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Dynamic recovery and recrystallization in a cold rolled ultra low carbon steel plate: a neutron and X-ray diffraction study

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The cold rolling of ultra low carbon steel is a complex process involving the formation of texture and the development of residual stresses. The mechanism for both texture and residual stress development is dislocation multiplication. Both texture and residual stresses can be determined by means of neutron and X-ray diffraction. In this paper, results of such measurements on a cold rolled plate (final thickness 10 mm) are presented along with theoretical predictions based on finite element calculations of the residual stress state. The results show that during cold rolling not only the formation and multiplication of dislocations play a role. It is seen that besides the creation of dislocations, two dislocation annihilation processes are involved, namely recovery and recrystallization. These happen despite the fact that the cold rolling takes place at relatively low temperatures ($20 < T < 150\,^{\circ}C$).

1. Introduction

The cold rolling process is characterized by complex and non-uniform plastic deformation. As a result of cold rolling, residual stresses develop in the material [1, 2]. Both the elastic and plastic behaviour of iron are to some extent anisotropic. Therefore, besides developing residual stresses, a cold rolled piece of iron will also possess a certain texture. Texture is characterized by the existence of preferred orientations in a polycrystalline material. The presence of texture imposes difficulties on stress measurements by means of diffraction techniques. Diffraction techniques are used to determine changes in lattice spacings, which are then converted to stresses using the laws of elasticity. However, the elastic behaviour of a textured material will be somewhere between that of a single crystal and a randomly oriented polycrystalline material. A strong texture usually develops after severe cold forming, inducing several hundreds of per cents of plastic deformation, e.g. in cold rolling of thin sheets and wire drawing to small sizes.

The formation of dislocations and their glide mechanisms are responsible for the development of texture as well as residual stresses. The formation of dislocations, however, also has the effect of strengthening the cold worked product. This work hardening increases with increasing plastic deformation.

Work hardening is known to disappear continuously during hot working of materials. The processes involved are dynamic recovery and recrystallization, where the latter takes place at higher temperatures than the former. The temperatures at which both dynamic recovery and recrystallization may take place decrease with increasing dislocation density due to increasing plastic deformation. During cold working (rolling, wire drawing etc.), an appreciable amount of heat is dissipated due to the internal friction within the material. This causes the temperature to rise as the heat cannot be extracted from the material immediately. It was shown by Aernoudt [3] and Denis *et al.* [4] that during wire drawing at room temperature, the combined effect of high dislocation density and temperature elevation can cause both dynamic recovery and recrystallization in the drawn material.

During cold rolling, the dislocation density at the surface region is higher than that at the interior of the plate. But the surface temperature will be somewhat lower than that at some depth below, due to the fast cooling at the surface. So dynamic recovery and/or recrystallization will be favourable at a region below

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the surface for a specific combination of temperature and deformation. If dynamic recovery or recrystallization takes place, they can have a distinct effect on the measured stress state as well as on the breadth of diffraction peaks measured as a function of the depth below the surface.

The influence of dynamic recovery and recrystallization on the texture of the rolled material will be different. While the dynamic recovery will give rise to a polygonization in the individual crystallites only, the recrystallization will actually form completely new crystallites through a nucleation and growth process. Therefore unlike recovery, the recrystallization may lead to a texture that is different from the non-recrystallized material.

In this paper, an analysis of the residual stress state of a cold rolled ultra low carbon steel (ULC) plate determined by means of X-ray and neutron diffraction are presented along with results of texture measurements as a function of depth. These results are compared to those from finite element calculations. The results indicate that dynamic recovery as well as recrystallization do play a role.

2. Experimental details

2.1. Specimen preparation

The cold rolled specimen was produced at the laboratories of Hoogovens Steel Works, IJmuiden, The Netherlands. The starting material was a ULC steel cube of 100 mm (volume 1 dm^3), with a chemical composition as shown in Table I. The steel was hot rolled in steps at about 640 °C to an intermediate thickness of 20 mm. After cooling and surface cleaning, the plate was cold rolled to a final thickness of 10 mm. The reduction scheme is presented in Table II. It has to be mentioned here that the time between successive rolling passes was short (a few seconds) compared to the time needed for complete cooling down to room temperature, which would be in the order of 20 to 30 minutes.

The final plate, having a length of about 1 metre, was used to produce several test specimens. A piece of 250 mm length and 110 mm breadth was cut for stress measurement purposes. The remaining part was used for the texture measurements.

The samples for texture analyses using neutrons were prepared in a special way. From the cold rolled plate, 1 mm thick slices were cut in planes parallel to the rolling plane using electric discharge machining. In this way slices were obtained representing different depths below the surface. (namely 0–1 mm, 1–2 mm etc.). These slices, with dimensions of about 100 mm × 20 mm × 1 mm, were further cut to strips of 100 mm × 9 mm × 1 mm, which were then cut to squares of 9 mm × 9 mm × 1 mm. During the entire cutting process, care was taken to note the rolling direction and depth origin of each slice. Finally, the little squares belonging to the same depth below the surface of the original specimen were stacked together to form small cubes, making sure that their individual (rolling direction) orientations were maintained parallel. In this way 10 specimens were prepared, so that the texture as a function of the depth below the surface with a depth resolution of 1 mm could be analysed.

TABLE I Chemical composition of the ULC steel and the high carbon steel used in the present experiments

Material	C	Si	Mn	Cr	V	Al
ULC	0.002	0.012	0.195	0.016	–	0.058
90 Mn V8	0.9	0.25	2.0	0.35	0.10	–

TABLE II Sequence for the cold rolling process on ULC steel. The diameter of the applied rolls was 228.6 mm

Pass number	Thickness after pass (mm)
0	19.8
1	19.5
2	18.9
3	18.4
4	17.9
5	17.1
6	16.5
7	16.0
8	15.3
9	14.7
10	13.8
11	13.1
12	12.3
13	11.6
14	11.0
15	10.3
16	9.9

2.2. Measurement and calculation techniques

2.2.1. Neutron stress measurements

If we assume the orientation of the principal axis system to be parallel to the rolling axis system of rolled plate, the stress state can be proved to be a biaxial one throughout the thickness of the plate. This can be understood from the fundamental laws of mechanics in the following way. All shear stresses are equal to zero in the specimen axis system (i.e. the principal axis system), hence the shear stress gradient in the direction perpendicular to the plate also equals zero. Now the stress perpendicular to the plate, σ_z, is proportional to the shear stress gradient and at the free surface $\sigma_z = 0$. Since at the surface the shear stress gradient equals zero, σ_z can never reach any value other than zero throughout the thickness of the plate. This reasoning applies for any flat plate for which one can be certain that the principal axes follow the main dimensions of the plate (when the principal axis system is rotated as a function of depth, this reasoning will not apply).

Determination of the biaxial stress state was done through two series of mutually perpendicular $\sin^2\psi$ stress measurements. During the first set, the rolling direction is kept parallel to the diffractometer axis,

while during the other, the rolling direction is maintained perpendicular to the diffractometer axis such that the diffractometer axis is parallel to the plane surface of the plate.

As the thickness of the rolled plate is 10 mm and the rolling process is expected to produce a symmetrical residual stress pattern, we have performed measurements only up to half the thickness of the plate. The depths at which measurements were performed range from 0.5 to 5 mm in steps of 0.5 mm. The applied prismatic gauge volume was about 1 mm × 1 mm × 20 mm, the 20 mm dimension oriented along the diffractometer axis. The ψ-values at which the diffraction peaks were scanned are in both cases: 0°, 30°, 60° and 90°. The scan results were used as input for a data reduction procedure, resulting in the longitudinal stress σ_l, the transverse stress σ_t and the stress free lattice parameter d_0. For this analysis the macroscopic elastic constants for iron were used; i.e. $E = 210\,000$ MPa and $v = 0.28$.

2.2.2. Neutron texture measurements

For the texture measurements a single crystal diffractometer operated in a texture goniometer mode was used.

The principle of a texture measurement is simple: the specimen is rotated around two axes in a step-wise manner. One axis is perpendicular to the surface of the specimen (the φ-axis), the other inclines the φ-axis from a fully vertical to a fully horizontal position in the diffraction geometry (the χ-axis). For each position in this two-dimensional orientation space, the intensity of one of the available crystallographic reflections (here: the Fe 1 1 0 direction) was measured by counting during the time needed for a fixed number of neutrons to pass by a monitor which is situated in the primary beam.

The representation of the measurement results is in the form of pole figures, according to standard procedures (Bunge [5]).

A more traditional way of determining texture pole figures is by X-ray diffraction. The advantage of neutron diffraction for texture analysis over X-ray diffraction is twofold:

1. The full φ-χ space can be scanned without the occurrence of a grazing angle geometry which is encountered with X-ray diffraction for $\chi > \approx 75°$.
2. As the irradiated sample volume is larger during neutron measurements, a better volume average can be obtained than with X-ray diffraction.

The second aspect is important as later we will compare the texture state of a 1 mm thick layer with residual stress measurements at a comparable spatial resolution (1.2 mm).

2.2.3. X-ray stress measurements

By using a portable Rigaku system, standard X-ray $\sin^2\psi$ measurements were performed as a function of the depth in the rolled plate. After successive removal of material by electrochemical polishing, the stress measurements were performed in two mutually perpendicular directions. On the surface at each stage in this way, the stresses σ_l and σ_t and the stress free lattice parameter d_0 as a function of depth were obtained. The stress results have been corrected for the relaxation due to the removal of material during electrochemical polishing, according to Moore and Evans [6]. The elastic constants used for the calculation of the stresses from the measured d-values were the same as those used for the neutron measurements.

2.2.4. Finite element calculations

The cold rolling process was simulated by means of a finite element method. This method can be characterized as a numerical way to solve a (complex) set of (partial) differential equations. These equations result from physical conservation laws (conservation of mass, momentum, energy etc.). In addition to these general laws, a mathematical description of the material – the constitutional equations – is required. The description of the materials used in the present study is based on a continuum theory for large elastic-plastic deformations. Thermal effects like expansion and phase transformations are also included.

A detailed description of the method is beyond the scope of this paper; however, it can be found in Huétink [7]. The simulation program used is called DIEKA and was developed at Twente University; the simulations were performed at Hoogovens, IJmuiden [8].

3. Results

In Fig. 1 the results for the longitudinal stress σ_l and the transverse stress σ_t as a function of the depth are given. In Fig. 2, the X-ray and neutron results for the stress free lattice distance, which are based on an assumption of a biaxial stress state, are given ($\sigma_z = 0$). In Fig. 3 the integral breadths of the X-ray diffraction peaks at $\varphi = 0°$ obtained in the transverse measurements are given.

The results of the texture measurements are presented in Figs 4 and 5 in the form of 10 (1 1 0)-pole diagrams each representing the texture at a specific depth interval. The figures indicate intensity contour lines, which represent different levels of diffracted intensity (or in other words the sharpness of the texture).

4. Discussion

From Fig. 2 it is clear that for both methods, the obtained value of the stress free lattice parameter d_0 does not change significantly with depth. For the X-ray measurement this can be expected because of the surface measurement character of this method. With the neutron measurements d_0, as obtained from an analysis of the measurement results on the assumption of a biaxial stress state, does not change with depth. This supports the correctness of the assumption that the stress state in the rolled plate is indeed biaxial. If the actual stress state had not been biaxial, this would have been seen from a varying d_0 with depth, thus proving the incorrectness of the biaxiality assumption.

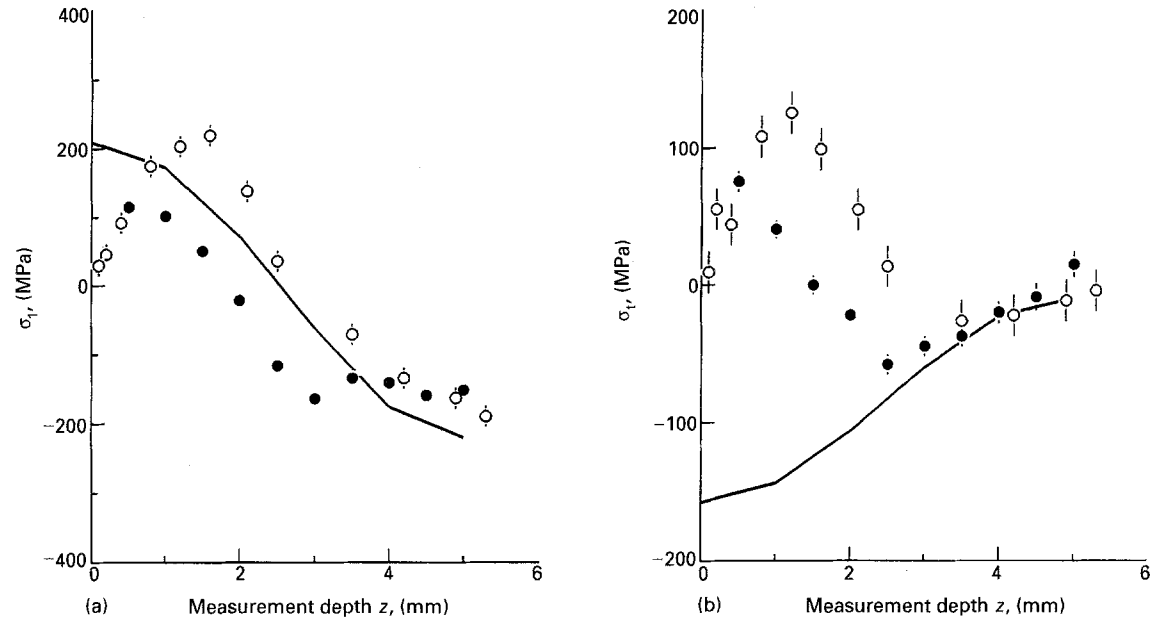

Figure 1 Stress determination results for (a) the stress in rolling direction σ_l and (b) the stress in transverse direction σ_t. The open symbols represent X-ray measurements, the closed symbols represent neutron measurements. The drawn lines are the result of finite element calculations.

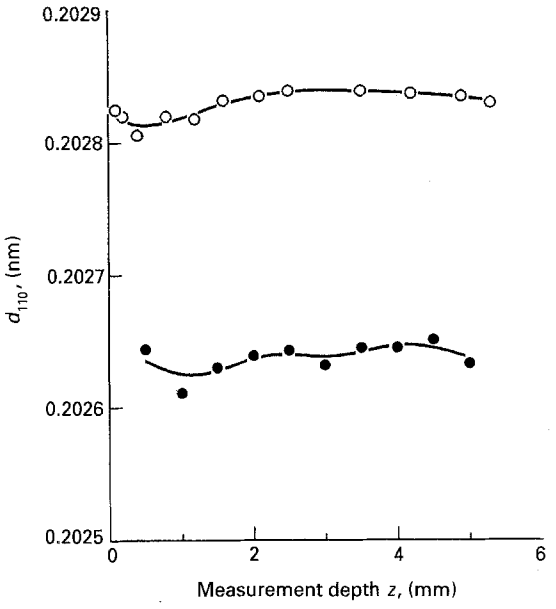

Figure 2 Values for the stress free lattice parameter d_0 as found by neutron and X-ray measurements. The open symbols represent X-ray measurements, the closed symbols are neutron values. The X-ray results, obtained using the (2 1 1)-reflection of iron, have been scaled to (1 1 0)-values. The drawn lines serve as guides to the eye.

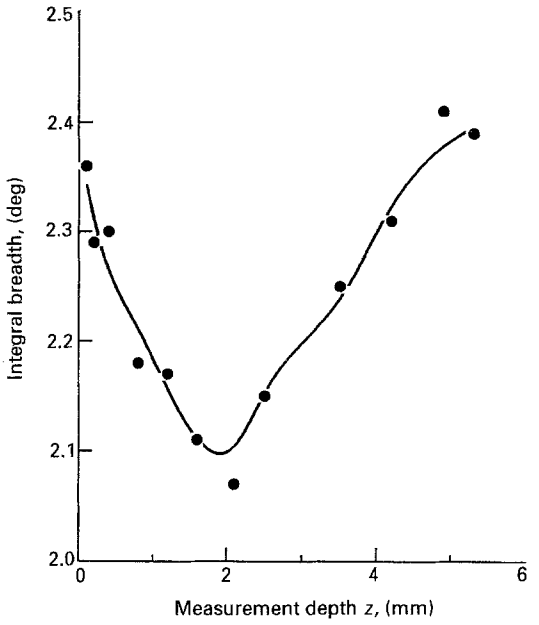

Figure 3 The integral breadth of the X-ray diffraction peaks at $\psi = 0°$, obtained on a rolled ULC cold rolled strip as a function of the depth below the surface. The solid curve serves as a guide to the eye.

In order to be able to compare the d_0-values as obtained by X-rays and neutrons, the X-ray values of Fig. 2, which have been obtained from the (2 1 1) reflection of iron, have been recalculated to the (1 1 0) values. The difference between the mean d_0-value of both methods may be attributed to the use of different instruments for these methods with different systematic errors associated with them. For stress measurements, the systematic error of the diffraction peak is of little importance as long as it is consistent for all the peaks involved in a stress measurement.

The stress measurement results presented in Fig. 1 show a reasonable agreement between the X-ray and neutron measurements. The results, however, suggest that perhaps some disturbance with the positioning of the sample in the neutron beam geometry might have occurred. When the neutron results curve is shifted by an amount corresponding to 1 mm, the neutron and X-ray result curves almost coincide. According to all the checks made, a misplacement could not have been the case. However, it cannot be totally excluded as it might have happened during operation.

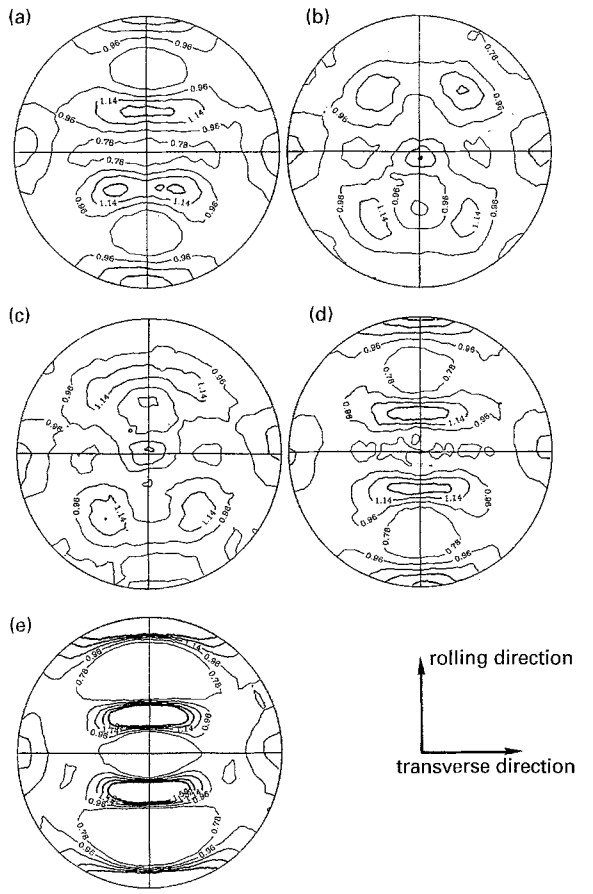

Figure 4 (1 1 0)-Pole figures of the samples taken from the cold rolled ULC strip at different depths below the surface (*cf.* Fig. 5). (a) $z = 0$–1 mm, level 0.78; (b) $z = 1$–2 mm, level 0.96; (c) $z = 2$–3 mm, level 1.14; (d) $z = 3$–4 mm, level 1.32; (e) $Z = 4$–5 mm, level 1.50.

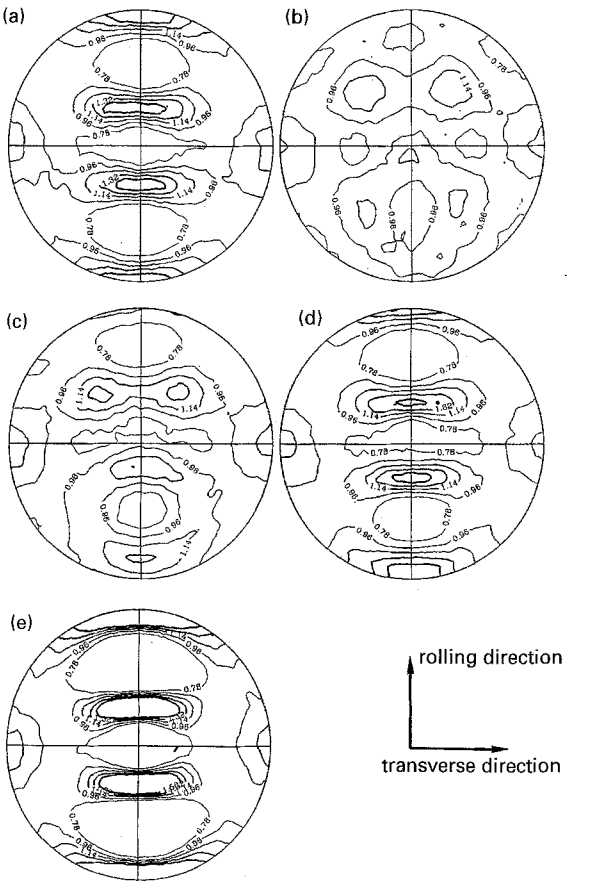

Figure 5 (1 1 0)-Pole figures of the samples taken from the cold rolled ULC strip at different depths below the surface (*cf.* Fig. 4). (a) $z = 9$–10 mm, level 0.78; (b) $z = 8$–9 mm, level 0.96; (c) $z = 7$–8 mm, level 1.14; (d) $z = 6$–7 mm, level 1.32; (e) $z = 5$–6 mm, level 1.50.

As mentioned in the introduction, the presence of a texture influences the determination of stress values from measured strain data, because the theory of elasticity involved in this is still not adequately solved. At this stage of the development of the neutron stress measurement technique, however, there are several problems to be solved which are related to the accurate determination of the lattice spacing itself. These problems should be solved before the elasticity-related texture problem can be tackled. As this holds for X-ray and neutron measurements it cannot explain their difference.

In Fig. 1, besides measurements of the stress state, finite element simulations based on the same rolling process are presented. For the case of σ_l there is a reasonable agreement for the bulk region, whereas in the case of σ_t there is no agreement at all. The finite element program DIEKA used in these simulations has been developed for modelling the cold rolling of thin sheet. When thin sheet is rolled, the material transport in the transverse direction is very limited due to a larger friction in the direction of the roll axes. In the present case, there has been about 10% net plastic strain in the transverse direction. This has not been accounted for in the simulations which must be the reason for the observed discrepancies between measurement and simulation for σ_t. The agreement for σ_l is good except for the near surface region where some discrepancies between measurement and calculations exist. There may be three reasons for this.

1. The finite element mesh used for the calculations consists of only five elements representing the depth direction. This cannot provide sufficient resolution for the very near surface region.

2. The residual stress state at the surface depends on the friction that exists between the rolls and the material during rolling. Therefore it depends strongly on the way this friction behaviour is modelled.

3. Dynamic recovery and recrystallization have taken place and the finite element calculations have not taken this into account.

When we compare the stress situation across the plate thickness (Fig. 1) and the corresponding profile-breadth changes (Fig. 3), obtained with X-rays, we notice that up to a depth of about 2 mm from the surface the residual stresses (both σ_l and σ_t) increase and then start decreasing in magnitude. On the other hand, the profile-breadth variations show an opposite trend. The fact that these trends show a maximum and a minimum for stress and profile-breadth, respectively, at a depth of 2 mm suggests that they are related to each other and their origin is the same.

If we assume the plastic deformation to be uniform and ignore a possible dynamic recovery and recrystall-

ization for the time being, then the profile-breadth should be nearly constant over the cross-section. On the other hand, as the deformation is highest at the plate surface, and decreases with depth, the breadth variations should show a minimum at the centre. The results of Fig. 3 do not support either of these situations. The presence of a minimum indicates a sharpening of the diffraction profile, which can only happen if there is dynamic recovery and/or recrystallization resulting in a reduction in microstrain and/or an increase in domain size. As mentioned before, the extent of the recovery and recrystallization will directly depend on the amount of deformation and the rise in temperature. Since the breadth of a diffraction profile is sensitive to microstructural changes (and not influenced by macrostresses), from the results of Fig. 3 we may thus conclude that the deformation and the temperature give the highest effect with regard to dynamic recovery and recrystallization at a depth of about 2 mm from the surface.

The texture results of Figs 4 and 5 strongly support the occurrence of localized recrystallization in the rolled plate. In the region around 2 mm below the surface (i.e. where the integral breadth from Fig. 3 reaches a minimum), the texture is completely different from the texture at both the surface and the centre of the plate. This cannot be due to dynamic recovery, but must be the result of partial or total recrystallization. The fact that the macrostresses (i.e. both longitudinal and transversal, see Fig. 1) reach a maximum value at 2 mm below the surface can be well understood if recrystallization has taken place. Owing to recrystallization, the density of the material decreases, so during recrystallization the material has a tendency to shrink. However, the recrystallized material is under constraint from both the central part and the near surface material of the plate, which have not recrystallized because the combination of the local dislocation density and temperature does not lead to recrystallization. These constraints can result in the formation of tensile stresses in the recrystallized material in both longitudinal and transverse directions. This also explains the noticeable differences between measurements and finite element calculations.

Conclusions

Based on the texture and stress measurements on a 10 mm thick ultra low carbon steel plate, it appears that simultaneous recrystallization has occurred during the cold rolling of the plate.

From the calculation of the stress free lattice parameters from neutron diffraction data through the thickness of the cold rolled ULC steel plate it can be said that the stress state of the cold rolled plate is a biaxial one.

A finite element model for the calculation of residual stresses that applies to a thin plate cannot be applied directly to a thick plate, because the plastic flow in the direction perpendicular to the rolling direction is neglected in the case of thin plate.

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