Orientation dependence of stored energy of cold work in semi-processed electrical steels after temper rolling

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Abstract

Microhardness measurements were carried out in a low carbon lamination steel after 6% of temper rolling, in order to evaluate local variations of work hardening as a function of crystallographic orientation. EBSD (electron back scattered diffraction) was used to determine grain orientations with respect to individual rolling planes and rolling directions. Hardness was shown to increase with the local Taylor factor. TEM observations and a well-known dislocation hardening model were used to confirm the equivalence between hardness values and the stored energy of cold work. A definite correlation between stored energy and Taylor factors could therefore be established, being more consistent than previous data reported in the literature. The improvement was thought to be related to the rather small plastic deformation, during which Taylor factors could be considered to remain constant.

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1. Introduction

Two different but directly related research topics were investigated in the present paper. From a technological point of view, the effect of grain orientation on the stored energy of cold work after temper rolling was analyzed in order to contribute to the understanding of texture formation during the final decarburizing and grain growth anneal applied commercially to non-oriented, semi-processed electrical steels. These steels are largely used as core material for rotating electrical machinery, representing an important commercial product for low-carbon cold-rolled sheet [1]. After annealing, the sheet may receive a so-called skin or temper-rolling pass between 5 and 10% reduction in thickness, after which it is sold to the customer as a "semi-processed" product. In this case, it is the manufacturer of the electrical equipment who, after cutting and/or punching, will apply a final decarburizing anneal that is also used to adjust the ferrite grain size for optimized magnetic properties. In addition, special steel compositions and processing conditions have been investigated in order to develop favourable textures which are well known in principle [2], but which remain difficult to be obtained in commercial practice [2,3].

From a scientific point of view, the relationship between stored energy and the Taylor factor was re-evaluated experimentally, using small amounts of plastic deformation in order to avoid the problem of grain rotations that are known to occur during larger deformations, and which have limited the extent of experimental confirmation of the importance of Taylor factors during previous investigations. It is generally accepted that stored energy is proportional to the amount of slip activity which, in polycrystalline materials, depends on grain orientation. The stored energy of cold work is therefore supposed to change from grain to grain according to the local crystallographic orientation as a function of applied stress. An important example can be found in the proper case of cold-rolled low-carbon steels for which, many years ago, the stored energy was shown to vary with the orientation of the rolling plane according to the sequence $E_{(1\overline{1}0)} > E_{(1\overline{1}1)} > E_{(2\overline{1}1)} > E_{(100)}$ [4,5]. Since stored energy represents the driving force for recrystallization, certain crystallographic orientations will be enhanced during annealing.
in comparison with others because of more favourable nucleation and/or growth kinetics. Measurements of stored energy, therefore, are directly related to the technologically important subject of controlling annealing textures. It should be realized, however, that the above specification of the rolling plane alone will only allow to work with average values of stored energy for a given set of grains because, despite of having a common rolling plane orientation, the grains may differ with respect to their rolling direction.

Many of the modern theories of plasticity are based upon the early concepts of Taylor [6] that state that the effect of crystal orientation on slip activity should be proportional to the Taylor factor, defined as

$$ M = \Sigma \delta \gamma_i / \delta \varepsilon $$

(1)

where $\delta \gamma_i$ stands for the amount of shear deformation on any one of the activated slip systems in a given grain, and $\delta \varepsilon$ represents the macroscopic plastic deformation applied externally. Thus, the stored energy of cold work has long been considered to be proportional to the Taylor factor which, for any individual grain, depends not only on the rolling plane but also on the rolling direction.

Previous attempts to confirm the proportionality between stored energy and Taylor factors had to use the effect of peak broadening on X-ray or neutron diffraction patterns [7,8] because, in the past, these techniques were the only ones which allowed to determine crystallographic orientation in statistically significant sample volumes. Today, electron back scatter diffraction (known as EBSD) in the scanning electron microscope (SEM) can be employed to determine the crystallographic orientation of a large number of individual grains on a polished sample surface and, in sequence, determine properties of these individual grains by any metallographic or surface microscopic technique. In fact, EBSD analysis was used recently not only to determine grain orientations after rolling, but also to measure stored energy as a function of orientation by reconstruction of the subgrain structure from EBSD data [9,10].

In the present paper, grain orientations were also determined by EBSD, while the amount of local plastic deformation was evaluated by microhardness measurements in selected ferrite grains taken in an optical microscope. It was assumed that the values of surface hardness should be at least as proportional to dislocation density, and therefore to stored energy, as the peak broadening effects that were used in the previous X-ray and neutron diffraction experiments, or the subgrain structure that was evaluated in the more recent EBSD analyses.

2. Experimental procedures

A commercial 0.07%C, 0.43%Mn, 0.08%Si, 0.011%S, 0.015%P, 0.047%Al and 0.0034%N steel sheet of 0.63 mm thickness, cold rolled from a 2 mm thickness hot rolled strip and batch annealed for 3h at 670°C, was cold rolled by a single pass to 6% reduction in thickness, representing a typical skin or temper rolling pass applied under industrial conditions. Longitudinal sections (containing the sheet normal and the rolling direction) were prepared for optical microscopy, EBSD and microhardness measurements. Thin-foil samples prepared parallel to the rolling plane were used for the observation of dislocation substructures in the transmission electron microscope (TEM). Carefully polished surface sections for EBSD were marked by hardness indentations and subsequently etched in a 2% nital solution to reveal the grain structure under the optical microscope, allowing easy access to the prior EBSD grain orientation data with respect to any individual ferrite grain that would be selected for a microhardness measurement. For the purpose of the present investigation, and taking advantage of the EBSD software package which allowed colouring of different rolling plane orientations, grain selection was based on (1 1 1), (1 1 0) and (1 0 0) rolling planes, in order to include orientations of both higher Taylor factors typical for (1 1 1) and some of the (1 1 0) rolling planes, and lower Taylor factors typical for (1 0 0) rolling planes. In addition, larger grains were selected preferentially because microhardness indentations placed in central regions of any given ferrite grain should then be less affected by the presence of nearby grain boundaries.

Taylor factors were calculated using the commercial TSL/OIM-3 software package for EBSD analysis based upon a zero-constraint Taylor model [11]. Microhardness measurements were performed under a load of 2 g. Thin foil samples for the TEM were electrolitically polished in a Struers Tenopol...
equipment using a standard 5% HClO₄ in CH₃COOH solution at about 12 °C and 40 V.

3. Results

Ferrite grain structures for the as-received (annealed) and the 6% cold-rolled samples are shown in Fig. 1. As expected, no significant modifications were introduced by the 6% rolling deformation on the scale of optical microscopy, and the grain size in both cases was found to be ASTM 9.5 or about 13 μm.

An example of the successful correlation between orientation and microhardness measurements in identical ferrite grains is shown in Fig. 2, where the same sample area was photographed using EBSD contrast in the SEM in Fig. 2(a), and metallographic contrast in the optical microscope in Fig. 2(b). It should be noticed that most of the ferrite grain boundaries were revealed by both EBSD and metallographic etching. In addition, the original shape of the grains as observed on the polished sample surface was maintained after nital etching, as can be seen by comparing regions A–D marked on Fig. 2(a) and (b).

Two other critical aspects had to be taken care of in order to obtain consistent levels of microhardness. Firstly, hardness measurements had to be restricted to larger ferrite grains, in order to minimize the hardening effect of near-by grain boundaries. Secondly, hardness measurements had to be applied rather shortly after sample preparation (within a few hours). This time limit was necessary to avoid surface oxidation that was found to increase the low-load microhardness values. Only after such precautions had been taken were consistent microhardness data obtained for different samples, representing either the annealed or the temper-rolled condition. Results are shown in Fig. 3 where, in Fig. 3(a), the hardness after 6% cold rolling can be seen to increase rather clearly with increasing Taylor factors while, in Fig. 3(b), no effect of the Taylor factor was encountered for the previously annealed sample.

Typical dislocation substructures that are supposed to be responsible for both stored energy and hardness values after temper rolling are shown in Fig. 4. Grain orientations were determined from selected area electron diffraction patterns taken at zero tilt angle, and by placing the thin foil into the sample holder with the original rolling direction aligned parallel to the tilt axis. Dislocation densities in the range of $1 \times 10^{10}$ cm$^{-2}$ were measured in several sample areas where grain boundary extinction fringes under dynamical two-beam conditions allowed to determine the local sample thickness, but the number of such areas was not sufficient to establish any quantitative and statistically meaningful correlation with the Taylor factors comparable to the hardness data presented in Fig. 3. On the other hand, visual inspection of the dislocation distributions photographed in different grains under similar contrast conditions (two-beam contrast using 110 reflections) suggested a consistent effect of the Taylor factor on substructure evolution: From simple dislocation tangling for lower Taylor factors, as shown in Fig. 4(a), to the formation of subgrain boundaries for higher Taylor factors, as shown by the local coincidence of dislocation cell walls with some prominent TEM bend contours in Fig. 4(b).

4. Discussion

Taylor factors continue to be important input parameters for many of the present theories of plasticity [6,12–15], and are routinely used to calculate individual grain rotations during plastic deformation when trying to predict deformation textures [16–18]. Such predictions, however, have met with only partial success in the past [18–20], and the real importance of Taylor factors in trying to predict texture could therefore be questioned [20]. On the other hand, the understanding of annealing textures is invariably based upon the orientation dependence of stored energy [21–23]. In fact, many authors have tried to explain the most common industrial annealing textures by nucleation...
or growth advantages that orientations of both higher or lower Taylor factors may encounter as a function of particular recrystallization mechanisms [24, 25]. It is therefore important to have an experimental confirmation of the otherwise only theoretical argument [20] that the stored energy of cold work should depend upon the Taylor factor.

Two papers have been published previously that tried to obtain this experimental confirmation [7, 8], but both dedicated to stored energy measurements after relatively large deformations (50% cold rolling of copper and 80% cold rolling of interstitial-free steel, respectively). In both cases, the very large scatter of the experimental data must have left serious doubts about the real effect of Taylor factors on stored energy. However, as stated by the proper authors of one of those papers [7], grain rotations known to occur during large deformations must have carried many individual grains through orientations that have different Taylor factors. Thus, at the moment of measurement (after large deformations), the total slip activity of any given region may have been affected more by grain rotation than by either the initial or the final Taylor factor.

In the present case of small deformations, on the other hand, Taylor factors had a much better chance to exhibit their real effect upon stored energy. The data presented in Fig. 3 should therefore be accepted as new and improved experimental confirmation of an important part of the Taylor theory of plastic deformation.

While dislocation substructures as observed in the TEM should be able to yield the most direct evidence of the local stored energy, and in fact have been used for this purpose in one of the classic papers on this subject [4], TEM observations in the present case have clearly shown some typical problems associated with this approach. In addition to the well-known difficulty of the TEM in providing quantitative information of statistical relevance, measurements of dislocation densities based upon the method of line intercepts were noted to become uncertain as soon as dislocations started to agglomerate first into tangles and thereafter into cell walls. Microhardness measurements, with their averaging potential over selected volumes of a given ferrite grain (to be selected by adjusting the test load to the grain size) should therefore be recommended as the more reliable method.

Despite of the difficulties with respect to quantification, the present observations of dislocation substructures are in good agreement with previous investigations. Thus, dislocation densities of about $1 \times 10^{10} \text{cm}^{-2}$ after 6% cold rolling compare favourably with the early observations by Keh and Weissman [26]. In addition, the initial transition of dislocation tangles to sub-boundary formation was observed for similar amounts of plastic deformation ($\varepsilon \geq 6\%$ in tension) in a more recent investigation [27].

In addition, the average level of work hardening of about $30 \text{HV}$ after 6% cold rolling is very much in accordance with previous relationships between dislocation densities and mechanical properties published in the early literature. From the work of Keh [28], the effect of dislocation density on yield strength in iron alloys can be quantified as

$$\Delta\sigma = m\beta \rho^{1/2}$$

where $\Delta\sigma$ is the dislocation contribution to yield strength, $m$ the average Taylor factor for polycrystals, $\rho$ a geometrical factor
that depends upon the type of dislocation interaction, $\mu$ the shear modulus (80,300 MPa in ferrite), $b$ the dislocation Burgers vector (0.25 nm in ferrite), and $\rho$ the measured dislocation density.

A value of $m(\alpha)= 0.38$ has been determined experimentally for pure iron [26]. Thus, for a dislocation density of $1 \times 10^{10} \text{ cm}^{-2}$, Eq. (2) would predict a yield strength increase of 76 MPa.

According to Cahoon et al. [29], yield strength of carbon steel may be related to hardness by

$$\sigma_y = \left(\frac{HV}{3000}\right)(0.1)^m - 2 \tag{3}$$

where $\sigma_y$ is the yield strength in kgf/mm$^2$, $HV$ the Vickers hardness number, and $m$ the Meyer’s hardness coefficient which, for the present steel, was determined experimentally by putting into Eq. (3) both the yield strength (271 MPa or 27.6 kgf/mm$^2$) and the macrohardness (102 $HV$) taken from the original as-received sheet, giving a value of $m = 2.10$. Eqs. (2) and (3) would therefore predict an increase in hardness of about 28 $HV$ for dislocation densities of $1 \times 10^{10} \text{ cm}^{-2}$, in excellent agreement with the experimentally observed 22–35 $HV$ from Fig. 3.

5. Conclusions

From the experimental results discussed above, the following conclusions may be drawn:

- In slightly rolled low carbon steels, the stored energy of cold work has been shown to increase in direct proportion to the Taylor factor.
- Microhardness measurements in individual ferrite grains may be taken as representative of local dislocation densities.

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