Analysis of Preferred Orientations in Duplex Chromium–Nickel Steels

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The textures and orientation correlations of grain and phase boundaries were studied in a two-phase stainless steel after hot rolling, annealing, and plastic deformation. It was concluded that the hot-rolled state is partly recrystallized, and that recrystallization is completed after an additional annealing at 1030 °C.

During plastic deformation of the hot-rolled material, both the harder austenite and the softer ferrite participate in the deformation. During deformation at 45° to the rolling direction, rigid rotation of the phase particles may additionally contribute to textural changes. The preferred occurrence of low-angle boundaries can be attributed to polygonization prior to recrystallization. Further preferred boundaries are attributed to twinning and oriented γ → α transformation.

Introduction

The physical properties of polycrystalline materials depend on the crystallographic anisotropy of the corresponding property and the orientation distribution function of the crystallites in the material. In steels, the mechanical properties are particularly interesting. Dual-phase steels consist of fcc austenite and bcc ferrite. Hence, their macroscopic mechanical properties depend on the orientation distribution functions (ODF) of the crystallites in both phases. Furthermore, the respective arrangement of the phases plays a role. This latter feature expresses itself in the shape of austenite and ferrite particles and their spatial distribution. Furthermore, the mutual orientation relationship of α and γ crystals across the phase boundary, i.e. the misorientation distribution function (MODF), influences the mutual interaction of the two phases. Finally, the interphase MODF must be compared with the two intraphase MODFs, i.e. the misorientation distribution functions of α-crystals and γ-crystals across their respective grain boundaries.

The ODF of both phases can be measured by X-ray textural analysis. The shape and arrangement of α and γ particles can be seen by optical metallography. These are two well-established techniques by now. The misorientation distribution across grain or phase boundaries can be determined only by measurement of the individual orientations of pairs of crystals adjacent to the boundaries. With respect to the small crystallite size, this usually requires electron diffraction.

The study of the 'textural quantities' ODF and MODF provides a good insight into the processes that have led to the formation of the microstructure of a material, particularly plastic deformation during hot and cold rolling, as well as α → γ transformations in the course of the heating path. Finally, the ODF, i.e. the 'texture' in the classical sense, is the most important structural parameter governing the plastic anisotropy of a material.

Hence, in the present paper the two textures (ODF) and three MODF in α-γ duplex steels were determined by X-ray and electron diffraction respectively in the different material states.

Material and Experimental Procedure

The investigations were carried out in the duplex steel X2 CrNiMoN22 5 (REMANIT 4462 Thyssen Edelstahlwerke). Its chemical composition is given in Table I. The material was hot-rolled 66 per cent with intermediate annealings at 1120 °C for 4 to 6 minutes after 30, 19, and 41 per cent partial reduction respectively. After that treatment, the material consisted of about 60 per cent austenite and 40 per cent ferrite in an elongated arrangement. The material turned out to be partially recrystallized.

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Mo</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.024</td>
<td>0.05</td>
<td>1.66</td>
<td>0.023</td>
<td>0.003</td>
<td>22.16</td>
<td>3.18</td>
</tr>
</tbody>
</table>

The hot-rolled material was further annealed for 30 minutes at 1030 °C and water-quenched. After that it was aged for 3 hours at 850 °C and 470 °C respectively. These two temperatures correspond to embrittlement ranges reported in the literature.

Furthermore, the influence of 20 per cent plastic deformation by tensile test in the original hot-rolling direction, as well as under 45 and 90° to this direction, was studied. Texture measurements were carried out by X-ray diffraction using the automatic texture diffractometer ATEMA-C.
and CoKα radiation in the back-reflection technique. In this way, three pole distribution functions of each phase were measured, i.e. (200) (220) (311) for austenite and (200) (211) (220) for ferrite. The measurements were done in angular steps of \( \Delta \alpha = 5^\circ \) and \( \Delta \beta = 3.6^\circ \) up to \( \alpha_{\text{max}} = 75^\circ \).

The individual orientation measurements were made by electron diffraction using the microscope Philips EM 430 with 300 keV. Transmission samples were prepared perpendicular to the transverse direction. Individual crystallite orientations were determined with the help of Kikuchi patterns. The patterns were evaluated directly on the microscope screen by use of the deflection coils, as described by Weiland and Schwarzer. The results of these measurements are individual crystal orientations, together with the microstructural images of the grains.

### Representation of Distribution Functions

In order to represent the crystal orientations, the crystal coordinate system \( K_A \) of each crystallite (e.g. its cubic axes) has to be referred to the macroscopic reference system (e.g. rolling, transverse, and normal direction), as shown in Figure 1. This requires at least three angular parameters to be specified, e.g. the Euler-angles \( \varphi_1 \varphi_2 \varphi_3 \) shown in Figure 2b. Alternatively, the crystallographic plane (hkl) parallel to the rolling plane and the direction [uvw] parallel to the rolling direction can be specified as shown in Figure 2a. For continuous orientation distribution functions, the representation by Euler angles is preferable. The texture is defined by the volume fraction of crystallites in the orientation \( g \):

\[
\frac{dV}{d\gamma} = f(g) = f(\varphi_1 \varphi_2 \varphi_3). \quad dg = \frac{1}{8\pi^2} \sin \varphi_1 d\varphi_1 d\varphi_2 d\varphi_3 \tag{1}
\]

\( K_A \longrightarrow g \longrightarrow K_B \)

This distribution function cannot be measured directly. It must be calculated from the measurable pole distribution functions of particular lattice planes (hkl):

\[
\frac{dV}{d\Omega} = P_{(hkl)}(\alpha \beta) = \frac{1}{2\pi} \int f(\varphi_1 \varphi_2 \varphi_3) d\gamma. \tag{2}
\]

Since X-ray diffraction does not ‘see’ a rotation of crystallite about the normal direction to the reflecting lattice plane, the pole distribution functions are integrals over the complete orientation distribution function \( f(g) \), as given in the right side of equation [2]. The function \( f(g) \) can be calculated from several experimentally measured pole density functions (e.g. (200)(220)(311) for austenite as mentioned above), thereby using a series expansion method. The result is expressed in multiples of the random orientation distribution. Lines of constant orientation density are shown in the sections of \( \varphi_1 = \text{const} \) respectively \( \varphi_2 = \text{const} \). The first kind of representation is more convenient for ferrite, the latter for austenite. It is to be mentioned that the ODF \( f(g) \) in equation [1] must be invariant with respect to all crystal symmetry rotations (i.e. the elements of cubic crystal symmetry). Furthermore, a sheet has macroscopically an orthorhombic sample symmetry (i.e. two-fold rotation axes in RD, TD, ND). All these symmetries are taken fully into consideration in the calculated ODF.

Except by X-ray diffraction, textures can also be determined from measurements of individual grain orientations, e.g. by electron diffraction.

The misorientation across a grain of phase boundary is illustrated in Figure 3. For a single boundary it is expressed by the rotation \( \Delta g \), which in turn may also be expressed by three Euler angles as the orientation \( g \). The misorientation distribution function (MODF) is then defined by the area fraction of boundaries having the misorientation \( \Delta g \):

\[
\frac{dV}{d\Delta g} = F(\Delta g) = F(\varphi_1 \varphi_2 \varphi_3). \tag{3}
\]

\( K_A \longrightarrow g \longrightarrow K_B \)

\( K_A \longrightarrow K_B^1 \longrightarrow \Delta g \longrightarrow K_B^2 \)

FIGURE 3. Misorientation distribution of crystal pairs across grain or phase boundaries
If crystallites that have the texture $f(g)$ are randomly distributed in space, then a particular MODF, $F_d(\Delta g)$, the uncorrelated MODF, is created, which can be calculated from the texture function by the self-convolution integral

$$F_d(\Delta g) = f(\Delta g) * f(g)dg.$$  \[4\]

If, however, particular types of boundaries, e.g. low-angle boundaries, are preferred, then the crystallites will develop another kind of neighborhood that expresses itself in an MODF in equation [3] different from that in equation [4]. In that case, the orientation correlation function\(^5\) is defined by the ratio of the two MODFs:

$$\rho(\Delta g) = \frac{F(\Delta g)}{F_d(\Delta g)}.$$  \[5\]

In the case of MODF, both the crystals forming a boundary have crystal symmetry, e.g. cubic symmetry. Hence, the MODF must be invariant with respect to all these symmetries. This is taken fully into account in the distribution functions.

MODF and correlation functions can be obtained only from individual orientation measurements of a large number of crystallites, e.g. by electron diffraction.

In a two-phase material as studied in this investigation, a texture function $f(g)$ has to be specified for each phase and an MODF for each type of boundary, i.e.

$$f^a(g), f^\gamma(g), F^a(\Delta g), F^\gamma(\Delta g), F_{\rho a}(\Delta g), F_{\rho \gamma}(\Delta g).$$  \[6\]

**Results**

The phase and grain structure of the hot-rolled state is shown in Figure 4, which is an optical micrograph taken parallel to the rolling plane.

![Optical micrograph of the investigated two-phase steel (parallel to the rolling plane)](image)

The textures of both phases obtained from X-ray diffraction are shown in Figure 5 in $\phi_1$ and $\phi_2$ sections respectively as mentioned above.

Figure 6 contains all six functions given in equation [6] determined from individual orientation measurements in the electron microscope. In this figure, only two sections, i.e. $\phi_2 = 0^\circ$ and $\phi_2 = 45^\circ$, are shown for each distribution function.

The textural changes after annealing are summarized in Figure 7, which shows sections through the ODF for $\phi_1 = 0^\circ, \phi_2 = 45^\circ$, and $\phi_2 = 0^\circ, \phi = 45^\circ$ respectively.

![Texture changes after various heat treatments](image)
Finally, Figure 8 gives the textural changes after 20 per cent plastic tensile deformation in the rolling direction, as well as under 45° and 90°.

The mechanical properties, i.e. yield stress, tensile stress, elongation, and r-value are given in Table II.

TABLE II
MECHANICAL PROPERTIES

<table>
<thead>
<tr>
<th>Sample direction</th>
<th>0.2% proof stress [MN/m²]</th>
<th>Tensile strength [MN/m²]</th>
<th>Elongation [%]</th>
<th>r-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rolling</td>
<td>551</td>
<td>750</td>
<td>29.0</td>
<td>0.45</td>
</tr>
<tr>
<td>45°</td>
<td>579</td>
<td>750</td>
<td>29.8</td>
<td>0.97</td>
</tr>
<tr>
<td>Transverse</td>
<td>636</td>
<td>820</td>
<td>27.5</td>
<td>0.86</td>
</tr>
</tbody>
</table>

Discussion

In the hot-rolled state, the textures of the two phases are mainly the deformation textures. Additionally, however, the ferrite texture shows a certain amount of Goss-texture (011)[100], and the austenite texture contains some cubic orientation (001)[100], both of which are typical recrystallization textures. Hence, it can be concluded, that the hot-rolled state is partly recrystallized. This conclusion was corroborated by hardness tests, which showed a further decrease in hardness after annealing at 1030°C. Furthermore, annealing at 1030°C drastically reduced the typical deformation texture components (001)[110] and (011)[211] in ferrite and austenite respectively, as seen in Figure 7. This state can thus be assumed to be fully recrystallized.

After aging at 850 and 475°C respectively, further small changes in textures can be seen in Figure 7. In view, however, of the unavoidable experimental errors, these changes are assumed to be within the range of error. Hence, it was concluded that, during these annealing treatments, no further textural changes due to recrystallization took place.

On the other hand, the mechanical properties changed, as was observed in the hardness tests. In these temperature ranges, the occurrence of the α and γ phases was reported. Furthermore, in the steel containing nitrogen, Cr2N precipitates were observed. In the present paper, however, only crystal orientations are being considered, and these are not influenced by these precipitates.

The textures of the hot-rolled state were also determined by individual orientation measurements in the electron microscope using Kikuchi patterns. The results are contained in the upper line of Figure 6. They should be identical with those in Figure 4 obtained by X-ray diffraction. In view of the much poorer statistics of electron diffraction compared with X-ray diffraction, the two results are in reasonable agreement.

The second line of Figure 6 contains the misorientations of grain and phase boundaries. In the ferrite, a high peak is seen in the orientation Δϕ = [00,00,03], corresponding to low-angle boundaries. This peak is much higher than the corresponding peak in the uncorrelated MODF, equation [4], shown in the third line of Figure 6. Hence, the orientation correlation, equation [5], shown in the fourth line indicates a strong preference for low-angle boundaries. This is assumed to be due to polygonization in the plastically deformed material prior to recrystallization. The same effect was observed in the austenite. Additionally, however, two further types of boundaries were seen in the austenite. Their frequencies are higher than in the uncorrelated MODF. One of them corresponds to Σ3 boundaries, i.e. 60° (111) rotations, which are assumed to be due to recrystallization twinning in the recrystallized part of the material. The other type of boundary corresponds to mutual rotations of the crystals about (100) axes with various rotation angles. These boundaries are assumed to result from the oriented transformation from ferrite to austenite. The orientation relationship consists of several symmetry variants. A ferrite grain will thus be transformed into several austenite grains according to these symmetry variants, which thus have specific orientation relationships. However, what is seen in the orientation correlation function in Figure 6 is not this orientation relationship, but a modified one resulting from further orientation changes of the austenite crystals during plastic deformation.

In the austenite–ferrite boundary MODF, the observed peaks are much smaller than in both grain-boundary MODFs. Hence, taking experimental errors and the poor statistics into account, relevant orientation correlation of α–γ phase boundaries was not observed here.

Plastic deformation results in strong textural changes in both phases as seen in Figure 8. Hence, it is concluded that both phases participate in plastic deformation. From micro-hardness measurements in both phases, it was concluded that austenite was the harder phase. However, its textural changes show that it contributes essentially to plastic deformation.

When the tensile direction is under 45° to the original rolling direction, then rigid rotation of both phases may contribute to the textural changes that are additional to the textural changes due to slip in the slip planes. This is illustrated in Figure 9.

Plastic anisotropy of the material expressed in the r-values of Table II may thus be explained on the basis of glide deformation in the two phases, as well as rigid rotation due to microstructural anisotropy.
It has been found in two-phase alloys that the harder phase may show more pronounced textural changes than in the single-phase state. It is thought that the particles of the harder phase are free to deform into the softer phase with less interaction than in a single-phase material. On the other hand, the deformation of the harder phase must be compensated by some 'turbulent' flow of the softer phase, which leads to an observed weakening of its texture.

References
