. After annealing the specimens were mechanically polished until reaching their semi-thickness. The annealed microstructure was characterized by optical microscope and by scanning electron microscope. Grain boundary characterizations by SEM were carried out on a Jeol "JSM-5800LS" scanning microscope. OIM Data Collection and OIM Analysis software were used for measurements and data conversion, respectively.

3. RESULTS AND DISCUSSION

After proper polishing and etching, the samples were taken to the optical microscope for microstructural analysis and two representative micrographs of samples HM810 and HO810 are shown in Fig. 1.



(a)



(b)

Figure 1. Optical micrography after annealing (a) inside and (b) outside magnetic field. Magnification 200X

According to it, the microstructures were completely recrystallized after 10 minutes of annealing inside as well as outside magnetic field. The average grain size of the samples was evaluated using the linear intercept method. And the results are showed in Tab. 1.

Table 1. Average grain size for samples annealed with and without field.

Sample	Grain Size (microns)
HM810	56
HO810	60

Figures 2 and 3 show the area fraction *versus* grain size distribution for samples HM810 and HO810, respectively. The area fraction of grains having two different crystal directions, belonging to the eta fiber; the (001) [001] (cube component) and the (110) [001] (Goss component); are being shown in these figures: These are two very important texture components for soft magnetic materials, as it is the case of electrical steel.

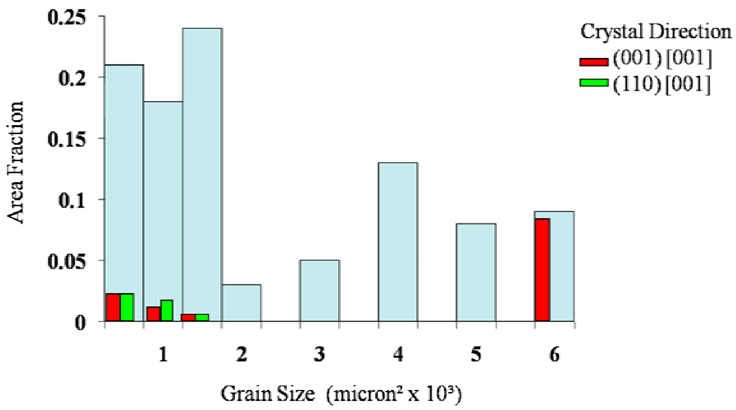


Figure 2. Grain size distribution after annealing inside magnetic field - sample HM810

The microstructure of the sample annealed inside filed is formed by smaller grains and retardation during nucleation, due to the application of magnetic field, could be the reason for that. Annealing microstructure with grains having crystal orientation (001) [001] is essential for electrical steel from the magnet efficiency point of view, since (001) is the easiest magnetization direction. According to Fig. 2 and 3, grains having (001) [001] orientation grew larger after magnetic annealing showing a possible effect of magnetic field on grains having certain orientations. This effect can be explained by the difference in magnetic free-energy generated by the field on the <100> nuclei (Martikainen and Lindroos, 1981).

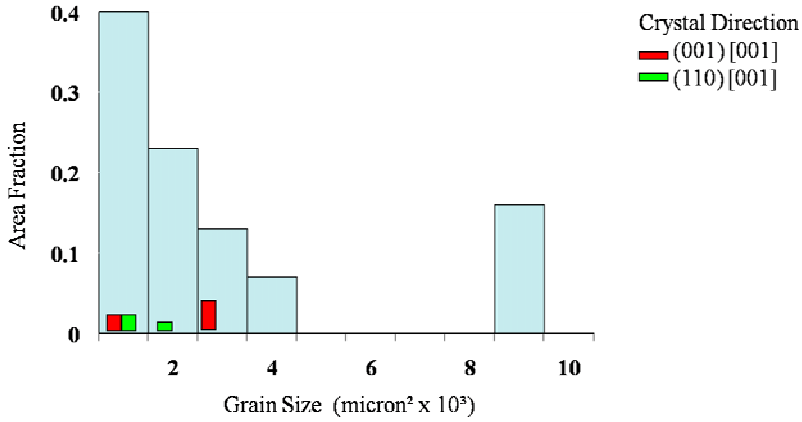


Figure 3. Grain size distribution after annealing outside magnetic field - sample HO810

The high energy boundaries theory (Rajmohan and. Szpunar, 2001) assumes that boundaries with misorientation between 20° and 45° are high energy boundaries and would migrate faster than the low (< 20°) and high (> 45°) angle boundaries. Figure 4 presents results of grain boundary misorientation angle, ranging from 20° to 80°, for the annealed samples. Sample HM810, annealed inside magnetic field, have shown a larger fraction of grains having high energy boundaries. The final grain size is a consequence of mobility of grain boundary, which is directly related to the energy of the boundary. The higher the fraction of high energy boundaries, the higher the probability of the microstructure to be formed by larger grains after secondary annealing.

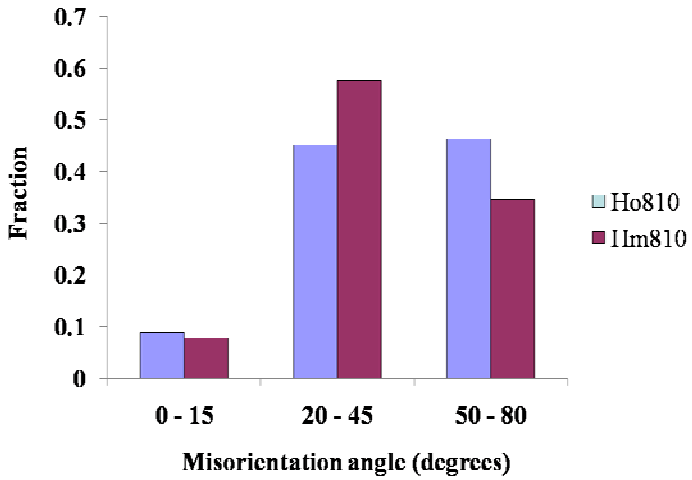


Figure 4. Misorientation angle distribution for samples HM810 e HO810

Coincidence site lattice boundaries namely Σ5 (Gangli and Szpunar, 1994)], Σ7 (Harase: 1992), Σ9 (Ushigami *et al*., 2002) are responsible for the growth of Goss grains. Lin *et al*, (1996) suggests that Σ3-9 boundaries collectively are responsible for the abnormal grain growth of Goss grains during secondary annealing. According to Fig. 5, the amount of Σ3 - 9 boundaries formed was the same regardless the presence of field during annealing. The Σ3 and Σ5 types of boundaries, however, seem to have been favored by magnetic field. Zhang *et al*., (2005) have found a similar result in a magnetically annealed medium plan carbon. In his work, Zhang has correlated the larger Σ3 boundary areas with larger grain sizes in the field-treated samples showing that the field applied has some effect on grain growth. Lee and Szpunar (1995) investigating a Fe-3%Si observed that Goss grains having higher amount of Σ5 CSL boundaries before secondary recrystallization grew faster than other grains.

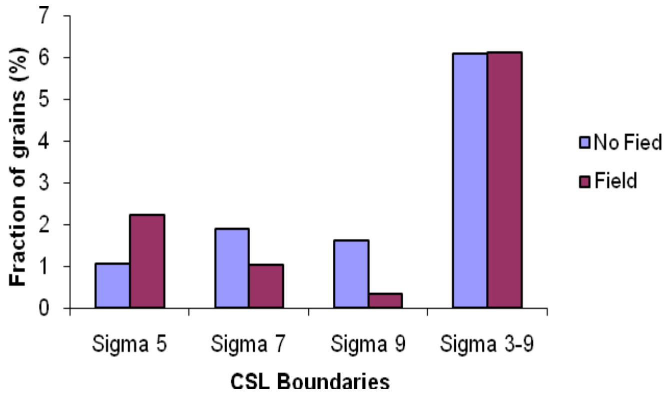


Figure 5. Coincidence Site Lattice for samples annealed without field and with field

3. CONCLUSIONS

According to the results, the microstructures generated after primary annealing inside and outside magnetic field were completely recrystallized and formed by small, medium and large grains. The sample annealed inside field showed smaller grains when compared to the other sample. The applied field could be the reason for a possible retardation during the nucleation and recrystallization processes leading to a difference in grain size between samples HM810 and HO810. Nevertheless, that very same magnetic field seems to have also promoted grain growth of (001) [001] grains. Other effects of magnetic field were the increase in the fraction of high energy boundaries and in the fraction of Σ5 and Σ3 types of boundaries, where the former is known to have a significant effect on abnormal Goss grain growth during final stage (secondary) annealing.

4. ACKNOWLEDGEMENTS

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