1. Introduction

Both destructive and non-destructive testing (NDT) techniques are available to characterize recovery and recrystallization processes which are experienced by the cold rolled steel microstructure during annealing. To date the most popular of these techniques are microstructural observations and hardness measurements.

Recovery is related to the annihilation of dislocations and their rearrangement into low energy configurations. Quantification of dislocation densities in materials can be achieved through transmission electron microscopy (TEM). However, as heavily cold rolled steels develop a highly complex substructure, such evaluation cannot be accurately achieved and TEM is replaced by indirect methods like magnetic techniques,1–3) X-ray line broadening, 4) X-ray peak resolution, 5) tensile stress 6,7) or Vickers hardness measurements.8,9) Among these techniques, magnetic measurements have the advantage of being non-destructive in nature. As theoretical considerations demonstrate that the coercive field, $H_c$, is proportional to the square root of the dislocation density, $\rho$:10)

$$H_c \propto \sqrt{\rho} \quad \text{...............................(1)}$$

this parameter has been the most used among several magnetic parameters to monitor recovery in different materials.1–3,11–13) During recovery, the grain structure remains constant and microstructural changes only occur in the cold rolling dislocation substructure inside the grains. The reduction in the dislocation density during recovery decreases the density of pinning sites for the motion of magnetic domain walls in the matrix, which is normally reflected as a decrease in $H_c$ values from the cold rolled state.1–3,11–13)

As a fast, although minimally invasive technique, hardness measurements have been shown not to have enough sensitivity to characterize the recovery processes in a heavily cold rolled low carbon steel9) and in an interstitial free (IF) steel to have small resolution at temperatures slightly below the onset of recrystallization, but to be absolutely insensitive at lower temperatures.12)

Recrystallization produces microstructure regeneration through the nucleation of substructure free volumes and by the recrystallization front migration through the material which results in a new grain microstructure producing the mechanical14) and magnetic softening of steel.15) There were attempts to apply magnetic techniques to monitor microstructural changes during recrystallization.16) However, in spite of the importance that NDT techniques have from an industrial point of view this subject has received little attention until recently.3,12,17–19) Recrystallized fraction is generally determined in the laboratory by metallographic observations, which are time consuming and often rather subjective, or by hardness measurements. Although hardness has provided good estimations of the recrystallized fractions in some extra low carbon (ELC) steels,20,21) in others (including IF steels) the mechanical softening significantly deviated from the recrystallized fraction.12,20,22,23) Higher softening fractions than recrystallized fractions are generally attributed to recovery concurrent with recrystallization. Moreover, NDT methods are applied at the plants as microstructurally blind quality control techniques and some information related to microstructure implicit in the signals is lost.

The microstructure affects magnetic domain wall movement, the different microstructural features (e.g. grain boundaries, dislocations, precipitates) acting as pinning elements.24–26) For example, the pinning of domain wall motion increases with increasing total grain wall density.
Since \( H_c \) reflects the amount and strength of pinning, \( H_c \) is expected to increase with the grain boundary density. In an equiaxed polycrystalline sample the density of grain boundaries is inversely proportional to the grain size, so that a decrease in the grain size produces an increase in the total grain boundary density. Therefore, both in theoretical and in experimental works, \( H_c \) has been found to be directly proportional to the inverse of the grain diameter, \( d_i \), of the material:

\[
H_c \propto \frac{1}{d_i} \quad \text{................................(2)}
\]

The difficulty arises when analyzing a complex microstructure in which more than a predominant microstructural element is present. As an example, to the best of the authors' knowledge, the simultaneous influence in \( H_c \) of the dislocation density and the grain size has not been previously analyzed, except for recrystallized microstructures with dislocation densities in the range of \( 10^{10} \)–\( 10^{12} \) m\(^{-2}\). This is several orders of magnitude lower than those expected in heavily cold rolled materials.

Within this context, in the present study, magnetic coercive field, hardness, metallographic measurements and electron back scattered diffraction (EBSD) analysis are applied in order to research the sensitivity and limitations of each of the techniques to monitor recovery and the onset and evolution of recrystallization.

2. Experimental Procedure

An extra low carbon steel, coiled at 650°C, industrially cold rolled through a reduction of 76% to a final thickness of 0.53 mm with a chemical composition in wt% of 0.03 C, 0.38 Mn, 0.0004 S, 0.11 Si, 0.037 P, 0.035 Al and 0.004 N and 0.03 C, 0.38 Mn, 0.0004 S, 0.11 Si, 0.037 P, 0.035 Al and 0.004 N and a hot band grain size of 12 \( \mu \)m was analyzed in the present study. Samples (200 mm long \( \times \) 14 mm wide) were cut perpendicular to the rolling direction of the cold rolled sheet. The obtained specimens were isothermally annealed at low temperatures, 300, 400 and 500°C, in order to promote recovery and avoid interaction with recrystallization, and at a higher temperature, 575°C, in order to produce recrystallization. Annealing was performed using a continuous annealing simulation device at a heating rate of 20°C/s up to the holding temperature, and gas cooling at a rate close to \(-60^\circ\)C/s back down to room temperature. Isothermal holding times ranged from 1 s to 10 000 s (2 h 47').

Near saturation magnetic hysteresis loops were measured for each annealed sample as reported in Refs. 3, 19). The coercive field \( H_c \) values were extracted from each hysteresis loop. Magnetic measurements were made with the magnetic field applied in the longitudinal direction of the specimens (in the transverse direction on the rolling plane).

The fraction of residual strain hardening (1-\( R_y \)), or its complementary fraction of magnetic softening, was determined in terms of the relative coercive field change (\( X_{HR} \)) as:

\[
1 - R_y = 1 - X_{HR} = \frac{H_c(T, t) - H_{cd}}{H_{co} - H_{cd}} \quad \text{................................(3)}
\]

where \( H_c(T, t) \) is the coercive field value at each annealing stage, and \( H_{co} \) and \( H_{cd} \) are the \( H_c \) values of the cold worked material and of the fully recrystallized samples, respectively.

The mechanical softened fraction, \( X_{HR} \), was determined by using the following expression:

\[
X_{HR} = \frac{H_o - H_i}{H_o - H_f} \quad \text{................................(4)}
\]

where \( H_o \), \( H_i \) and \( H_f \) are the HR(30T) hardness measurements of the cold rolled material, of the material after an annealing treatment during a holding time \( t \) and after being fully recrystallized, respectively.

Metallography was performed on the longitudinal section of the sheet (RD (rolling direction)/ND (short transversal direction)) after applying conventional polishing techniques and 2% nital etching. Quantitative metallography (point counting method) was carried out on images obtained by optical microscopy to obtain the recrystallized volume fractions, \( X_v \), of the samples. The equivalent diameter and the mean linear intercept of the recrystallized grains were determined by quantitative metallography from the optical images.

Some samples were additionally polished in the same section using a colloidal silica solution and EBSD observations were carried out with a Philips XL30cp scanning electron microscope (SEM) with W-filament, using a TSL (TexSEM Laboratories) Pegasus 4000 (OIM 4000 with CCD DigiView III camera) system. A step size of 0.15 \( \mu \)m was used in acquiring the EBSD data.

3. Results

Figure 1 shows the evolution of the relative changes in the coercive field as a function of the annealing time in logarithmic scale for the different annealing temperatures. It is observed that in the range 300°C≤\( T \)≤500°C, the coercive field decreases progressively with annealing time. At 500°C, \( H_c \) initially follows this same trend, but stagnates for annealing times higher than 3 160 s. At 575°C, initially between 10 and 25 s \( H_c \) undergoes the same type of initial drop as at the rest of the temperatures. It stagnates for annealing times between 25 and 200 s, diminishes slightly between 200 and 400 s and decreases abruptly afterwards, which is a significantly different behaviour than that at
lower temperatures.

The graph in Fig. 2 displays the evolution of hardness (HR30T) with annealing time. It can be seen that for annealing treatments performed at 300 and 400°C with soaking times as long as 10 000 s, the hardness of the annealed steel, as compared with that of the cold rolled material, slightly increases, and shows a small decrease at 500°C. By contrast, at 575°C the hardness experiences a continuous decrease from the very beginning of the annealing treatment, followed by an important drop after 200 s to reach a final full softening after 1 600 s. As can be seen in Fig. 3, optical microstructural observations at different annealing stages at this temperature show that recrystallization takes place throughout this annealing time range. It is observed that nucleation takes place at initial grain boundaries and at the interior of certain grains.

The evolutions of the recrystallized fraction at 575°C and the fractional hardness softening are plotted as a function of the annealing time in Fig. 4. It is observed that the hardness softening fraction of the steel is higher than the recrystallized fraction, the main deviations arising at low fractions. The relative magnetic softening fraction, using the value of $H_c$ measured before the onset of recrystallization at 575°C

![Graph showing hardness evolution with annealing time](image)

**Fig. 2.** Hardness values after a holding time of 10 000 s at 300, 400 and 500°C and after different annealing times at 575°C.

![Micrographs showing microstructure evolution](image)

**Fig. 3.** Optical micrographs showing the evolution with time of the microstructure during annealing at 575°C: a) 25 s, b) 200 s, c) 400 s, d) 800 s, e) 1 600 s, f) 3 200 s.
(10 s) as $H_{\text{co}}$ (labelled $H_{\text{co}}^{11032}$), is also plotted on the same graph. It can be seen that such data superimpose at low fractions to those of the hardness softening, while after 200 s they approximately overlap with the recrystallized fraction.

The recrystallized grain size distributions for the different annealing times at $575^\circ$C are plotted in Fig. 5. It is observed that after 200 s of annealing time a volume fraction of $X_v=0.12$ leads to a mean grain size of 2.5 $\mu$m. At 400 s some broadening of the distribution takes place and the principal peak shifts to slightly lower grain sizes. When the annealing time increases, the size distribution is clearly shifted to higher values. After 1 600 s of annealing, when nearly the entire material is recrystallized, the mean grain size is of around 4.5 $\mu$m.

Specimens were selected in order to study in more detail the microstructural behaviour at the early stages of recrystallization. EBSD images were obtained after a short annealing treatment (25 s) at $575^\circ$C, on the specimen annealed during a long period of time (10 000 s) at $500^\circ$C and on the one annealed at $400^\circ$C during 10 000 s. According to the data in Figs. 1 and 2, the second specimen is slightly softer (mechanically and magnetically) than the material after 25 s holding at $575^\circ$C. This is why this specimen was selected for the EBSD study, while the third one was taken as reference material. The maps showing the high angle boundaries ($\theta > 15^\circ$) have been selected from all the EBSD images obtained, as can be seen in Fig. 6.

The image obtained on the sample annealed at $400^\circ$C (Fig. 6(a)) presents heavily deformed grains with low image quality indices that appear as dark areas when a high angle...
boundary map is drawn. Some light areas can also be observed, some of them displaying small crystallites only a few microns in size. However, the distribution of the high angle misorientations in the sample does not allow these crystallites to be identified as recrystallized grains, but as belonging to the deformed microstructure.

After the same holding time at 500°C, the high angle boundary map reveals (Fig. 6(b)) the presence of several crystallites a few microns in size that appear surrounded by well defined high angle boundaries. Most of the time these crystallites appear forming clusters. The misorientation profile in Fig. 6(c) has been obtained across one of these clusters, which gives a good example of the well defined high angle boundaries surrounding these recrystallized grains.

At 575°C a holding time of 25 s (see Fig. 6(d)) leads to an intermediate state between the two already described. The overall image quality is higher than the one reached after the annealing treatment at 400°C, but the signs of recrystallization are scarce and well defined high misoriented crystallites appear forming clusters. The misorientation profile in Fig. 6(c) has been obtained across one of these clusters, which gives a good example of the well defined high angle boundaries surrounding these recrystallized grains.

4. Discussion

In non-stabilised steels, the presence of some free nitrogen and the dissolution of some carbon during annealing are both unavoidable. When carbon and nitrogen dissolve in α-iron, they tend to enter the lattice interstitially and produce a local lattice straining, which could contribute to a degradation of the magnetic properties, which would be reflected as an increase of the coercive field. However, some experimental results obtained in carbon steels showed that the amount of carbon in solid solution in α-iron was not a very significant factor affecting structure-sensitive magnetic properties. Additionally, it is expected that in cold rolled steels these elements segregate to dislocations, instead of entering interstitially into the ferrite matrix. Consequently, their direct effect on the coercive field measurements should be minor even if some influence on the recovery rate should be expected.

The microstructural observations carried out after the annealing treatments performed at the lowest annealing temperatures, between 300 and 500°C (up to 3 160 s at 500°C) did not show any indication of recrystallization. So, the variations observed at these temperatures in \( H_c \) measurements can be attributed to recovery mechanisms. The evolution of \( H_c \) with the annealing time can be fitted in the range 300–500°C by a logarithmic function of the form:

\[
1 - R_y = b - a \ln t \quad \text{.................................(5)}
\]

where \( a \) and \( b \) are constants for a given temperature. This type of interdependence can be directly related to the decrease of the dislocation density due to recovery and has previously been applied to other steels. The values of the \( a \) and \( b \) constants determined by least square fitting of the experimental data to Eq. (5) are plotted in Fig. 7 as a function of the annealing temperature. It can be observed that the \( b \) coefficient decreases monotonously with temperature, while the \( a \) coefficient diminishes monotonously only slightly after 400°C, showing a different trend at the lowest temperature of 300°C.

The only \( H_c \) measurement point that deviates from the general trend described by Eq. (5) within the above mentioned temperature range is the one corresponding to a long annealing time at 500°C. Under this condition, some mechanical softening is also evident (see Fig. 2). In order to investigate the eventual onset of recrystallization for this annealing condition, EBSD observations were carried out. At initial recrystallization stages, it is difficult to unambiguously identify recrystallized grains from the deformed matrix. In the present case, those crystallites surrounded by well defined grain boundaries, even if small in size, have been taken as recrystallized grains. After a 10,000 s holding at 500°C, a small recrystallized fraction is clearly observed (see Figs. 6(b) and 6(c)). This explains the deviation of the point corresponding to this treatment from the general trend applicable when only recovery is operative (Eq. (5)).

After 25 s holding at 575°C, some small crystallites appear on the optical images, (see Fig. 3), but this is not an unambiguous indication of the onset of recrystallization. EBSD images do not provide additional insight about this point (Fig. 6(c)) and the state of the material can be classified as being between the state after a long annealing at 400°C and the onset of recrystallization observed at 500°C with well defined high misoriented grains. It is to be noted that, in terms of hardness and of \( H_c \), the material after annealing for 25 s at 575°C, is also placed in between the two other specimens. This indicates that all the applied techniques: \( H_c \) measurements, hardness and EBSD observations distinguish that the degree of recrystallization is higher after a long annealing at 500°C than after a short one at 575°C.

Coming back to the recovery stage, it can be observed that the hardness of the steel (Fig. 2) is almost insensitive to the microstructural changes taking place in the material during the annealing treatments performed at the lowest annealing temperatures, 300 and 400°C, even after long annealing treatments. Similar results have previously been reported for ELC steel. It has been shown that annealing for long times (10,000 s) at these temperatures produce, in the present steel, a slight increase in hardness (see Fig. 2) as compared with the value of this variable in the cold rolled material. This is probably due to some aging (bake hardening) produced by the interstitial elements, mainly carbon in solid solution that interacts with dislocations.

It should be noticed that the total relative decrease in \( H_c \)
at low annealing temperatures is much higher than that detected by hardness measurements, in agreement with previous results in ELC\textsuperscript{33} and in IF\textsuperscript{31} steels. Thus, as a general rule, it can be said that the magnetic coercive field shows a higher degree of resolution than hardness measurements when researching recovery phenomena.

On the other hand, the comparison between the evolution of the softening and the recrystallized fraction curve (see Fig. 4) shows that at 575°C simultaneous recovery with recrystallization takes place. This concurrent recovery effect is larger at low recrystallized fractions and decreases as recrystallization progresses and the deformed areas decrease, in agreement with results reported in Refs. 12), 36). It can be seen that the magnetic softening overlaps with the mechanical softening at the onset of recrystallization, but after a certain recrystallization fraction is reached (~12%), the magnetic softening and the recrystallized fraction superimpose, while the mechanical softening is slightly higher. In the absence of previous knowledge which would allow the different contributions to $H_c$ to be quantified for complex microstructures, like those in heavily cold rolled and in partially recrystallized samples, only a qualitative analysis on the effect of the eventual microstructural refinement taking place during recrystallization can be carried out.

It has to be taken into account that the grain structure varies during the progress of recrystallization from that of the elongated grains in the cold rolled material to that of the equiaxed microstructure at the completion of recrystallization. The grain boundaries present obstacles to magnetic domain wall motion and act as pinning centers for the magnetic domain walls. Therefore the domain wall mobility is determined by the volume density of the grain boundaries.\textsuperscript{26}

The grain boundary surface area per unit volume, $S_v$, can be related to the grain size, expressed as the mean linear intercept (MLI) length, $L$, according to\textsuperscript{37}:

$$ L = \frac{2}{S_v} \quad \text{(6)} $$

In a deformed microstructure, both the dislocation network and the grain boundaries are expected to affect the motion of the magnetic domain walls. For a planar-linear microstructure produced by rolling, the surface area of the deformed grains per unit volume, $S_{\text{def}}$, can be expressed as\textsuperscript{37,38}:

$$ S_{\text{def}} = \frac{0.429}{L_{\text{RD}}} + \frac{0.571}{L_{\text{TD}}} + \frac{1}{L_{\text{ND}}} \quad \text{(7)} $$

with $L_{\text{RD}}$, $L_{\text{TD}}$ and $L_{\text{ND}}$ being the MLI lengths in the longitudinal, transverse and short transverse directions, for the present case 50, 12 and 2.9 $\mu$m, respectively. For consistency with Eq. (2), an effective MLI, $L_{\text{eff}}$, can be calculated by inserting the value of $S_{\text{def}}$ in Eq. (6). It has to be noted that this $L_{\text{eff}}$ is different than the equal volume equivalent grain size $L_{\text{eq}}$, but corresponds to the size of the equivalent equiaxed grain with the same grain boundary area per unit volume ($S_{\text{def}}$) as the true elongated grains.

When a cold rolled material recrystallizes, $S_v$ changes from the value given by Eq. (7), which depends on the initial grain size and the applied cold rolling reduction, to the value obtained when inserting in Eq. (6) the MLI corresponding to the fully recrystallized material. In a partially recrystallized material the contributions to $S_v$ come from the part of the initial grain boundaries not yet consumed by recrystallization ($S_{\text{def}}$) and from the grain boundaries surrounding the new grains ($S_{\text{eq}}$).

Assuming that $S_{\text{def}}$ in the partially recrystallized specimen decreases proportionally to the degree of recrystallization, the surface area per unit volume can be estimated as:

$$ S_{\nu_x} = \left( \frac{0.429}{L_{\text{RD}}} + \frac{0.571}{L_{\text{TD}}} + \frac{1}{L_{\text{ND}}} \right) (1 - X_v) + \frac{2}{L_{\text{eff}}} X_v $$

$$ \quad \text{(8)} $$

with $L_{\text{eff}}$ being the MLI for the grains in the recrystallized areas. The above estimation does not take into account the effect of preferential nucleation on initial grain boundaries, but assuming saturated nucleation, it can be applied as a first approximation. The corresponding effective MLI, $L_{\text{effX}}$, for a partially recrystallized microstructure can be estimated through the application of Eq. (6) to the values of $S_{\nu_x}$ during the progress of recrystallization.

The obtained values for the variation of $L_{\text{defX}}$ are shown in Fig. 8, which indicate that the effective MLI in this steel varies little during recrystallization. The initial microstructure has a hot band grain size that when cold rolled gives a specific grain boundary area equivalent to the one of an equiaxed microstructure of about 5 $\mu$m grain size (MLI). As recrystallization takes place there is some refinement of the microstructure (non-recrystallized areas being substituted by fine recrystallized grains, Fig. 3), leading to an effective MLI of about 4 $\mu$m for a softened fraction of 0.44 ($X_v=0.3$) followed by some effective microstructure coarsening (due to the increase in the size of recrystallized grains) to reach a recrystallized grain size at the completion of recrystallization of about 5 $\mu$m (MLI).

This has different implications that can explain the behaviour of $H_c$ shown in Fig. 1:

- If the effective size of the microstructure remains constant during recrystallization, the main contribution to the evolution of $H_c$ is expected to be due to the annealing out of dislocations.
- The initial refinement of the microstructure explains why the experimental $H_c$ at the beginning of recrystallization (at 575°C for $t=400$ s, $X_v=0.3$) has higher values than the expected ones when extrapolating to 575°C the re-

Fig. 8. Effective mean linear intercept (MLI) during recrystallization at 575°C.
Acknowledgement

The authors acknowledge the financial support from the Spanish Ministry of Education and Science (Ministerio de Educación y Ciencia) (MAT 2006-05805). M. Oyarzábal would also like to express her gratitude to the Basque Government for her research grant.

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