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Journal of Magnetism and Magnetic Materials 316 (2007) e842–e845
Journal of magnetism and magnetic materials
www.elsevier.com/locate/jmmm

Nondestructive characterization of recovery and recrystallization in cold rolled low carbon steel by magnetic hysteresis loops

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Available online 19 March 2007

Abstract

How structure sensitive parameters derived from hysteresis loops can provide nondestructive information about the evolution of the microstructure of cold rolled low carbon steel as a result of recovery and recrystallization processes during the annealing is shown. The coercive field, remanent induction and hysteresis losses can be used to monitor the decrease in the dislocation density during recovery. These parameters are also influenced by the average grain refinement that takes place during recrystallization, which compensates the variation produced by the annihilation of dislocations during recrystallization. The maximum of the induction and of the relative differential permeability are shown to be very sensitive to the onset and to the monitoring of the recrystallization, respectively. The correlations between coercive field and remanent induction and hysteresis losses can also be used to distinguish between recovery and recrystallization.

PACS: 81.70.Ex; 75.60.−d; 81.40.Rs; 81.40.Ef

Keywords: Nondestructive characterization; Magnetic hysteresis loop; Recovery; Recrystallization; Low carbon steel

1. Introduction

Although some attention has been devoted to the study of the effect on the magnetic properties of recovery and recrystallization which occur during the annealing of plastically deformed electrical steels [1–2], little has been published concerning heavily cold rolled low carbon non-electrical steels [3,4] and specifically about the use of magnetic methods with the practical aim of developing tools for nondestructive evaluation of the evolution of the microstructure during these metallurgical processes.

Recovery involves both the annihilation of dislocations and their rearrangement into low energy configurations. Recrystallization leads to the suppression of dislocations by the nucleation of defect free volumes and by the migration through the material of the recrystallization front, resulting in a new grain structure with a low dislocation density. The loss by these processes of the dislocations introduced by work hardening produces mechanical softening of the steel [5].

In a previous paper [6], the evolution of the substructure during recovery and of the microstructure as a consequence of recrystallization of a cold rolled low carbon steel were studied by metallographic observations. In the same study, on the one hand, it was observed that hardness measurements in this type of steels do not give any information about the recovery processes and only experience an important drop at the onset of recrystallization. On the other hand, coercive field (H_c) measurements (Fig. 1) showed a higher degree of resolution and demonstrated that they could be used to satisfactorily monitor recovery during low temperature annealing (300–500 °C, and up to 11 s at 600 °C) [6,7], during which the grain structure remains constant and microstructural changes only occur in the cold rolling dislocation substructure inside the grains. As theoretical considerations indicate that H_c is proportional to the square root of the dislocation density (ρ) [8], the evolution of H_c could directly be related to the evolution of ρ during low temperature annealing [6,7]. At

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doi:10.1016/j.jmmm.2007.03.110

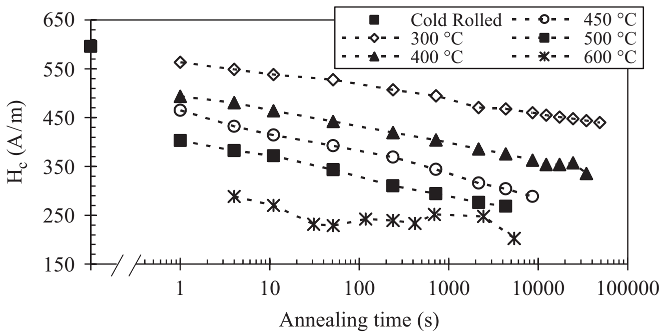

Fig. 1. Evolution of the coercive field (H_c) with annealing time (fractions of recovery were calculated from these values in Refs. [6,7]).

the highest isothermal annealing temperature studied (600 °C), as soon as the recrystallization was initiated a mechanical softening was produced [6]. However, although a corresponding decrease in H_c would be expected due to the decrease in ρ produced by the movement of the recrystallization front through the material, H_c stagnated. The stagnation of H_c during recrystallization (30–2460 s at 600 °C) was attributed to the dependence of H_c on both ρ and the inverse of the grain size (d_g) [9]. The initial grain size of the studied material is quite large (60 μm) and remains constant during recovery. However, during recrystallization (see optical micrographs in Fig. 2 of Ref. [6] showing its progress) the nucleation of new strain free grains leads to an effective grain refinement (complete recrystallization grain size is 8 μm), which produces an increase in H_c and therefore showed to compensate for the expected decrease produced by the dislocation annihilation taking place during recrystallization.

Following the above summarized study, the present one focuses on research into the use of other structure sensitive magnetic hysteresis loop parameters for the nondestructive characterization of the microstructural changes produced due to recovery and recrystallization during the annealing of the cold rolled low carbon steel presented above.

2. Experimental procedure

Samples (80 mm long × 12 mm width × 0.3 mm thickness) from industrially produced low carbon steel, 84% cold rolled, were isothermally annealed in laboratory under several conditions [6] in order to promote various degrees of recovery or recrystallization. The steel composition was 0.03%C–0.19%Mn–0.13%Al–0.0035%N–0.012%P–0.01% Si.

The magnetic hysteresis loops were measured using a system designed and constructed at the authors' laboratory. The external magnetic field was produced by a magnetic yoke composed of a 200-turn coil wound around a U-shaped magnetic laminated core. It was excited using a sinusoidal magnetizing current produced by a programmable function generator connected to a power amplifier. The magnetic induction signal was obtained after integrating the induced voltage in an encircling coil wound around the samples while the tangential magnetic field strength was measured using a Hall probe placed at the surface of the samples.

Four major hysteresis loops were recorded for each test applying a sinusoidal magnetic field strength of about 4.1 kA/m at 1 Hz, which was sufficiently high to saturate the measured samples. These were demagnetized prior to each test. The sampling frequency was 5 kHz, which gives 5000 measuring points for each cycle. Coercive field (H_c), remanent induction (B_r), maximum relative differential permeability (μ_{max}), maximum induction (B_{max}) and hysteresis loss (W_h) values were derived as parameters from each hysteresis loop. The parameters obtained from the four-hysteresis loops applied in each test were averaged and the measurements shown were calculated as the average of two complete tests. Experimental errors were: for H_c, B_r and W_h: $<0.5\%$; for μ_{max}: $\sim 5\%$; for B_{max}: $<0.2\%$.

3. Results and discussion

In general, it was observed that the annealing of the cold rolled steel produces a progressive improvement of the magnetic properties. The B–H curves become steeper, providing lower H_c and W_h, and higher B_r and μ_{max} values. Figs. 2–4 show, respectively, the evolution of B_r, μ_{max}, and B_{max}, as a function of annealing time. The value obtained for the cold rolled sample is also included for reference in

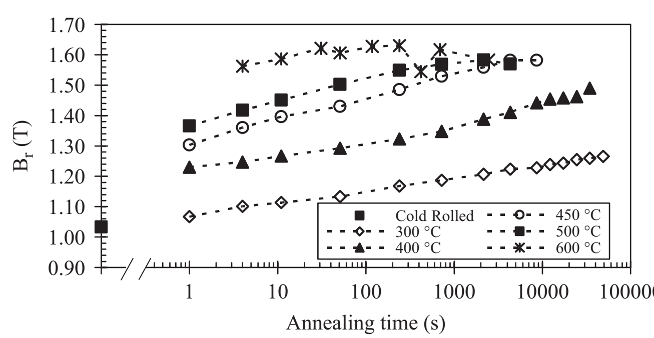

Fig. 2. Evolution of the remanent induction (B_r) with annealing time.

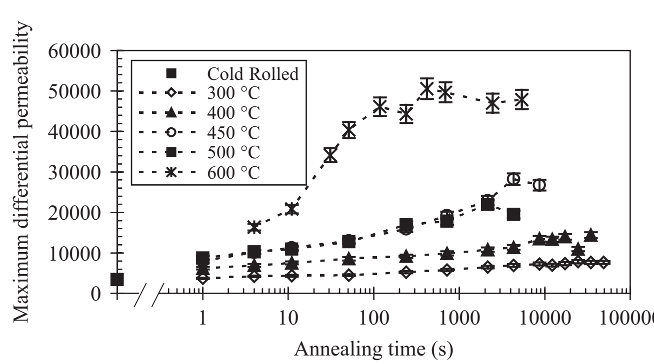

Fig. 3. Evolution of the maximum relative differential permeability (μ_{max}) with annealing time.

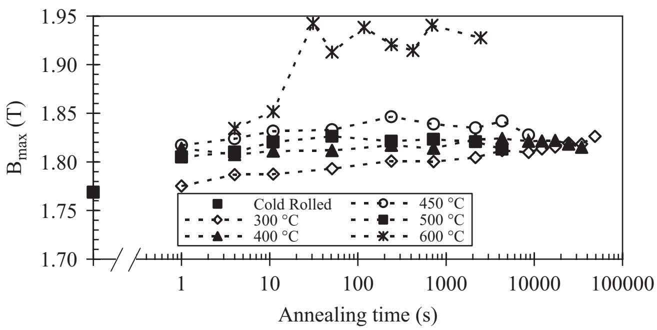

Fig. 4. Evolution of the maximum induction (B_{max}) with annealing time.

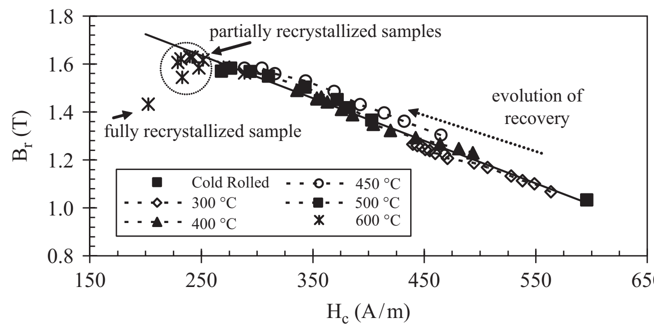

Fig. 5. Correlation between H_c and B_r during recovery and recrystallization.

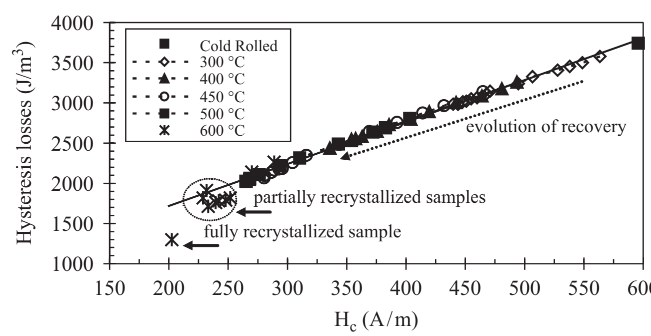

Fig. 6. Correlation between H_c and W_h during recovery and recrystallization.

all the figures. It can be seen that B_r (Fig. 2) presents a progressive increasing tendency with the annealing time during recovery (300–500 °C and up to 11 s at 600 °C) and then, it shows an increase with a slower slope up to 240 s during recrystallization, up to a recrystallized fraction of 85%, to eventually decrease at the final stages of recrystallization. This response can be attributed to the reduction of individual pinning site energies caused by the rearrangement of dislocations into lower energy configurations [10]. The μ_{max} (Fig. 3) gradually increases during recovery, slowly at 300–400 °C, with a slightly higher slope at 450–500 °C and up to 11 s at 600 °C. At the onset of recrystallization (11 s at 600 °C) μ_{max} experiences an important increase during the progress of recrystallization up to 400 s, when the microstructure showed a fraction of 90% recrystallization. These results are in agreement with Husain et al. [11], who found by alternating current potential drop measurements a continuous increase of relative permeability during recrystallization.

Although saturation magnetization is a structure-insensitive property, the maximum induction measured in the approach to saturation at a magnetic field of 4.1 kA/m was found to vary with microstructure (Fig. 4). During recovery it only experienced a very slight increase reaching the same saturation value irrespective of the temperature. However, it showed a slow variation up to 11 s at 600 °C during recovery, a strong increase between 11 s and 31 s at the onset of recrystallization, and a slight variation around a constant value in the range 31–2460 s with the progress of recrystallization. The slight variation of B_{max} during recovery could be attributed to the low sensitivity to variations in the dislocation density at high magnetic field values [12].

It can be observed that B_r (Fig. 2) reveals an opposite trend to the one shown by H_c (Fig. 1). The correlation between these parameters is shown in Fig. 5. A linear correlation between these parameters is satisfied while the dislocation density is reduced by recovery processes, which only affect the substructure of the steel. As soon as the recrystallization is activated, the same relationship is no longer satisfied because of the additional effect of the varying grain size.

The W_h evolution as a function of annealing time is also very similar to the one found for H_c and it is not explicitly shown for lack of space. The correlation between W_h and H_c is shown in Fig. 6. It is observed that, the linear correlation between W_h and H_c is also only verified during recovery.

4. Conclusions

H_c, B_r, μ_{max} and W_h adequately monitor the reduction in the average dislocation density due to recovery during low temperature annealing of cold rolled low carbon steel, when mechanical techniques such as hardness [6] do not show enough sensitivity.

H_c, B_r and W_h are also affected by the grain refinement occurring during recrystallization, hiding the influence of the concurrent large decrease in dislocation density. However, the correlations between these parameters could be used to distinguish between recovery and recrystallization and to recognize the onset of recrystallization.

B_{max} and μ_{max} can be used to detect the onset of recrystallization and μ_{max} to follow its progress.

Acknowledgment

The financial support of the MITyC of Spain under the project: FIT-020400-2005-285 is gratefully acknowledged.

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