Evolution of Texture at the Initial Stages of Continuous Annealing of Cold Rolled Dual-Phase Steel: Independent of Heating Rate

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Abstract:

The performance of cold rolled dual phase (DP) steels depends on their microstructure, which results from the thermomechanical processing conditions, involving hot rolling, cold rolling and continuous annealing. The knowledge on the influence of each annealing stage on the microstructure formation is essential for manufacturing high quality DP steels. In the present work, the effects of some intercritical annealing parameters (heating rate, soaking temperature, and soaking time on the texture and microstructure of a cold rolled DP steel (0.08%C-1.91%Mn) were investigated. The soaking temperature was chosen in a way that all the ferrite got recrystallized before the temperature was reached. The three different heating rates allowed the samples to get recrystallized in three different ways: below Ac₁, just around Ac₁ and above Ac₁. The microstructure of specimens quenched after heating stage and slow cooling stage, simulated on a Gleeble, was analyzed using optical and transmission electron microscopy. {332} fiber texture along with {112}<111> texture component were observed after heating to the soaking temperature as well as after slow cooling. The overall intensity of the texture as well as textural component was observed to be nearly independent of the heating rate as well as cooling rate. The textural evolution was correlated with the volume fractions and morphology of carbides, which depend on the annealing processing parameters.
Introduction:

It is well known that formability of cold rolled and annealed (CRA) steels are affected by the microstructural parameters e.g. grain size and texture prior to cold rolling, as well as composition and the distribution of carbides and second phases present [1-3]. Processing parameters such as rolling procedure along with annealing method also strongly influence the properties of the steel strip [3]. A combination of microstructure and processing parameters can control the recrystallization behaviour of the steel to produce highly formable steel; however, the economical as well as environmental constraints must also be considered to maintain productivity by decreasing the annealing time as possible [4].

The development of lightweight vehicles with high passive safety and fuel economy has been accomplished through the use of high strength steels, like multiphase steels. The dual phase (DP) steels consisting of ferrite and martensite is excellent choice for the above because of their low yield strength, high tensile strength, continuous yielding with good uniform elongation [5-13]. DP steels containing 10-20% of martensite typically have an UTS of ~600MPa, YS of 300-400MPA and a relatively high ductility (24-30% of total elongation) [6, 13,14].

Two important commercial annealing processes are batch annealing (BA) and continuous annealing (CA). Continuous annealing is becoming the preferred method over the batch annealing because of uniform mechanical properties, a short processing time and excellent flatness [4]. The continuous annealing process to produce cold rolled DP steels have a typical processing schedule: heating to the intercritical temperature regions, soaking in order to allow the nucleation and growth of austenite, slow cooling to the quench temperature, rapid cooling to transform the austenite into martensite, overaging and air cooling. The amount and morphology of the constituents formed depend on the annealing parameters [15-19]. The effect of the intercritical annealing parameters on the microstructure i.e. evolution of different microconstituents and mechanical properties have been studied in detail [14, 15, 18]. There are several works on the characterization of texture evolved during intercritical annealing [20-22]. The development of continuous annealing as an alternative to traditional batch annealing revolutionized the steel industry and there is a renewed interest for research in the area of texture and associated anisotropy. Many different factors have been shown to be important for optimizing texture development [1, 23-25]. The influence of carbon is now believed to be one of the most important parameter in commercial steels, but it is related with other process variables.
In the present paper, an attempt has been made to investigate the textural evolution of cold rolled DP steel at the initial stages of continuous annealing i.e. heating, soaking and slow cooling, of an industrial annealing cycle using a Gleeble thermo-mechanical simulator [26]. An attempt has been made to correlate the textural evolution with the carbides present.

**Experimental Procedure:**

An industrial heat was melted using a LD converter and cast into slabs on a 2-strand continuous caster. The chemical composition obtained is C: 0.08, Mn: 1.91, Si: 0.04, Al:0.035. The slabs (252 mm of initial thickness) were heated to 1209°C and then hot rolled on two reversing four-high roughing mill (29.67 mm) and a six-stand four-high finishing train to the thickness of 3.23 mm. The finishing temperature was kept above Ar$_3$, 870°C, and after cooling the strip was coiled at 600°C. After pickling in a hydrochloric descaling line, the hot rolled coils were cold rolled in 6 passes on a five-stand, three four-high and two six-high, tandem mill to the final thickness of 1.23 mm (~62% reduction). After cold rolling, the 150 mm x 45 mm x 1.2 mm samples were machined and used for the simulation of the industrial continuous annealing, Fig.1a, utilizing a Gleeble [26].

Annealing was carried out within the intercritical temperature range, which was determined theoretically from the chemical composition of the material to be Ac$_3$ = 854°C and Ac$_1$ = 704°C [27]. The variables studied were the heating rate, the temperature and time of soaking and the quench temperature. The processing schedules and parameters used are given in Fig.1b and Table 1. After each annealing stage, the samples were water quenched for microstructural and hardness examination.

Longitudinal sections of samples after rolling and annealing were prepared following standard metallographic procedures and examined by optical, scanning electron microscopy (SEM) and transmission electron microscopy (TEM). For observation of the grain structure the polished specimens were etched with 2% Nital. To reveal the presence of carbides, a 4% Picral etchant was used, while LePera etchant was applied to highlight the martensite/austenite (MA) constituent [18]. The volume fractions of the constituents were measured by manual counting and by the IMAGE PRO-PLUS™ image analyzer software according to the ASTM standard E 562-02 [28].
Table I: Processing parameters of the annealing process simulation: H and SC denote heating and slow cooling steps, respectively

<table>
<thead>
<tr>
<th>Legend</th>
<th>Heating</th>
<th>Soaking</th>
<th>Slow cooling</th>
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<tbody>
<tr>
<td></td>
<td>T(°C)</td>
<td>Time (s)</td>
<td>T (°C)</td>
</tr>
<tr>
<td>H1</td>
<td>820</td>
<td>155</td>
<td>-</td>
</tr>
<tr>
<td>SC1</td>
<td>-</td>
<td>-</td>
<td>750</td>
</tr>
<tr>
<td>H2</td>
<td>780</td>
<td>207</td>
<td>75</td>
</tr>
<tr>
<td>SC2</td>
<td>-</td>
<td>-</td>
<td>710</td>
</tr>
<tr>
<td>H3</td>
<td>750</td>
<td>310</td>
<td>113</td>
</tr>
<tr>
<td>SC3</td>
<td>-</td>
<td>-</td>
<td>675</td>
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Selected samples were analyzed using transmission electron microscopy. Thin foils of 3 mm diameter were punched out from about 0.2 mm thick slices, mechanically thinned to 0.12 mm and electro polished in a solution of methanol with 5% perchloric acid at -35°C at 30 V. Thin foils were examined using a transmission electron microscope Philips CM 20 operated at 200 kV.

After annealing, texture analysis was performed at the normal direction (ND) (i.e. on the sheet plane) with Cu-Kα radiation. Texture was measured on an area of 24 x 14 mm² with a flat surface that had been prepared by mechanical polishing and grinding. The thickness of the sample was maintained at 0.3 mm such that X-ray beam cannot be transmitted through it and the sample can be held easily by the specimen holder. From four incomplete pole figures (0° ≤ α ≤ 80°), {111}, {200}, {220} and {311}, ODFs were calculated after correction assuming orthorhombic sample symmetry by Arbitrarily Defined Cells (ADC) method [29].
Fig. 1: (a) Schematic showing the continuous annealing process;
(b) Schedule of annealing: H and Sc denote heating and slow cooling, respectively.
Results

The microstructure of the cold rolled steel is presented in Fig.2a. It consists of elongated grains of ferrite and deformed colonies of pearlite. Some small islands of MA constituent, shown as white areas in Fig. 2b, are also present in the microstructure. The texture has been represented by $\phi_2=45^\circ$ sections of the ODF in Fig.2c. It shows presence of $\{221\}<110>$, $\{001\}<010>$, $\{110\}<110>$, $\{112\}<111>$ and $\{441\}<118>$ components.

![Fig.2:](image)

(a) Optical micrograph after cold rolling with 2% Nital etchant;
(b) Optical micrograph after cold rolling with LePera etchant;
(c) $\phi_2=45^\circ$ sections of ODF after cold rolling.

Microstructure of annealed samples (Stages I)

After cold rolling, samples were heated to the intercritical range at different temperatures with different heating rate. Variation in heating rate allows the ferrite to recrystallize in different proportions before the soaking temperature is reached. Recrystallization was found to be completed during heating to about 685, 710 and 730°C for samples heated as per routes H3 (2.4°C/s), H2 (4°C/s), H1 (5.3°C/s), respectively. The corresponding figure of the graph depicting the evolution of recrystallization is presented in Fig.3. Therefore, all the ferrite gets recrystallized before reaching the intercritical soaking stage. The optical micrographs of the samples after quenching from this stage are shown in Fig. 4. The microstructure consists of a mixture of ferrite, martensite, MA constituent and Fe$_3$C carbides. Increasing the soaking temperature from
750°C to 820°C resulted in lower amounts of ferrite (dark gray areas) in the microstructure, Figs. 4a and 4c. This is due to the larger amount of austenite formed at higher temperatures, which transforms into martensite and carbides (light gray and white regions) on quenching.

![Graph showing recrystallization volume fraction as a function of temperature](image)

**Fig.3**: Fraction of ferrite recrystallized as a function of heating rate.

TEM micrograph of samples quenched on reaching the soaking temperature (H1) consists of small crystals of martensite and retained austenite (RA) located at ferrite grain boundaries (Fig. 4d). The size and volume fraction of such constituents are the lowest at this stage of annealing. Fine spherical Fe₃C precipitates and some retained austenites are also observed in the ferrite matrix. The Mn content in the steel was high enough to stabilize the austenite. For the samples heated to lower soaking temperature and quenched after that, the amount of ferrite is higher and the amount of second constituents (martensite, MA and carbides) is lower (Fig.4b and 4c). The effect of the processing routes on the volume fractions of carbides (dark constituents) present in the microstructure is shown in Fig. 5, which shows nearly independent of soaking temperature and time.

*Evolution of texture of annealed samples (Stages I)*

The φ₂=45° section of the ODFs corresponding to stage I, have been presented in Fig. 6. Fig.6a corresponding to heating rate H1, shows formation of new components such as
{332}<573>, {332}<179> apart from {001}<010>, {110}<111> and a scatter around {112}<111>. The components observed after H2 are {332}<110>, {332}<289>, {332}<573>, {110}<111> and {112}<111> (Fig.7b). After heating rate H3, the components are {001}<010>, {221}<110>, {110}<441>, {441}<118> and {112}<111>. A comparison of the textural components finds an evolution and disappearance of components with heating rate.

Fig.4: SEM micrographs of samples after various CA schedule: (a) H1, (b) H2, (c) H3; (d) TEM micrographs after CA schedule H1.

Microstructure of annealed samples (Stages III)

After slow cooling (Stage III, Fig. 1b), the microstructure of all samples consists of ferrite with some carbides, small amount of granular bainite and martensite formed at grain boundaries of the recrystallized ferrite grains (Fig.7). The volume fraction of MA constituent has increased
whereas total amount of second phase constituents have decreased in all samples as compared to the corresponding samples after Stage I. The amount of carbides has been decreased considerably and the difference is slightly higher for the lower annealing temperatures as presented in Fig. 5.

![Graph showing effect of processing schedule on the volume fraction of dark constituents](image)

**Fig.5: Effect of processing schedule on the volume fraction of dark constituents (mainly carbides).**

*Evolution of texture of annealed samples (Stages III)*

After Slow cooling I, the textural components are \{112\}<111>, a partial \{332\} fiber, a \{110\} fiber and a splitting of \{001\}<010> orientation (Fig.9a). Fig.9b shows formation of \{112\}<111>, \{001\}<010> along with splitting around that, A nearly complete \{332\} fiber and a \{110\} fiber after cooling rate SC2. Cooling rate SC3 shows evolution of \{112\}<111>, diffuse \{001\}<010>, a partial \{332\} fiber up to \{552\}<025> and a \{110\} fiber (Fig. 9c).

An estimation of gamma fiber evolution has been presented in Table II. It shows the intensity of gamma fiber is comparably higher in SC1 and it is slowly decreasing with the cooling rate. There is also increase in the intensity of the alpha fiber with the increasing heating rate and increasing cooling rate. Goss component is found to be insensitive to the heating rate as well as the cooling rate.
Fig. 6: $\varphi_2=45^\circ$ sections of ODF after various processing schedule: (a) H1, (b) H2 and (c) H3.

TEM micrographs corresponding to slow cooling treatment SC1 have been presented in Fig. 8a-b. Fine spherical Fe$_3$C precipitates were observed in the ferrite matrix and in some retained austenite phases.

### Table II: Evolution of texture components at different stages of annealing schedule

<table>
<thead>
<tr>
<th></th>
<th>CR</th>
<th>H1</th>
<th>H2</th>
<th>H3</th>
<th>SC1</th>
<th>SC2</th>
<th>SC3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gamma fiber</td>
<td>7.34</td>
<td>7.75</td>
<td>7.10</td>
<td>6.99</td>
<td>8.56</td>
<td>7.92</td>
<td>7.32</td>
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</table>

### Discussion:

During heating with various rates to the soaking temperature, the samples get recrystallized. Recrystallization was completed at about 685$^\circ$, 710$^\circ$ and 730$^\circ$C for samples treated with heating rates H3, H2 and H1, respectively. This means that in all routes when the intercritical soaking stage is reached, the ferrite grains are fully recrystallized. This is in agreement with earlier reported data for different chemical compositions [16,17,19]. Therefore, the sample with the heating rate H3 got recrystallized well below the Ac$_1$ temperature (704$^\circ$C); Samples with heating rate H1 recrystallized above the Ac$_1$ temperature. Only samples with heating rate H2 got recrystallized just around Ac$_1$ temperature. Therefore, the spheroidisation of carbides can take place with the heating rate, H3 and H2. The samples with heating rate H3 also experiences lower soaking temperature and longer time whereas that of H2 experiences higher
soaking temperature for shorter duration. After Ac₁ temperature ferrite and spheroidised carbides will transform to austenite. As the carbides are getting finer in size, the dissolution will be higher. Therefore, the amount of austenite will be higher. This leads to higher MA constituent in H2 sample compared to H3. Upon slow cooling, the amount of ferrite will be higher and the amount of second phase will be lower. For samples with high heating rate, ferrite recrystallization temperature will be well above the Ac₁ temperature; so the fine ferrites formed will lead to formation of fine austenite at a shorter time. As the soaking time is lesser and with a higher cooling rate, the resulting microstructure will be fine ferrite with quite large fraction of second phases.

![SEM micrographs](image)

**Fig.7:** SEM micrographs of samples after various CA schedule: (a) SC1, (b) SC2, (c) SC3.

To track the progress of recrystallization, the temperature for 50% recrystallization (T₀.₅) is used and plotted as function of temperature in Fig.10 [26]. It shows that with increasing heating rate, T₀.₅ increases except for the H2. There is drop in T₀.₅ at this heating rate. Similar behaviour is observed for temperatures at 10% recrystallization (T₀.₁), which can be considered as Tₙ. For temperatures with 90% recrystallization (T₀.₉), which is a useful approximation of Tᵢ, shows continuous increase in temperature with increasing heating rate. Increasing heating rate generally implies increase in nucleation temperature due to the finite time required to produce viable nuclei [30] and hence, increase the nucleation rate as $\dot{N} \propto \exp (-Q_n/RT)$ where $Q_n$ is the activation energy for nucleation [30]. Here it is obvious from Fig.10 that at the heating rate H2, there is a drop in the nucleation temperature compared to that of H1 and H3. It is obvious from
the figure that $T_{0.1}$ increases with heating rate, H1 and H2 and it is highest for H3. This is an unusual deviation. Higher $T_{0.1}$ for H3 indicates that at this route, initially the coarse carbides get dissolved. A comparison of Fig.5 indicates the amount of carbides is lower with heating rates H3 and H2; although the soaking temperatures for those heating rates are quite different. With heating rate H2, the recrystallization finish temperature and $A_c_1$ temperature is nearly identical; it also shows low nucleation time. The different behaviour arisen at this condition can be explained in terms of carbon present in solid solution as well as in combined form as cementite or pearlite [31,32]. However, the respective contributions of the above factors are difficult to separate experimentally.

![TEM micrograph of samples after processing route SC1: (a) BF and (b) DF.](image)

After soaking, the microstructure becomes more homogeneous, the amount of austenite formed increases and the amount of ferrite decreases with increasing soaking temperature. This is due to the diffusion-controlled mechanism of this phase transformation, where the higher temperature is more effective than the shorter heating and soaking time. As a consequence, a higher volume fraction of martensite is formed on quenching. The formation of austenite from ferrite is a diffusion controlled phase transformation. Thus, the volume fraction of austenite and its coarseness increase with increasing soaking temperature and time. The microstructure is finer for samples heated to or annealed and quenched from 820°C, because the heating rate is higher
and the time spent on heating and soaking is shorter. These samples also contain higher amount of second constituents (martensite, cementite and bainite), Fig. 4a. This distribution suggests that the formation of austenite islands took place after the complete recrystallization of ferrite and the partial spheroidisation of cementite.

![Fig.9: ϕ = 45° sections of ODF after various processing schedule: (a) SC1, (b) SC2 and (c) SC3.](image)

The formation of ferrite on cooling affects the volume fraction of the second constituent formed, Fig. 8. The longer the slow cooling (lower slow cooling temperature) the more austenite transforms to ferrite and the lower is the amount of austenite available to form martensite on subsequent quenching. This was accordingly reflected in the volume fractions of constituents present in the microstructure, Figs. 7 and 8. Although the amount of carbides decreased substantially, there was probably not enough time for their complete dissolution. TEM studies have confirmed the SEM observations.

The TEM studies confirm that the carbide spheroidisation took place already during the reheating to the soaking temperature. Coarsening of carbide particles with each subsequent stage of annealing was also visible. Therefore, the effects of carbides are quite important in the formation of {111} textures at the present case. Although there has been substantial progress in understanding the effect of C on annealing textures, controversy still exists over the respective contributions of the above factors, since those are difficult to separate out experimentally. Annealing of cold rolled low carbon steel sheet involves three independent reactions, mainly (a)
partial dissolution of cementite, during heating, releasing some carbon which diffuses into the matrix, (b) recovery and (c) recrystallization of the deformed ferrite. The amount of solute in the matrix during annealing depends on the amount of carbon in solution in the matrix at the end of cold rolling and how much Carbon is released from the cementite particles.

Fig.10: Effect of heating rate on the temperature to start recrystallization ($T_{0.1}$), to complete 50% recrystallization ($T_{0.5}$) and complete recrystallization ($T_{0.9}$).

Ushioda et al. [33] proposed an analysis of texture development during the continuous annealing of cold rolled low carbon steel sheet. It has been reported that the final texture is determined primarily by a competition between the dissolution of cementite and recrystallization of the deformed ferrite matrix. The amount of carbon getting liberated by the dissolution of the cementite particles during annealing is a function of hot band coiling temperature, annealing temperature and heating rate during annealing. In the present case, the effect of hot band coiling temperature is not there.

Toda et al. [34] showed that coarse carbide dispersions are known to be favourable for the development of $\{111\}$ texture during rapid annealing. This is due to less amount of carbon getting dissolved into the ferrite matrix from the widely spaced coarse carbides during rapid annealing. This effect appears to be strong enough to counterbalance the detrimental effect of the
nucleation of grains of random orientation around the carbide particles during recrystallization. It is well established that the presence of coarse hot band carbides favours the development of desirable annealing textures. This arises because fine carbides are able to dissolve more rapidly and to diffuse carbon into the ferrite matrix more effectively than coarse carbides. Such mechanism has the potential to play a role during annealing, because the activation energy for the dissolution of cementite, 23 kcal/mol [35] is quite lower compared to that for recrystallization, 82 kcal/mol [33]. Ushioda et al. [33, 36] made a detailed study on the kinetics of cementite dissolution. It is evident from their work that the dissolved carbon content of the ferrite can be minimized by having widely spaced cementite particles in the hot band. It is also reported in the above work, when heating rate is quite low (~10^{-2} K/s) i.e. for batch annealing, the dissolved carbon in ferrite is practically independent of the interparticle spacings. However, when the heating rate is increased beyond 10^{-1} K/s, coarse, widely spaced carbides lead to a reduction in the amount of solute carbon, because of the long distances and short times available for the carbon atoms to diffuse.

Therefore, if the steel is heated rapidly after cold rolling, the recrystallization of ferrite will take place before any significant re-solution of the carbon can occur. The absence of solute carbon seems to be instrumental in the production of strong \{111\} textures, though the precise mechanism of this effect is not clear.

In the Table II, it has been shown that volume fraction of \{111\} fiber after heating rate H3 is nearly similar to that of the cold rolled state; whereas there are gradual increase with the heating rates i.e. after H2 and H1. After heating rate H1, it shows that there are several submicron level carbide particles and they can not promote particle stimulated nucleation leading to random nucleation. However, at lower heating rate, there was presence of quite coarse carbides due to higher dissolution time. It can be pointed out in Fig. 5 that the carbides (dark constituents) are nearly independent of the heating rates; hence, the size of the carbides are crucial for PSN; this might be leading to lower volume fraction of \{111\} texture after heating rate H2 and H3. This increase in evolution of \{111\} texture is mainly due to the increase in heating rate and related to the dissolution of the carbides. The textural evolution of this \{111\} fiber shows a gradual increase after slow cooling treatments irrespective of the cooling rates. There is no effect of second phase particles i.e. carbides at this stage.
Later, Hutchinson and Ushioda [37] demonstrated that the dissolved carbon is most effective during recovery and the early stages of recrystallization. Carbon interacts with substitutional atoms such as Mn to form relatively immobile complexes, which are much more effective in retarding recovery. Therefore, it can be pointed out here, that the effect of heating rate in this low carbon steel on the evolution of \{111\} texture is observed at the initial stages only. Also, at this stage the dissolution of carbides with varying heating rates are also have effect in the evolution of \{111\} texture.

Conclusions

The textural evolution at the initial stages of continuous annealing has been investigated and it has been observed that the textural evolution was controlled by high heating rates as well as dissolution of carbides.

References: