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Effect of Warm Rolling Temperature on the Microstructure and Texture of Microcarbon Dual-Phase (DP) Steel

Qiangqiang Yuan, Zhigang Wang *, Yinghui Zhang *, Jieyun Ye, Yao Huang and Ankang Huang

School of Material Science and Engineering, Jiangxi University of Science and Technology, Ganzhou 341000, China; 13576897221@163.com (Q.Y.); yejieyun2008@163.com (J.Y.); taohua-daozhu@163.com (Y.H.); hak1365947268@163.com (A.H.)

* Correspondence: wzgang2008cn@163.com (Z.W.); jxustzyh@163.com (Y.Z.); Tel.: +86-0791-8314-422 (Z.W.); +86-0791-8314-421 (Y.Z.)

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Abstract: The effect of warm rolling temperature on microstructure and texture of microcarbon dual-phase (DP) steel was investigated through scanning electron microscopy (SEM), electron backscatter diffraction (EBSD), and transmission electron microscopy (TEM). The results showed that with the increase of rolling temperature, the density and thickness of the deformation band first increased and then decreased. Ferrite and fine martensite were observed in the annealed sheet, and the ferrite had a much more homogeneous distribution in the sample rolled at 450 °C. During warm rolling, the ferrite developed a dominant γ -fiber and a weak α -texture. During the annealing of the rolled sheet, the intensity of the γ -fiber was increased and a weak $\{001\}<100>$ texture developed in the sample rolled at room temperature. An increase in the rolling temperature generated an initial decrease and subsequent increase in the strength of the unfavorable $\{001\}<110>$ texture in the annealed sheet. In addition, the strength reached a maximum at 550 °C due to an increase in the dissolved carbon in the matrix, which was result of carbide dissolution. By contrast, the intensity of the γ -fiber remained relatively higher and was deemed the weaker $\{001\}<110>$ component in the annealed sheet rolled at 450 °C. Therefore, a larger texture factor ($f_{\gamma\text{-fiber}}/f_{(\alpha\text{-fiber}+\lambda\text{-fiber})}$) can be produced under this process.

Keywords: Nb-Cr micro-alloying steel; warm rolling; texture; microstructures; deformation band

1. Introduction

Warm rolling technology is an effective method is used to improve the strength and deep-drawing properties of advanced high-strength steel (AHSS). In general, the tensile strength of interstitial free (IF) and bake-hardenable (BH) steel hardly exceeds 500 MPa because of the single ferrite matrix [1,2]. Ordinary high-strength or ultra-high-strength steels, such as dual-phase (DP) and transformation-induced plasticity (TRIP) steel, generate poor deep drawing properties and R-values that generally do not exceed 1.2 due to the abundance of C in solid solution in the matrix [3]. Due to the influence of the C atoms in the solid solution, development of the deep drawing texture (mainly consisting of γ -fiber) in microcarbon steel is not as complete as that in ultra-low carbon steel. Han et al. [4] presented a method to control the amount of C in the solid matrix solution via the low-temperature precipitation and high-temperature dissolution of the precipitates with chrome (Cr) and molybdenum (Mo). Wang et al. [5] found that the intensity of $\{001\}<110>$ was significantly reduced by fixing the amount of C in the solid solution of Cr-Mo DP steel. Barnett [6] and Jonas [7] observed a very favorable in-grain shear band for the formation of γ -fiber in IF steels or low carbon (LC) steels, which is conducive for the development of the deep drawing texture. However, the low-temperature

deformation test results indicated the presence of very few shear bands and negative strain rate sensitivity (m) in the LC steel [8], resulting in the generation of dynamic strain aging (DSA) behavior. Many reports indicated the abundance of DSA behavior in LC steel at lower rolling temperatures, which inhibits the formation of shear bands and worsens the deep drawing texture [9,10]. Therefore, it is necessary to avoid or delay the DSA temperature region during the warm rolling of LC steel. Mohammad et al. [11] found that the addition of microalloying elements, such as Cr, can delay the DSA behavior. In addition, the in-grain shear band can also be generated during warm rolling at low temperatures. Cuddy and Leslie [12] reported similar conclusions, wherein the DSA behavior can be inhibited the C-Cr atom pair as well as the successful generation of shear bands after warm rolling.

Cr can increase the shear band contents, inhibit DSA behavior by forming large-sized precipitates, and improve tensile properties [13]. In addition, the present work also added Nb in the tested steel with the aim of fixing the C component in the solid solution. Nb has a strong affinity to C, allowing C atom pinning in the solid solution by forming precipitates and promoting favorable texture formation, including $\langle 111 \rangle // ND$ ($\langle 111 \rangle // \text{normal direction}$) fiber and $\{554\} \langle 225 \rangle$ component [14]. Hoseini and Anijdan [15] reported that Nb can intensify the $\{111\}$ texture, particularly the $\{111\} \langle 112 \rangle$ component following 400 °C rolling. In addition, the NbC precipitates were reportedly able to strengthen the matrix [16].

The present study reported the effect of the warm rolling temperature on the microstructure and texture of the C-Cr-Nb DP steel during warm rolling and annealing. The main focus was the influence of the rolling temperature on the γ -fiber texture and α -fiber texture given that the former is favorable to deep drawabilities. Meanwhile, the evolution of the microstructure and texture was investigated and the causes of the texture formation and disappearance were further analyzed by scanning electron microscopy (SEM), electron backscatter diffraction (EBSD), and transmission electron microscopy (TEM). The results of this study provide the theoretical and parametric support for the warm rolling design process of microcarbon DP steel.

2. Experimental Methods

Table 1 presents the chemical compositions of the experimental steel, in which rare earth (RE) elements were employed to purify the molten steel [17]. The tested steel was smelted in a 25 kg vacuum induction melting furnace and forged into a stock with dimensions of 90 mm $\times$ 60 mm $\times$ 30 mm.

Table 1. Chemical composition of investigated alloys in mass fraction (%).

C	Si	Mn	P	S	Nb	Cr	Al	N	RE
0.034	0.39	1.3	0.044	0.007	0.052	0.044	<0.01	0.003	0.014

Figure 1 presents a schematic illustration of the manufacture process. The forged billet was heated and soaked at 1200 °C for 2 h. The specimens were then hot-rolled to 5 mm at a finishing temperature of 870 °C and cooled at 700 °C held for 1.5 h to simulate the coiling process. The hot-rolled specimens were heated to a rolling temperature (250 °C to 550 °C, interval: 100 °C) that was maintained for 30 min, fed into the roll mill to generate 2-mm-thick specimen under a single pass, and finally quenched in water. Subsequently, the annealing process was performed on the bright annealing furnace (Type: SK-10-13H, Beijing Yanbei Jianxing Boiler Industry Co., Ltd., Beijing, China), to which the specimens were heated to 830 °C (heating rate: 8 °C S⁻¹; AC1: 780 °C; and AC3: 900 °C, which was calculated using JMatPro 9.0 software, England) and held for 120 s under nitrogen gas and then rapidly cooled to room temperature using cool water.

The microstructure and texture observed in RD-ND (rolling direction and normal direction) plane using scanning electron microscopy (SEM, Carl Zeiss AG, Jena, Germany) coupled with electron backscattered diffraction (EBSD; OXFORD Corporation, Oxford Instrument Technology Co., Ltd., Shanghai, China) with Channel 5.0 HKL software (Tango, Mambo and Salsa model,

$\phi 2 = 45^\circ$ cross-section, OXFORD). The EBSD-related experimental parameters are detailed as follows: a working voltage of 20 kV, a working distance of 15 mm, and a step size between 0.28 μm and 0.36 μm . In all of the descriptions, the α -, γ -, and λ -fibers were equivalent to $\langle 110 \rangle // \text{RD}$, $\langle 111 \rangle // \text{ND}$, and $\langle 100 \rangle // \text{RD}$. TEM specimen preparation was applied as follows: thin slices were cut from a 2-mm thick sheet with wire cutter and ground to 50 μm and then punched into a circle with a diameter of 3 mm; the 5% perchlorate alcohol solution was used for electropolishing; and the voltage was set between 30 V and 40 V. The SEM and EBSD specimens were applied by mechanical polishing and electropolishing.

Figure 1. Schematic illustration of the manufacturing process.

3. Results

3.1. Microstructure Feature

The warm rolling temperature significantly influences the thickness and distribution of the deformation band [18]. Figure 2a presents the warm-rolled microstructure composed of a small pearlite block (red dotted line frame) in the matrix. Figure 2b,c present the grain boundary carbides and pearlite fiber band, respectively, that may have been caused by the fragmentation of pearlite in the hot-rolling sheet after room temperature rolling. The ferrite elongated along the RD-direction (Figure 2c–f). The elongated pearlite bands and a wide ferrite deformation band thickness were observed at 250 °C (Figure 2c). At 350 °C, the deformation band thickness was similar to that at 250 °C, though a significantly reduced deformation pearlite band was observed. A more refined deformation band was detected at a temperature of 450 °C and at the highest density (proportion of the deformation bands area in the total field), during which the large size carbides disappeared to present a more uniform deformation. The deformation degree dramatically decreased and the band thickness significantly widened at 550 °C possibly due to the dynamic recovery at this temperature.

Figure 2. The hot rolling, room temperature, and warm rolling specimen microstructures. (a) Hot rolling: block pearlite; (b) room temperature rolling: carbides; (c) 250 °C: elongated pearlite bands; (d) 350 °C: widened band; (e) 450 °C: refined deformation band; and (f) 550 °C: widened band again.

Figure 3 presents the annealing microstructure, which is composed of ferrite and martensite. The deformed ferrite grain has a finishing recrystallization that tended to be equiaxed. The martensite grain size was larger and the distribution was uneven compared to the specimen that was rolled at room temperature. However, the specimen below 350 °C was not completely recrystallized given the presence of some thin deformed bands after annealing (red dotted line in Figure 3b,c), which may be a result of the stored energy, wherein obvious martensite aggregation was observed at room temperature and at 350 °C (red dotted box in Figure 3a,c). The deformed band disappeared, a relatively clear grain boundary was observed when the rolling temperature was increased to 450 °C (Figure 3e). In addition, Figure 3f indicates the presence of a martensite island in the ferrite grain boundary. The ferrite grain size and martensite distribution at 550 °C were less uniform than at 450 °C.

Figure 3. SEM microstructure after annealing. (a) Room Temperature: martensite aggregation; (b) 250 °C: vestigial bands; (c) 350 °C: slight martensite aggregation; (d) 450 °C: uniform distribution; and (e,f) 550 °C.

3.2. Texture Feature

Figure 4 presents the orientation distribution function (ODF) diagram of the $\phi 2 = 45^\circ$ section of the warm-rolled sheet and the frequency of the warm-rolled texture. Slightly different grain

orientation distributions as well as significant differences in intensity were observed at different rolling temperatures. The rise in the rolling temperature generated an initial increase and subsequent decrease in the intensity of $\{001\}\langle 110 \rangle$, reaching a maximum at 450 °C (Figure 4d). The $\{001\}\langle 100 \rangle$ and $\{001\}\langle 100 \rangle$ components were observed at room temperature (Figure 4a) and 250 °C (Figure 4b). The intensity of the γ -fiber after warm rolling was stronger than when rolled at room temperature except at 550 °C (Figure 4e). A strong $\{001\}\langle 110 \rangle$ component was observed at both 350 °C (Figure 4c) and 450 °C (Figure 4d). Figure 4f presents the volume fraction of several typical texture orientations and texture factors ($f_{\gamma\text{-fiber}}/f_{(\alpha\text{-fiber}+\lambda\text{-fiber})}$) [19], wherein the volume fraction of the $\{111\}$ //ND texture reached a peak at 450 °C, which was conducive to the formation of the γ -fiber. In comparison, a mere 22% of the γ -fiber was observed at 550 °C (Figure 4f). The content of the $\langle 100 \rangle$ //ND texture was less than that of the $\{111\}$ //ND texture, which was mainly concentrated on the $\{001\}\langle 110 \rangle$ texture at both 350 °C and 450 °C. The 450 °C and 550 °C specimens presented the lowest and highest α -fiber contents, respectively. In addition, the 450 °C specimen exhibited the largest texture factor, as shown in Figure 4f for $\{111\}/\{110\}+\{100\}$. The rolling temperature greatly affected the γ -fiber and $\{001\}\langle 110 \rangle$ content. Warm rolling was highly unlikely to produce $\{001\}\langle 100 \rangle$ as compared to rolling at room temperature. $\{001\}\langle 110 \rangle$ was more easily produced between 250 °C and 450 °C. In addition, warm rolling at 550 °C was more prone to generating α -fiber.

Figure 4. ODF (Orientation distribution function) diagram of the $\phi_2 = 45^\circ$ section of the warm-rolled sheet. (a) RT; (b) 250 °C; (c) 350 °C; (d) 450 °C; (e) 550 °C; and (f) warm-rolled texture volume fraction.

Figure 5 presents the orientation density line of the warm-rolled sheet. The main texture of the α -fiber shifted from $\{223\}\langle 110 \rangle$ to $\{112\}\langle 110 \rangle$ with the increase in temperature, wherein the weakest $\{001\}\langle 110 \rangle$ content was observed at lower temperatures (room temperature and 250 °C). The orientation density presented significant increases between 350 °C and 550 °C. Figure 5b shows the γ -fiber density of the warm-rolled sheet. The total density line showed high intensity at 450 °C, especially the $\{111\}\langle 123 \rangle$ component. In addition, other rolling temperatures exhibited relatively weak orientation densities, especially at 550 °C.

Figure 5. Orientation density at different rolling temperatures. (a) α -Orientation: strong intensity between $\{112\}<110>$ and $\{223\}<110>$; and (b) γ -orientation: high density at 450 °C.

Figure 6 presents texture that was mainly concentrated in the α - and γ -fiber lines after annealing, wherein different intensities were presented. Although strong γ -fiber and $\{001\}<110>$ components were observed at room temperature, a weak $\{001\}<001>$ component was still present. An increase in the temperature resulted in the disappearance of $\{001\}<100>$ as well as an obvious decrease in $\{001\}<110>$ intensity. Weak γ -recrystallization fiber and strong α -recrystallization fiber was detected following rolling at 250 °C and 350 °C, indicating these temperatures were not conducive to the deep drawing properties. However, the γ -fiber enhanced, a strong $\{554\}<225>$ component, and almost no $\{001\}<100>$ and $\{001\}<110>$ were observed at 450 °C. Better developed $\{111\}<112>$ and $\{554\}<225>$ adjacent components were detected at 550 °C. In addition, $\{001\}<110>$ exhibited enhanced intensity and the $\{110\}<223>$ components was also observed. Figure 6f presents the volume fraction of several typical orientations of annealed sheet. The $\{110\}$ texture content exhibited an inverse relationship with the temperature. The $\{111\}$ texture content reached a maximum value at 450 °C, which accounted for about 47% of the total content.

Figure 6. Annealing textures for the specimens. (a) RT: strong cubic component; (b) 250 °C: weak γ and strong α ; (c) 350 °C: same conditions as 250 °C; (d) 450 °C: strong γ -fiber; (e) 550 °C; and (f) annealing texture volume fraction.

Figure 7 presents the α - and γ -fiber density calculation results. The density of the unfavorable texture was higher in the adjacent $\{001\}<110>$ and $\{221\}<110>$ components at room temperature. At 250 °C, the α -recrystallization texture was concentrated between $\{001\}<110>$ and $\{111\}<110>$. Weak α -recrystallization texture was observed at 350 °C, whereas higher density was observed in the adjacent $\{112\}<110>$ content at 550 °C, indicating relatively low γ -recrystallization texture that was still not conducive to the deep drawing properties (Figure 7b). Nevertheless, at 450 °C, the specimen exhibited strong γ -recrystallization texture. In addition, density of $\{112\}<110>$ orientation was two-fold higher than that of the $\{111\}<110>$ orientation in Figures 6d and 7b due to the texture inheritance.

Figure 7. Orientation density after annealing: (a) α -orientation; and (b) γ -orientation.

The texture evolution beginning from the warm rolling to annealing was further analyzed. Figure 8 lists the eight typical textures that were analyzed, wherein the intensity of the γ -fiber and $\{554\}\langle 225 \rangle$ component increased with temperature from 250 °C to 450 °C. The textures slightly weakened after annealing and reached a relative balance at 450 °C. Although the γ -fiber content increased after annealing the 550 °C specimen, it was still weak possibly due to the dynamic recovery. The α -deformation texture content first decreased and then increased across the temperature range of room temperature to 550 °C, during which the lowest content was observed at 450 °C, at which time all of the content decreased after annealing. The γ -fiber only increased or remained stable after annealing when the $\{223\}\langle 110 \rangle$ and $\{112\}\langle 110 \rangle$ contents exceeded 20% in the warm-rolled sheet, such that the γ -fiber increased after annealing. The $\{001\}\langle 110 \rangle$ density of the λ -fiber significantly decreased after annealing at 350 °C as compared to annealing at room temperature or at 250 °C. Warm rolling was able to greatly reduce the influence of $\{001\}\langle 100 \rangle$ and $\{001\}\langle 110 \rangle$. In summary, rolling within the temperature range of 250 °C to 450 °C exhibited significantly higher texture factors as compared to other temperatures (Table 2). Moreover, maximum texture factor strength was observed at 450 °C for both the warm-rolled and annealed specimen, indicating optimized deep drawing properties, as indicated by the results presented in Figures 4f and 6f (green line).

Figure 8. Several typical texture volume fractions of the warm-rolled and annealed sheets.

Table 2. Texture factor of the warm-rolled and annealed sheets ($f_{\gamma\text{-fiber}}/f_{(\alpha\text{-fiber}+\lambda\text{-fiber})}$).

Texture Factor/Temperature	RT	250 °C	350 °C	450 °C	550 °C
Warm-rolled sheet	0.759	0.813	0.898	1.103	0.417
Annealed sheet	0.578	0.575	0.643	1.006	0.685

4. Discussion

Generally speaking, warm rolling in low carbon steel is presented when the rolling temperature is lower than the ferrite recrystallization temperature but higher than room temperature rolling [20]. Compared with austenitic rolling, warm rolling avoids the effect of phase transition, thus resulting in more uniform final steel mechanical properties. In the process of warm rolling, the deformation band is an important microstructure. The influence of the temperature on the deformation bands is discussed in this experiment, as gradual form is observed with the rise in rolling temperature due to inhomogeneity deformation and can be related to the local plastic instability during the rolling process [21]. Therefore, the deformation band was not easily generated at room temperature as well as under an excessive temperature (such as 550 °C). The dislocation density decreased, the ferrite deformation band widened, and the deformation degree decreased due to dynamic recovery at a rolling temperature of 550 °C. Uniform and narrow deformation bands were formed in specimen at 450 °C rolling, which provided more nucleation points for martensitic transformation in quenching. In addition, evenly distributed ferrite grain as well as a small and uniformly distributed martensitic phase was also observed. At 250 °C and 350 °C, the uneven deformation band and the incomplete

broken pearlite group resulted in the uneven distribution and a larger martensite grain during the annealing process.

The α -deformation texture was easily formed during warm rolling. In addition, warm rolling was more likely to produce $\{001\}\langle 110\rangle$ mainly due to the strongest uniform deformation band at 450 °C. Figure 9a shows the orientation distribution diagram at 350 °C. Figure 9a presents the mildly deformed $\langle 111\rangle//\text{ND}$ grains (blue color), and Figure 9b further indicates the deformed grain belonging to the $\{111\}\langle 112\rangle$ orientation (purple color). In addition, severely deformed and elongated grains that were mainly representative of the α -orientation grains (Figure 9c) were observed in the green color area. However, the grain will not stop rotating immediately when the rolling temperature was too high (550 °C). That is, the grain continue to rotate to reach the stable state, such that the experimental results showed that the stable orientation was close to the $\langle 110\rangle//\text{RD}$ orientation.

Figure 9. Orientation distribution of the warm-rolled texture. (a) Transitional band and $\{111\}\langle 112\rangle$ grains; (b) 350°C IPF (inverse pole figure); and (c) $\langle 110\rangle//\text{RD}$ grain distribution.

Deformation bands were formed by the interactions between the dislocations as well as between the precipitates and dislocations, thereby resulting in crystal bending [22]. According to Figure 10a, the dislocation density increased on the Cr-rich carbide region at low temperatures. In addition, the interactions between the dislocations also increased (Figure 10b), thereby resulting in local stress concentration, inhomogeneity deformation, and difficult grain rotation. A transition band was formed between the two deformation bands. The transition band between two $\{111\}\langle 112\rangle$ grains easily formed α -texture (Figure 9a,b; white and black line). Precipitate interaction with the dislocation was more likely to occur at higher temperatures when the atomic diffusion rate was equal to the slip rate of the dislocation [23] (Figure 10b). A rolling temperature of 450 °C induced the precipitates to undergo dislocation pinning as well as resulted in plane slip dislocations, thus promoting deformation band

production, which resulted in increased $\{001\}<110>$ content [24]. The cellular dislocation substructure was observed and certain grains exhibited dynamic recovery (Figure 10c) as the rolling temperature continued to increase to 550 °C. In addition, unfavorable C atomic content was observed in the matrix due to the development of deep drawn texture, which served as an effective mean to fixing C by forming carbides. However, no M23C6 carbides were found in the TEM photographs. A similar conclusion was produced based on the JMatPro software calculations (Figure 10d), which showed no M23C6 precipitates at 550 °C, indicating that the C content of matrix was higher than that at other temperatures, which was not conducive to the development of the deep draw texture. Dynamic recovery was observed at 550 °C, and the dislocation density was sharply released, indicating the likely formation of the α -fiber [25].

Figure 10. Dislocation density and morphology: (a) 350 °C and EDS (Energy Dispersive Spectrometer); (b) 450 °C: interaction (dislocation and precipitated); (c) 550 °C: cell structure; and (d) precipitate re-dissolution calculations.

The grain with γ orientation gradually consumed the grain with α -orientation during annealing because the former has relatively higher deformation stored energy. Warm rolling technology promoted the formation of the γ -deformation texture according to an analysis on the deformation texture, thus promoting the production of the γ -recrystallization texture, especially for the specimen at 450 °C.

However, the recrystallization texture was mainly inherited from the hot-rolled sheet. In addition, it was impossible to form recrystallization texture at room temperature rolling. Figures 4 and 7 present a small difference between the main texture types under warm rolling and annealing conditions, especially for the γ -fiber. These difference were due to the transformation between $\{111\}\langle 112 \rangle$ and $\{111\}\langle 110 \rangle$ during annealing [26,27]. However, in the annealing, the deformed $\{111\}\langle 112 \rangle$ and $\{111\}\langle 110 \rangle$ component will be converted to recrystallized $\{111\}\langle 110 \rangle$ and $\{111\}\langle 112 \rangle$ component correspondingly, indicating an insignificant difference between the two components [28].

The grain orientation and size distribution of the annealed specimens at 450 °C and 550 °C were studied to further explore the effect of the warm rolling temperature on the recrystallization texture. The results indicated that the grain orientation of $\langle 111 \rangle // \text{ND}$ dominated at an annealing of 450 °C. However, many small grains were observed at 550 °C even after the complete recrystallization. In addition, these grains was mostly likely followed non- $\langle 111 \rangle // \text{ND}$ grain orientation (Figure 11b; dotted circle). According to Figure 11c, the specimen at 550 °C exhibited a greater dispersion coefficient (0.74513) compared to the specimen at 450 °C (0.68894). A normal distribution of the grain size and a relatively uniform ferrite and martensite distribution were observed at 450 °C, indicating mixed crystal microstructure production at a rolling temperature of 550 °C after annealing.

Figure 11. IPF map of the annealing sheet. (a) 450 °C; and (b) 550 °C: fine grains; and (c) grain size distribution.

5. Conclusions

- (1) The warm rolling of C-Cr-Nb steel was more conducive to the formation of a deformation band as compared to room-rolling. Deformation bands with higher densities and narrower thicknesses were observed in the sample rolled at 450 °C. The annealed sheet had a ferrite and fine martensite

microstructure that annealed at the inter-critical region. The ferrite matrix exhibited a much more homogeneous distribution after annealing in the sample rolled at 450 °C as compared to other rolling temperature.

- (2) The {001}<110> component was easily obtained after room temperature rolling. The γ -fiber texture is the dominant texture in rolled samples. With the increase of rolling temperature, {223}<110> component gradually changed to {112}<110> component, and the {001}<110> component gradually disappeared.
- (3) An increase in the rolling temperature initially presented a decreased and subsequent increase in the unfavorable texture {001}<110> strength in the annealing sheet. The maximum was measured at 550 °C based on the increase of dissolved carbon in the matrix, which was a result of the dissolution of the chromium-based carbides. By contrast, the intensity of the γ -fiber and {554}<225> component remained relatively high and was deemed the weaker {001}<110> component in the annealed sheet after a rolling temperature of 450 °C.

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