Influence of cold rolling reduction on mechanical and magnetic properties and on ultrasound features in low carbon steel

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Abstract

The electric, magnetic, ultrasonic and mechanical properties of ferritic steels are all related to the microstructure and this provides opportunity for non-destructive evaluation if the particular relations linking them are well understood. In this work, the effect of the microstructural changes produced by cold rolling in a low carbon steel is investigated by these techniques. Parameters derived from both magnetic and ultrasonic measurements are finally related with the increase of the dislocation density produced by cold rolling deformation.

1. Introduction

Non-destructive evaluation (NDE) systems based on magnetic and ultrasound techniques are receiving increasing interest for on-line assessment of microstructure and mechanical properties during processing and production of steel strips. On-line measurements give the opportunity to adapt process parameters to the actual state of the material. Industrially produced microstructures have different degrees of complexity and the mechanical behaviour is defined by the composition, grain size, dislocations densities, crystallographic texture and second phases [1,2]. The same happens between NDE techniques and the microstructural variables [3], but the exact relations between the parameters derived from these measurements and the microstructure or the mechanical behaviour are not straightforward, even for the simplest microstructures. Consequently, it is important to separate the different microstructural contributions to both magnetic and ultrasound measurements and this interest increases with the complexity of the microstructure. However, one of the main difficulties is that it is almost impossible to vary over a sufficiently broad scale a single microstructural parameter at a time, while keeping the rest constant. Cold deformation for example affects the shape of the grains and produces dislocations and grain subdivisions [4-6], but it also affects crystallographic textures that have a strong influence on the electric, magnetic and mechanical properties of the steel [7].

Magnetic measurements allow the non-destructive monitoring of the change in the dislocation density during recovery [4,8,9], variations in grain size [10] and the combined effect of dislocation density and grain size during recrystallization [6]. Most of the times,
the research works addressing the effect of deformation on the magnetic parameters apply cold reductions (i.e. up to 40% in Refs. [11,12], but lower in others works [13]). These reductions are significantly lower than those usually applied industrially in order to induce the formation of favorable textures during annealing [14].

For the determination of material properties, ultrasonic waves can also be used, because they are able to examine the complete volume of the sample and depend on the elastic constants of the material. Important sound quantities are speed and attenuation and their dependence on frequency. Direction dependent properties caused by textures show up especially in the propagation of shear (transversal) waves where the amplitudes are perpendicular to the direction of propagation. The connection between wave propagation, elastic constants and orientation distribution functions (ODF) are given in literature [15].

This work investigates the influence of the microstructural changes induced by the cold rolling deformation on electric, magnetic, ultrasonic and mechanical properties. The possibility of using magnetic and ultrasonic measurement parameters to characterise the cold rolling reduction of steel sheets is particularly focused.

2. Experimental methods

An industrial hot rolled extra-low carbon steel with the following composition was used in this work: 0.048C – 0.191Mn – 0.010P –0.001Si –0.040Al –0.031N (wt-%). The hot rolled sheet with an initial thickness of 3 mm was cold rolled in a pilot plant to 8 different degrees of cold reduction in the range between 12 and 81% at 10% intervals.

Samples for microstructural characterisation were prepared by conventional methods, followed by Nital 2% etching and colloidal silica polishing for both optical and EBSD (Electron Back Scattered Diffraction) observations, respectively. The EBSD scans were performed in a FEG–SEM JEOL JMS 7100 F equipped with a NORDLYS II camera. The observations were performed on the longitudinal section.

Mechanical properties were analysed by tensile testing and Vickers hardness measurements. Two tensile tests per condition were carried out at room temperature and a strain rate $10^{-3}$ s$^{-1}$ on a 5982 Instron testing machine.

The electrical resistances of the samples were characterised in a 4-point measurement setup using a commercial microOhm meter. The magnetic hysteresis loop measurements were performed at room temperature using a single sheet tester previously described elsewhere [16]. Near saturation AC major hysteresis loops, applying a maximum magnetic field strength of about 5500 A/m, were recorded at 0.1 Hz using an encircling coil wound around machined strip samples (100 mm in length, 10 mm width) samples. The coercive field value, $H_c$, was determined from each hysteresis loop as the average of the absolute values from both branches of the tangential field where the induction becomes zero.

Longitudinal ultrasonic waves were generated with a standard probe head of a frequency of 4 MHz, incident in the normal direction of the sample. Normally the sound speed is determined by measuring the time of flight of the wave tracks. In the thinnest sheets with
a thickness comparable to the wavelength, the determination of the sound speed can be
done using resonances of the material near the probe frequency. Only stationary wave
modes can propagate under these boundary conditions, so a frequency analysis with FFT
gives characteristic sets of resonance frequencies. The data were recorded with an
oscilloscope and the spectra calculated with fast Fourier transform (FFT).

3. Results and discussion

3.1 Microstructural evolution with cold reduction

The evolution of the microstructure with the applied cold reduction is shown in EBSD
images in Figure 1. The grain microstructure progressively elongates in the rolling
direction, while an ingrain substructure develops.

![EBSD IPF (inverse pole figure) maps referred to ND (nodal direction) for the indicated
cold reductions.](image)

3.2 Electrical resistivity measurements

Electrical resistivity measurements on the four samples with cold reductions equal or
larger than 51% were performed using a 4 point measurement with a microOhm meter
model OM 21 from the company AOIP, Ris Orangis, France. Resistivity values of
0.129 \( \mu \Omega \text{m} \pm 1\% \) were measured. The reduction percentage had no effect on the electrical
resistivity (within 1% variation), which is in agreement with the results reported for
stainless steel at room temperature [17], which means that the dislocation density
variation produced by cold rolling has a negligible effect on the resistivity.

3.3 Magnetic measurements

The evolution of the hysteresis loops with the applied cold reduction is shown in Figure
2-a). After the first cold reduction stage, the shape of the hysteresis loop becomes swelled
and as the reduction ratio increases the loop inclines to the horizontal axis. Figure 2-b)
shows the evolution of the coercive field (Hc) values, derived from the hysteresis loops,
as a function of the cold rolling reduction. It clearly shows the monotonic increase of Hc
with the applied cold reduction.
3.4 Ultrasonic measurements

Figure 3-a) shows the spectra recorded from the rolled samples as a function of sample thickness. The shift of the resonance peak in frequency is due to the thickness reduction. To determine the sound speed, the order of the resonance must be respected. In Figure 3-b) the calculated ultrasound longitudinal wave velocities in the rolled samples is shown as a function of the cold rolled reduction ratio. Main source of error in this case is the proper determination of the thickness.
A decrease of sound speed with increasing rolling degree (decreasing thickness) is found (without taking into account the outlier data found at 51% cold reduction sample). This is due to the higher density of dislocations the ultrasound waves have to encounter with increasing deformation. In the same way, the attenuation suffers a slight change for cold reductions below 51% followed by a sharp increase. The steep increase corresponds to the change in the shape and amplitude of the spectra shown in Figure 3-a) at 1.2 mm at the state of 61% cold reduction.

The thickness of the material is smaller than one wavelength in the cold reduction stages larger than 61% (below 1.5 mm thickness). The limit for the propagation of ultrasound is reached when the thickness equals half a wavelength. Near this region resonance spectra show up an increasing number of harmonics that can mix up and interfere. Figure 3a) shows these spectra at the last three thickness levels from 1.2 mm to 0.6 mm (61% to 81% cold reduction). So, the steep increase of the attenuation might be caused by changing interference conditions. In any case, the attenuation rises with increasing deformation and dislocation density. Moreover, this transition in the attenuation coincides with a range of reductions for which the grain aspect on the EBSD images changes drastically and the original grains become subdivided (Figure 1). However, this effect should be investigated more in depth.

3.5 Correlation between mechanical properties and magnetic/ultrasound properties

Figure 4 shows the evolution of the relative coercive field (Hc) values, derived from the hysteresis loops, as a function of the cold rolling reduction, together with the relative Vickers hardness (Hv) and relative tensile strength (Rm). To facilitate the comparison of the results in this graph, the variation of the values for each variable has been normalised by the corresponding value of the reference (0% reduction) sample and plotted as a percentage.

The relative change of these variables cannot be fitted by a linear relation over all the range of applied cold reductions, but two stages can be identified. In the first of them up to about 10-15% rolling reduction, the variation of the variable is almost the same for Hc and Rm but Hv has a higher slope. During this first deformation stage, a large density of dislocations is generated, as observed by electron microscopy [11,18,19]. Beyond cold reductions about 15-20%, the experimental points follow a linear relation that is the same for Hc and Rm. Hv moves to higher values although it almost keeps the same slope. During this second deformation stage, which relates with the formation of dislocation cell structures [11,18], all these variables increase monotonically with the cold rolling reduction.

The correlation between the US longitudinal wave velocities and Hv is shown in Figure 5. The velocity increases initially to reach a value of 6020 m/s and after this, a continuous linear decrease is observed with increasing hardness. In agreement with this, it has been reported that the sound velocity increases with elongation at small deformations, and then decreases gradually with increasing strain [20]. The decrease in the sound velocity with increasing plastic deformation can be due to the formation of the texture, geometric size change and increase in the dislocation density.
3.6 Estimation of dislocation density

Given that, for both steels the hot rolled material before cold rolling was available, the Taylor’s equation was applied for estimating the dislocation density, $\rho$, of each of the cold rolled samples:

$$\rho = \left(\frac{\sigma - \sigma_{yo}}{\alpha M \mu b}\right)^2,$$

where $\sigma$ is the yield strength in each cold rolling condition, $\sigma_{yo}$, is the yield strength before cold rolling and the constants are $\alpha \approx 0.3$, the Taylor’s factor $M=3$, shear modulus for ferrite $\mu = 80000$ MPa and the Burgers vector for ferrite $b = 2.5 \times 10^{-10}$ m. It is observed that as the cold reduction increases, the steel becomes harder and the ductility impairs. For cold reductions above 21%, the ductility decrease makes the specimen to fail into the plastic deformation region, but below the 0.2% plastic strain commonly prescribed for defining the yield strength. Consequently, a prescribed plastic strain criterion of 0.1% was applied to all the curves. Another difficulty is that the tensile curves for the cold reduction lower than ~20% have a yield point plateau. For these cases, the extrapolated lower yield strength was determined. This correction is only relevant for the hot-rolled material prior to cold rolling because it moves down the yield strength about 35 MPa (which represents around 15% of the value), but after 12% cold rolling, the difference only represents about a 3%, which is negligible.

Applying the Taylor’s equation (Eq. 1) together with the experimental 0.1% yield strength before and after deformation and the indicated values for the constants leads to a dislocation density in the order of magnitude of $10^{14}$ m$^{-2}$, which is of the same range estimated in Ref. [11] for measurements made by transmission electron microscopy.
The $H_c$ values are represented as a function of the estimated dislocation density values in Figure 6, leading to a linear type of relation that can be expressed by the following equation:

$$H_c \ (\text{A/m}) = 288 + 6.5 \times 10^{-13} \rho$$  \hspace{1cm} (2)

where $\rho$ is given in $(\text{m}^{-2})$. In the same way, assuming the geometric factors of the sample can be neglected, the following relation holds between $VLW$ and the estimated dislocation density, at least for applied strains higher than 20%:

$$VLW \ (\text{m/s}) = 6046 - 1.25 \times 10^{-13} \rho$$  \hspace{1cm} (3)

Figure 6 shows a good linear correlation between $H_c$ and $\rho$ over a large range of cold reductions, significantly relevant for industry. Under these conditions, calculated dislocation densities increase monotonously with applied deformation within an order of magnitude that makes this variation not detectable by electrical resistivity measurements and produces a maximum variation of the ultrasound speed slightly higher than 1%. These results show that the magnetic techniques have a high sensitivity to cold rolling.

4. Conclusions

Parameters derived from magnetic and ultrasonic non-destructive measurement techniques are affected by the cold rolling reduction in a low carbon steel. However, the microstructural changes produced by the cold rolling deformation had no measurable effect (at least within the measurement error of 1%) on the electrical resistivity.

Magnetic coercive field, Vickers hardness and tensile strength increase monotonically in two stages that can be related with the generation of a large amount of dislocations at small strains and with the formation of dislocation cell structures as the cold reduction increases. The dislocation density has been calculated by Taylor’s equation based on the experimental yield strength values at 0.1% deformation. Linear relationships are found between the dislocation density and both the coercive field and the ultrasound longitudinal velocity. The former is monotonic and shows that $H_c$ increases linearly with $\rho$. The $V(LW)$ has a maximum at around 20% reduction and beyond this point decreases almost linearly with the cold reduction.
Acknowledgements

The research leading to these results has received funding from the European Union’s Research Fund for Coal and Steel (RFCS) research programme under grant agreement nr. RFSR-CT-2013-00031.

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