Monitoring Recovery and Recrystallization in Interstitial Free (IF) Steel by Magnetic Hysteresis Loop Measurements

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Abstract. In this paper, how structure-sensitive parameters derived from the hysteresis loop, like coercive field, remanent induction, and hysteresis losses, can be used to characterise recovery and recrystallization in cold rolled Ti-Nb stabilised interstitial free ultra low carbon steel is shown. The effect on hysteresis measurements of isothermal annealing treatments applied to industrially cold rolled samples at different temperatures is related to the microstructural changes which take place during the annealing as a consequence of the metallurgical recovery and recrystallization processes. It has been observed that the remanent magnetic field, the coercive field and the hysteresis losses can be used to monitor recovery. Furthermore, recrystallization can be characterised by coercive field and hysteresis loss measurements. Additionally, the effect on magnetic parameters of hardness indentations made after the annealing is studied. It has been shown that remanent magnetic field measurements are affected by hardness indentations present in the samples, while coercive field and hysteresis losses do not change.

Introduction

Low carbon steels are usually cold rolled and subsequently annealed to soften the material, enhance its formability and obtain the desired drawing texture. During an annealing treatment the cold rolled steel substructure experiences recovery and recrystallization processes. Recovery involves the rearrangement of dislocations into low energy configurations and the annihilation of dislocations, while recrystallization implies the nucleation and growth of new defect free grains.

Nowadays several magnetic properties have been reported to be very useful to non-destructively characterise stress [1-3] and microstructure [4-7] in ferromagnetic steels. When a magnetic field is applied to a ferromagnetic steel, those domain walls whose magnetic domains present a magnetisation in the same direction as the applied field start to move, increasing their domain sizes at the expense of neighbouring domains. In an ideal material these domain walls move continuously, but in a real material there are a lot of pinning sites such as dislocations, grain boundaries, second phase precipitates and stresses that hinder this movement. Therefore the magnetic parameters are expected to be useful to characterise the above mentioned recovery and recrystallization processes.

A recent work by authors and co-workers [6] has shown that the coercive field (H_c) measurement technique can be effectively employed to monitor the kinetics of recovery and recrystallization processes occurring during the isothermal annealing of a cold rolled Ti-Nb stabilized interstitial free (IF) ultra low carbon steel. As a continuation of that work, the present study focuses on the research into the use of some other structure sensitive parameters derived from the hysteresis loops on the same IF steel. The evolution during isothermal annealing treatments, performed in a range of temperatures, of parameters such as the remanent magnetic field and the hysteresis losses is related in the following sections to the microstructural changes produced due to recovery and recrystallization processes. The capability of these parameters to non-destructively monitor these metallurgical processes is analysed.

Additionally, the effect on magnetic parameters of hardness indentations performed on the steel samples is studied.

Experimental procedure

The material used in this work is an industrially produced Ti-Nb stabilised interstitial free (IF) ultra low carbon (ULC) steel, cold rolled to a final thickness of 0.8mm through a reduction of 75% and with a composition: 0.003 C - 0.11Mn - 0.02Nb - 0.012Ti (wt-%). A series of isothermal annealing treatments were performed within the range of 350ºC to 650ºC to obtain various degrees of recovery and recrystallization. Further details on the annealing treatments can be found in [6].

The magnetisation curves were measured with a system constructed at the authors' laboratory [7]. The magnetic field is applied to the samples using a U-shape magnetic yoke with a 200 turn coil wound around it. A sinusoidal excitation field with a frequency of 1Hz and a maximum amplitude of 4100A/m (enough to reach the saturation magnetisation) generated with a 12-bit resolution signal generator was applied. This field was measured using a Hall probe placed at the surface of the sample and the magnetic induction signal was obtained by the integration of the induced voltage on a 50 turn encircling coil wound around the samples.

Magnetic measurements of the samples annealed at 350ºC, 450ºC and 550ºC were made at room temperature immediately after the samples had been removed from the annealing simulation device. Unfortunately, hardness measurements were made on the samples annealed at 650ºC before the magnetic measurements were taken. Therefore, in order to study the possible effect of the hardness indentations present on the samples on the magnetic measurements, hysteresis loop measurements were taken as usual before the hardness measurements and also after the hardness indentations on the samples annealed at 550ºC, and the comparison is presented.

The coercive field (H_c), remanent induction (B_r) and hysteresis loss (W_h) values were derived as parameters for each hysteresis loop. These measurements were calculated as the average of two complete tests. For each test four major hysteresis loops were recorded and the values obtained were averaged.

Results and discussion

Effect of hardness indentation on magnetic parameters

The influence of hardness indentations present on the steel samples was analysed in samples annealed at 550ºC for 10s, 100s and 1000s. The H_c, W_h and B_r magnetic parameters, measured before and after the hardness measurements were made, are represented in Figure 1-a, 1-b and 1-c, respectively.

It can be seen that the coercive force and hysteresis loss values do not change after the hardness indentations are made (see Figure 1-a and 1-b, respectively). This can be explained because new pinning sites, that hinder the domain wall movement, are not created as a consequence of the hardness indentations.

The internal stresses caused by hardness indentations produce a change in the magnetic anisotropy, with the consequent loss of some transverse domains. This is reflected as a change in the remanent magnetization, since variations of this parameter happen with the loss of only one of the transverse domains [1]. Figure 1-c clearly shows that hardness indentations significantly affect remanent induction measurements.

Therefore, considering these results, it was concluded that only samples corresponding to annealing treatments performed in the range of temperatures 350-550ºC could be considered for the study of the use of B_r for non-destructive characterisation of the annealing processes. Still, H_c and W_h measurements could be employed to characterise magnetically the samples annealed at 650ºC.

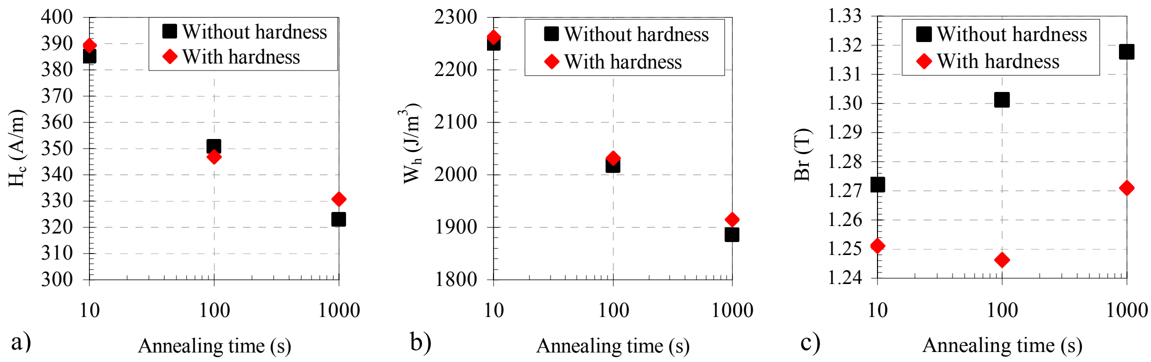

Figure 1. Magnetic parameters measured before and after the hardness measurements were made on the samples annealed at 550ºC; a) the coercive field, b) hysteresis loss, c) remanent magnetisation.

Previous observations

In a previous work by authors [6], the evolution of hardness measurements and coercive field values during the annealing of a cold rolled interstitial free steel was studied and related to the microstructural changes observed by optical metallography. Optical metallographic observations in that study showed that the initial deformed grains, elongated in the rolling direction, were still present in the samples annealed for the longest times at 350ºC, 450ºC and 550ºC, without any indication of recrystallization. The same happened for the shortest annealing time (10s) at 650ºC. These examinations showed that only recovery contributed to the variations observed on the magnetic measurements at these stages. However, for longer annealing times at 650ºC, recrystallization was observed to activate (see micrographs in Figure 2 of [6]).

On the one hand, in the same study, the evolution of H_c with the annealing time at low temperatures was directly related to the decrease of the dislocation density during recovery processes, since H_c is proportional to the square root of the dislocation density [8]. Additionally, in this IF steel, H_c measurements were found to be useful to characterise the recrystallization process, whereas in a previously studied cold rolled extra low carbon (ELC) steel the evolution of H_c did not follow the progress of recrystallization [9]. This was attributed to the additional dependence of H_c on the inverse of the grain size [10]. In the ELC steel the variation of the grain size between the initial cold rolled steel, being 60μm, and the complete recrystallization grain size, 8μm, was quite large comparing with the variation found in the IF steel of the present study, where the grain sizes were found to be 27μm and 11μm, for the cold rolling and recrystallized states, respectively [6].

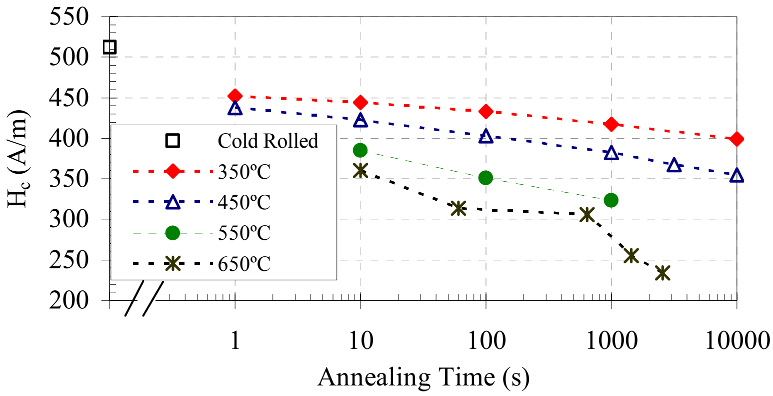

Figure 2. Evolution of the coercive field as a function of the annealing time at different annealing temperatures.

On the other hand, H_c measurements (absolute values shown in Figure 2) showed a much higher degree of resolution to monitor the recovery processes than conventional hardness measurements. The softened fractions determined from hardness in [6] are represented as a function of the annealing time at 550ºC and 650ºC in Figure 3. It can be observed that at 550ºC there are hardly any changes in hardness, while H_c gradual variations are found even at the shortest annealing times at the lowest temperature studied, 350ºC. Additionally, some differences were found between the plot of the recrystallized and the softened fraction as a function of the annealing time, which were attributed to the concurrent recovery effects with recrystallization.

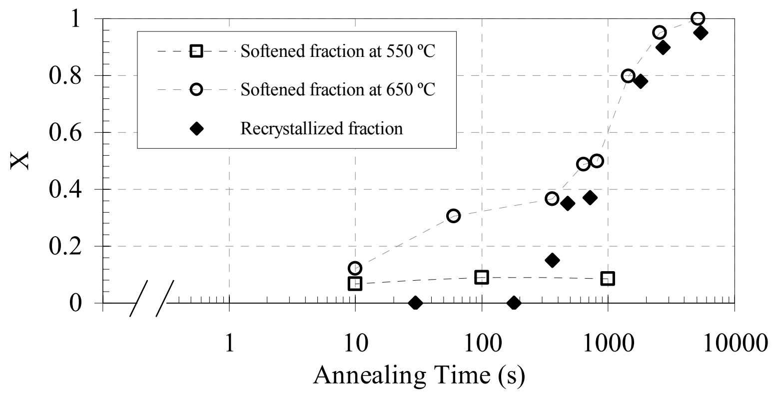

Figure 3. Evolution of the annealing time of the softened fraction (at 550ºC and 650ºC) and recrystallized fraction (at 650ºC) determined by hardness measurements and optical metallography, respectively.

Evolution of other structure sensitive parameters derived from the hysteresis loop

The effect of increasing the annealing time in the hysteresis loops is shown in Figure 4. The B-H loops are observed to become steeper with annealing, providing lower H_c, lower W_h and higher B_r values. Additionally, in Figure 5 the differential relative permeability is represented as a function of the magnetic field. The maximum differential permeability is observed to increase as the annealing progresses and it happens at smaller amplitudes of the applied field. During the annealing treatment the annihilation of the dislocations remove the internal stresses existing in the material with the consequent rotation of the magnetisation to the easy axis, which is observed in the increase of the maximum permeability [2-3]. Furthermore, these results also show that the gap between the magnetic fields corresponding to the two maxima is very similar to twice the value of the H_c, as reported in [11].

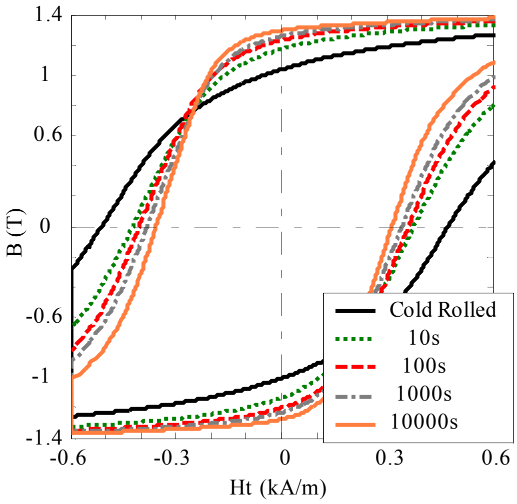

Figure 4. Hysteresis B-H loops showing the effect of the annealing treatment at 450ºC.

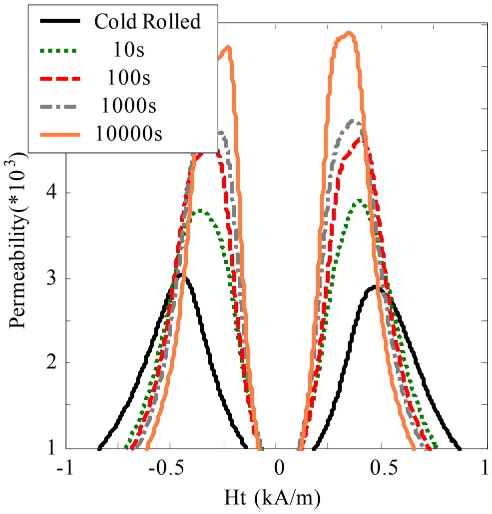

Figure 5. Evolution of the diferential permeability with the annealing treatment at 450ºC

In Figure 6 the evolution of remanent induction, $B_{r,}$ as a function of the annealing time is represented for the lowest temperatures (350ºC, 450ºC and 550ºC), in which only recovery effects occurred. It can be seen that B_r presents a progressive increase with the annealing time. These results could be explained due to the reduction in individual pinning site energies caused by the rearrangement of dislocations into low energy configurations and possibly by the removal of internal stresses caused by the annihilation and rearrangement of dislocations. This description is in agreement with arguments given by Thompson and Tanner [2] to explain the decrease of B_r found with increasing plastic strain. The results obtained for the samples annealed at 650ºC are not plotted because as previously indicated, hardness indentations present on those samples affect B_r measurements.

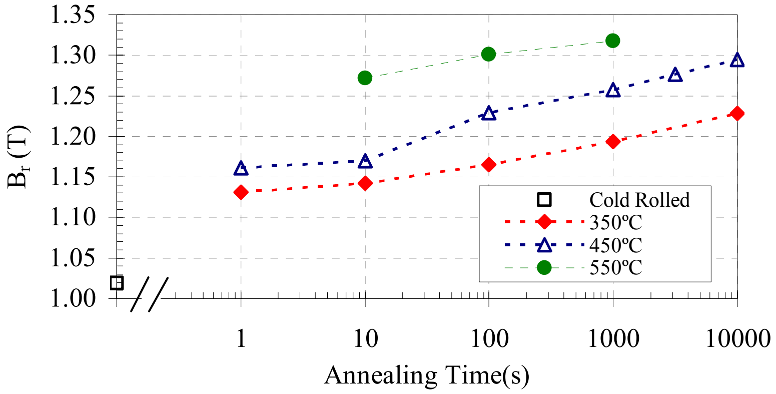

Figure 6. Evolution of remanent induction values during isothermal annealing at low temperatures, when only recovery processes take place.

Hysteresis loss values as a function of the annealing time are represented in Figure 7. On the one hand, it is observed that at 350ºC, 450ºC, 550ºC and at the shortest time (10s) at 650ºC, as a consequence of the recovery processes taking place in the substructure, a progressive improvement of the magnetic properties occurs, providing less hysteresis losses. This is explained by the fact that the domain wall movement turns out to be easier due to the reduction of the dislocation density occurring during recovery, so that less energy is needed to magnetise the material. These results are in agreement with the model proposed by Sablik and Landgraf [12] in which W_h is explained to be also proportional to the square root of the dislocation density.

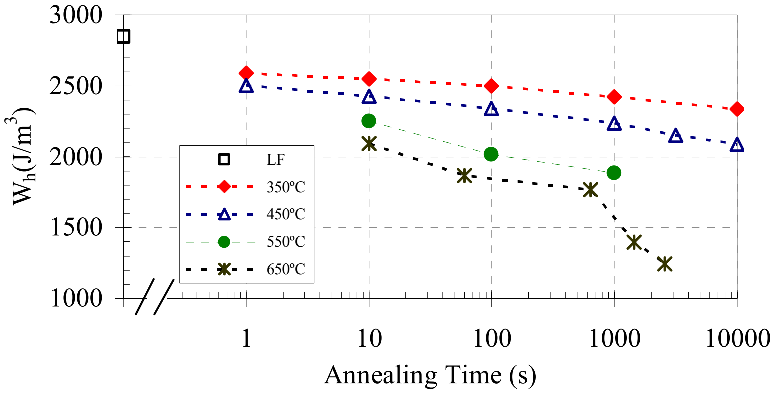

Figure 7. Evolution of the hysteresis losses during isothermal annealing at different temperatures.

It is observed that the evolution of hysteresis losses is very similar to the one found for the coercive field (compare Figure 7 with Figure 2). So, if the coercive field was found to be proportional to the recrystallized fraction in [6], the hysteresis losses are also expected to be useful to characterise the recrystallized fraction.

Figure 8 shows the linear relationship obtained between the W_h and the recrystallized fraction. It is observed that the decrease of W_h can be used to characterise the recrystallized fraction. It should be noted that in a previously reported cold rolled ELC steel [13] the evolution of W_h did not follow the progress of recrystallization. This was attributed to the additional dependence of the hysteresis losses on the grain size, as a decrease in the average grain size results in higher hysteresis losses [14], compensating the decreasing effect on hysteresis losses of the reduction in the average dislocation density during recrystallization. As previously mentioned in Section 3.2, in the ELC steel the variation of the grain size between the initial cold rolled steel and the complete recrystallization state is quite large (from 60 to 8 μm) [9] comparing with the variation found in this IF steel (from 27 to 11 μm) [6].

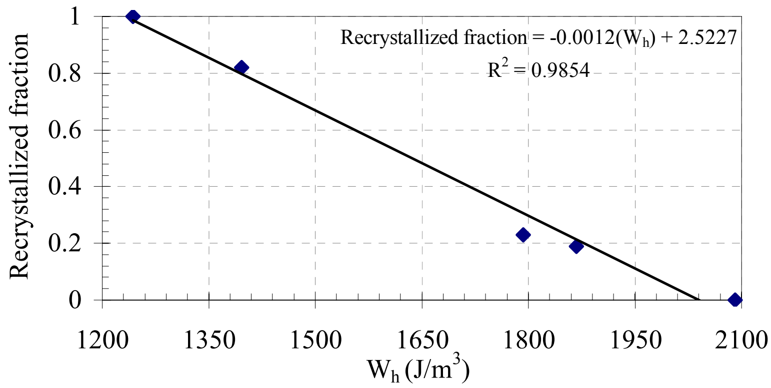

Figure 8. Linear relationship between the variation of hysteresis losses and the recrystallized fraction.

In Figure 9 the correlation between W_h and H_c on the analysed annealing states is shown. It is observed that, the linear correlation between W_h and H_c is satisfied while the average dislocation density is being reduced by recovery processes. This result can be attributed to the fact that both parameters are proportional to the average dislocation density and to its variation during the recovery. Additionally, it is shown that as soon as the recrystallization is activated, the same relationship is no longer satisfied because of the additional effect of the varying grain size and the generation of new grain boundaries.

During the recrystallization a different linear relationship is satisfied between W_h and H_c. It should be emphasized that in a previously reported ELC steel [13], although a linear relationship was verified during the recovery processes, a different linear correlation was not fulfilled during the recrystallization. This could be due to the fact that the variation of the grain size, as previously mentioned, was quite large in the ELC steel comparing with the variation found in this IF steel. As a consequence, it can be observed that, in both steels immediately after the recrystallization is activated the linear correlation satisfied during the recovery is no longer satisfied, with the loss of linearity in the ELC steel and the change of the slope in this IF steel. Therefore this correlation could be used to distinguish between recovery and recrystallization and to recognise the onset of recrystallization in both types of steels.

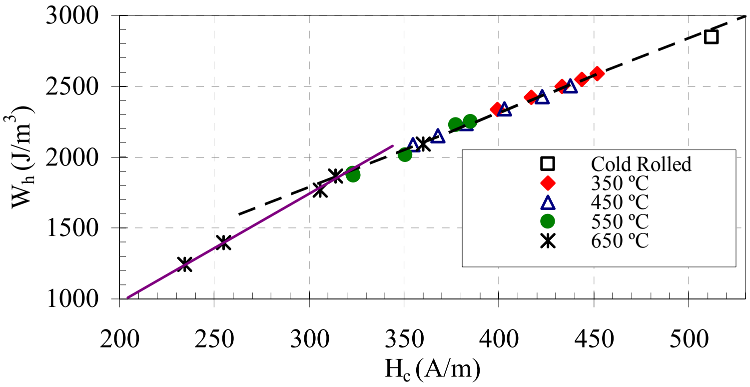

Figure 9. Correlation between the hysteresis losses and the coercive field at different annealing states.

Conclusions

On the one hand, in the present work it is shown how remanent induction and hysteresis loss parameters derived from the measurement of hysteresis loops can be useful to non-destructively characterise the extent of recovery during the isothermal annealing of a cold rolled Ti-Nb stabilized interstitial free ultra low carbon steel. Furthermore, the hysteresis losses, as the coercive field, have been observed to effectively monitor the recrystallization in this steel. Additionally, a linear correlation between the coercive field and the hysteresis loss is satisfied during the whole recovery process, whose slope changes and is maintained constant during recrystallization. This effect could be used to detect the onset of recrystallization and to distinguish it from recovery.

On the other hand, the effect on the magnetic parameters of the hardness indentations made on samples has been also analysed. It has been shown that the coercive field and the hysteresis losses values are not affected by these indentations, whereas the remanent induction is very sensitive to the stresses introduced by them.

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References

[1] M. Makar, B.K. Tanner, "In situ measurement of the effect of plastic deformation on the magnetic properties of steel", *Journal of Magnetism and Magnetic Materials,* 184, 2**,** 193-208, 1998.

[2] M. Thompson, B.K. Tanner, "The magnetic properties of specially prepared pearlitic steels of varying carbon content as a function of plastic deformation", *Journal of Magnetism and Magnetic Materials,* 132, 1-3**,** 71-88, 1994.
[3] V.E. Iordache, F. Ossart, E. Hug, "Magnetic characterisation of elastically and plastically tensile strained non-oriented Fe-3.2%Si steel", *Journal of Magnetism and Magnetic Materials*, 254**,** 57-59,2003.
[4] D.C. Jiles,"Magnetic properties and microstructure of AISI 1000 series carbon steels, *Journal of Physics D-Applied Physics*, 21**,** 1186-1195, 1988.
[5] V. Moorthy, S.Vaidyanathan, Baldev Raj, T. Jayakumar, B.P. Kashyap, "Insight into the Microstructural Characterization of Ferritic Steels Using Micromagnetic Parameters", *Metallurgical and Materials Transactions A*, 31A, 1053-1065, 2000.
[6] A. Martínez-de-Guerenu, M. Oyarzabal, F. Arizti, I. Gutiérrez, "Application of coercive field measurements to the evaluation of recovery and recrystallization in cold rolled interstitial free (IF) steel", Proceedings of the *International Conference on Microalloying for new steel processes and applications*, Donostia-San Sebastián (Spain), 7-9 September, 2005; Materials Science Forum, 500-501, 647-654, 2005.
[7] A. Martínez-de-Guerenu, K. Gurruchaga, F. Arizti, "Use of Magnetic Techniques for Characterisation of the Microstructure Evolution during the Annealing of Low Carbon Steels", *9th European Conference on Non-Destructive Testing (ECNDT)*, Berlin (Germany), 25-29 September, 2006. This proceedings.
[8] H. Traüble: *Magnetism and Metallurgy*, Vol. 2, Ed. A.E. Berkowitz and E. Kneller, Academic Press, New York, p. 621, 1969.
[9] A. Martínez-de-Guerenu, F. Arizti, M. Díaz-Fuentes, I. Gutiérrez, "Recovery during annealing in a cold rolled low carbon steel. Part I: Kinetics and microstructural characterization", *Acta Materialia*, 52, 3657-3664, 2004.
[10] A. Mager, "Über den EinfluB der KorngröBe auf die Koerzitivkraft", *Annalen der Physik*, 6, F. Lpz. 11, 15, 1952.
[11] Dong-Won Kim, Dongil Kwon, "Quantification of the Barkhausen noise method for the evaluation of time-dependent degradation", *Journal of Magnetism and Magnetic Materials*, 257, 2-3, 175-183, 2003.
[12] M.J. Sablik, F.J.G. Landgraf, "Modelling Microstructural Effects on Hysteresis Loops With the Same Maximum Flux Density", *IEEE Transactions on Magnetics*, 39, 5, 2528-2530, 2003.
[13] A. Martínez-de-Guerenu, K. Gurruchaga, "Nondestructive characterization of recovery and recrystallization in cold rolled low carbon steel by magnetic hysteresis loops", *Third Joint European Magnetic Symposia (JEMS)*, 26-30 June 2006, San Sebastián (Spain); Proceedings to be published in Journal of Magnetism and Magnetic Materials.
[14] J. Degauque, B. Astie, J.L. Porteseil, R. Vergne, "Influence of the grain size on the magnetic and magnetomechanical properties of high-purity iron", *Journal of Magnetism and Magnetic Materials*, 26, 1-3, 261-263, 1982.