Correlation of grain growth phenomena with magnetic properties in non – oriented electrical steels

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Abstract. This paper presents a combination of two types of method targeted to investigate the stages of the microstructure evolution in annealed non-oriented electrical steels by means of magnetic measurements and metallographic analysis. The indirect magnetic testing, carried out by Barkhausen noise was associated with the direct structural investigation by Scanning Electron Microscopy measurements. The goal of this work was to study the influence of heat transport phenomena on grain growth processes in non-oriented electrical steels, which were subjected to different annealing conditions. The results determined from the magnetic measurements and predicted from micrograph observations show a relatively good concordance.

1. Introduction
Non – Oriented Electrical Steels (NOES) are soft polycrystalline magnetic materials and are so called because it contains a grain structure with no preferred orientation [1-10]. For this reason they have found extensive applications in domestic appliances that require isotropic magnetic properties in the plane of the sheet [3-6].

The variety of NOES in general depends on the chemical content of Si. Silicon is a ferrite stabilizer, which increase the resistivity and decreases hysteresis loss [4-6, 10]. In order to promote the favourable texture development of (100) plane, which is favourable for magnetization, it is important to control the final microstructure of NOES, in terms of grain size and crystallographic texture [6, 11-12]. Additionally, the rolling in the ferrite region below the austenitization temperature is required [6, 11-12]. Therefore, NOES magnetic properties are controlled by the microstructure.

For this reason, NOES are subjected to various thermally activated process, which initiates recrystallization and grain growth phenomena. During annealing the grain size rapidly increases, because of the large amount of stored energy in the grains, released at elevated temperatures [10-12]. However, the non-linear migration of the grain boundaries and the abnormal growth of certain types of grains at the expense of others, resulting in the final microstructural inhomogeneity, which deteriorates the final magnetic properties of the NOES [4-6, 10-12]. Thus, grain growth kinetics significantly affects the evolution of the texture and, consequently, the final magnetic properties [6].

It is well known that magnetic methods are influenced by the microstructural features of the examined ferromagnetic materials [13-34]. As a result, the annealing influence the final microstructure of the material, resulting in variations of the output magnetic signals. Although, the non-destructive
testing using various methods has been of great interest in recent years [35-46], a quantitative or qualitative evaluation on how the microstructure affect the magnetic properties has not predicted yet.

Hence, the present paper presents a combination of two types of method targeted to investigate the stages of the microstructure evolution in annealed non-oriented electrical steels by means of magnetic measurements and metallographic analysis. The indirect magnetic testing, carried out by Barkhausen noise was associated with the direct structural investigation by Scanning Electron Microscopy measurements. The goal of this work was to study the influence of heat transport phenomena on grain growth processes in non-oriented electrical steels, which were subjected to different annealing conditions.

2. Experimental procedure
Commercial samples of NOES sheets were investigated. The cold rolled specimens were subjected to annealing under laboratory conditions. Before the heat treatment each specimen was cut into a strip of 10 cm$^2 \times 3$ cm$^w$ with the longest side parallel to the rolling direction. The surface of these specimens was cleaned, degreased and dried. Each specimen was heated to a different temperature within the range of 350 °C – 950 °C at a heating rate of at least 10 °C/s. The specific temperature selected was kept constant for 12 min (Table 1).

| Annealing temperature (°C) | Holding time (min) | Cooling conditions |
|---------------------------|--------------------|--------------------|
| NOES 1                    | 350                | 12                 | Air cooled         |
| NOES 2                    | 550                | 12                 | Air cooled         |
| NOES 3                    | 600                | 12                 | Air cooled         |
| NOES 4                    | 700                | 12                 | Air cooled         |
| NOES 5                    | 800                | 12                 | Air cooled         |
| NOES 6                    | 920                | 12                 | Air cooled         |
| NOES 7                    | 950                | 12                 | Air cooled         |

The microstructure of the specimens were examined along the longitudinal cross-section using Scanning Electron Microscopy (SEM). The samples were polished and subsequently etched using a 2% Nital solution for 10-30s until the grain boundaries were revealed. The JEOL JSM 6100 electron microscope was connected with a Noran TS 5500 Microanalyzer for EDS analysis on cross – sections of steel sheets.

Magnetic Barkhausen noise (MBN) was measured using the MEB-2C device. A triangular shaped magnetic field produced by a generator was applied on the surface of the sample by an electromagnet in the shape of a probe. The discrete changes in the resulting local magnetization (MBN) were sensed at the ends of a receiving coil as voltage pulses. All MBN measurements were taken at a constant threshold at different positions of the annealed sample. The rms value of MBN measurements were recorded.

3. Results
All the annealed NOES samples presented a Body Centered Cubic (BCC) ferritic matrix which is characterized by micro-segregation of silicon, strongly in grain boundaries (figure 1). The commencement of abnormal grain growth is represented in figure 1e. At this point, the abnormally growing grain seemed to consume the primary recrystallized grain matrix (figure 1d). The microstructure obtained after heat treatment at 920 °C for 12 min represented a totally abnormal grain growth (figure 1g). Hence, the abnormal grain growth can be obtained during annealing in a very narrow temperature range, within very short time frames. The optimum set of conditions for the complete development of abnormal grain growth in the investigated NOES samples can be annealing at 950 °C for 12min and an extremely high, for this grade of steel, heating rate of 10 °C/s.
It has to be noted, that the microstructure observed by the SEM did not provide all the information required concerning recovery the process during recrystallization, for example the decrement in the dislocations density.

Figure 2 summarizes the average grain size distribution in the investigated NOES samples. It is obvious that last process initiated a comparatively fast and abnormal grain growth mechanism. The MBN results were plotted in figure 3 as a function of the annealing temperature. It was evident, the decrement of the magnetic response, as the annealing temperature is increased. Because the ferrite grains became coarser, as the annealing temperature is increasing, the MBN values of the investigated specimens was decreasing. Grain boundaries act as strong pinning centers for the magnetic measurements. As a result, finer samples have higher magnetic output signal. In a region of annealing temperatures between 550°C and 650°C a slight increment of the magnetic signal was observed, as a result of the strong microsegregation of Si in grain boundaries.

Figure 1. Microstructure developed in the (a) as received sample and after annealing at (b) 350 °C, (c) 550 °C, (d) 600 °C, (e) 700 °C, (f) 800 °C, (g) 920 °C and (h) 950 °C for 12min.

Figure 2. Average grain size of the as received and annealed samples.

Figure 3. Variations of the MBN rms values at various temperatures.

4. Conclusions

A combination of metallographic analyses with magnetic measurements on the annealed NOES specimens allowed the distinguish of the microstructure evolution stages. The anisotropic behavior of the grain boundaries migration under non-equilibrium conditions, leads to a strong decrease of the MBN values.
5. References

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