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Materials

Experimental study on Ti+Nb bearing ultra-low carbon bake hardening sheet steel hot-rolled in the ferrite region

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Abstract: A Ti+Nb bearing ultra-low carbon bake hardening sheet steel hot-rolled in the conventional austenite region and in the ferrite region with lubrication was experimentally studied. Subsequent cold rolling and continuous annealing processes were also conducted. The results show that microstructures of ultra-low carbon bake hardening hot strips at room temperature are basically irregular polygonal ferrites. The yield strength, ultimate tensile strength, *n* value, and *r* value of the No.2 specimen hot-rolled in the ferrite region with lubrication are 243 MPa, 364 MPa, 0.29, and 1.74, respectively, which are similar to those of the No.1 specimen hot-rolled in the conventional austenite region. The elongation rate and bake hardening value of No.2 specimen are 51% and 49.4 MPa, respectively, which are greater than those of No.1 specimen. The No.2 specimen hot-rolled in the ferrite region with lubrication exhibits good mechanical properties and relatively excellent baking hardening performance. Therefore, the hot rolling experiment of Ti+Nb bearing ultra-low carbon bake hardening steel in the ferrite region with lubrication is feasible and can be considered in the future industrial trial production.

Key words: ultra low carbon steel; bake hardening; texture; microstructure; mechanical properties

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1. Introduction

Ultra-low carbon bake hardening (ULC-BH) steels initially have a lower yield strength and excellent formability in delivery condition, and these characteristics of ULC-BH steels are suitable for fabrication of the outer body parts of automotive vehicles. The dislocation density in the matrix increases substantially during press forming, so the diffusion distance of interstitial solute carbon and nitrogen atoms to dislocation sites is shortened. During paint baking treatment, interstitial solute carbon and nitrogen atoms in the matrix will be thermally activated and migrate to pin mobile dislocations to form Cottrell atmospheres, resulting in the return of discontinuous yielding accompanied by an increase in yield strength [1-7]. In the present work, free interstitial solute carbon and nitrogen atoms of Ti+Nb bearing ULC-BH steel are fully scavenged by the strong carbide and nitride formers such as titanium and niobium during the steelmaking stage. Interstitial solute carbon atoms are then liberated by the dissolution of NbC precipitates during the subsequent high temperature continuous annealing process and retained in the matrix through the rapid cooling process. The mobile dislocations produced through press forming are pinned by solute carbon atoms during subsequent paint baking, leading to an increase in yield strength due to the bake hardening effect.

For the hot rolling of ULC-BH steel in the ferrite region, the strip temperature before entering the finishing mill is decreased below the GS line of the Fe-C equilibrium diagram to complete $\gamma\rightarrow\alpha$ phase transformation through a rapid cooling system between the roughing and finishing mill. So the finish rolling is conducted completely in the ferrite region while the rough rolling is still in the austenite region. The hot rolling in the ferrite region and cold rolling of ULC-BH steels are to obtain homogeneous ferrite

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grains, coarser second phase particles and a higher proportion of {111} texture. Then these grains with preferred orientations may be developed into pronounced {111} recrystallization texture through subsequent continuous annealing cycles. Many scholars have conducted extensive researches on the ferrite region rolling, the results show that hot rolling in the ferrite region is feasible, the finished materials have pronounced {111}<110> annealing texture and manifest good deep drawability through subsequent cold rolling and continuous annealing process. The ferrite region rolling has already been carried out in low carbon steel, ultra-low carbon steel, and interstitial free steel. However, the ferrite region rolling of ULC-BH steel with lubrication was little reported in literatures [8-14].

The main objective of this paper is to study the influence of the ferrite region rolling on the mechanical properties, deep drawability, and bake hardenability of Ti+Nb ULC-BH steel. The feasibility of the ferrite region rolling with lubrication of Ti+Nb ULC-BH steel is also preliminarily evaluated. As the processing conditions of the ferrite region rolling with lubrication are different from those of the conventional austenite region rolling, so the influence of ferrite region rolling on the evolution of microstructures and textures as well as the morphology, distribution of the second phase particles and bake hardenability are different from the conventional austenite region rolling. Therefore the experimental study on the ferrite region rolling with lubrication of ULC-BH steel in this paper is of great theoretical significance and reference value of industrial production.

2. Experiments

A Ti+Nb bearing ULC-BH steel was melted in a laboratory vacuum induction furnace, then the steel ingots were forged into slabs for hot rolling. After mechanical machining, the final dimension of the slabs is 100 mm×130 mm×33 mm. The chemical composition of the Ti+Nb bearing ULC-BH steel is shown in Table 1.

Table 1. Chemical composition of experimental ULC-BH steel wt%

C	Si	Mn	P	S	N	Al	Ti	Nb
0.0028	0.012	0.27	0.078	0.0057	0.0030	<0.01	0.020	0.032

The experiments of the conventional austenite region rolling and the ferrite region rolling with lubrication of Ti+Nb ULC-BH steel were conducted. No.1 specimen is the conventional austenite region rolling steel, and No.2 specimen is the ferrite region rolling with lubrication.

The conventional austenite region rolling process was as the following. Slabs of Ti+Nb ULC-BH steel were reheated to 1150ºC in a resistance furnace and soaked for 1 h, then rolled down to the desired strip dimension through 5 passes in the laboratory mill at the finishing temperature of 910ºC. The allocation of hot rolling reduction was 33→22→15→10→6→4.5 mm. The hot strips were water cooled to 700ºC through a laminar cooling system, and then were put into a muffle furnace at the corresponding temperature for 1 h, followed by furnace cooling to simulate the coiling process. Moreover, the ferrite region rolling with lubrication of ULC-BH steel was also experimentally studied in the laboratory rolling mill. The ferrite region rolling process was as the following. The start rolling temperature was 1100ºC. The first three passes simulated rough rolling, and the allocation of hot rolling reduction was 33→22→15→10 mm. The hot strips were laminarly cooled to 840ºC for another two passes simulating finishing rolling, and the allocation of reduction was 10→6→4.5 mm. The exit temperature was maintained at 750ºC. And then the hot strips were put into a muffle furnace at 700ºC for 1 h, afterwards furnace cooled to room temperature to complete the simulation of the coiling process. The accumulated hot rolling reductions for the two experimental steels were both approximately 86%. The diagrams of the two hot rolling processes are shown in Fig. 1.

The roll gap lubrication technology is generally used in the ferrite region rolling process. Hot rolling with lubrication can effectively reduce the rolling load as well as increase the reliability of rolling mill and stability of the hot rolling process. In this study, 2vol% dilute lubrication oil was used in the ferrite region rolling process. The lubrication oil was composed of cylinder oil and vegetable oil (1:1 in volume ratio) mixed at 60-70ºC), which was sprayed to the roll gap using the pressure equipment.

Cold rolling and continuous annealing for the ULC-BH steel were conducted after the two hot rolling processes. Cold rolling reductions of the two experimental steels were both 80%. The continuous annealing cycles of the two experimental steels were simulated using a salt bath. The annealing temperature and time were 870ºC and 60 s, respectively. After continuous annealing the steel sheets were water cooled to room temperature. The influence of the conventional austenite region rolling and the ferrite region rolling on the microstructures and mechanical properties of the ULC-BH steel were investigated through the observation of microstructures, textures, dislocations, and second phase precipitates of the hot-rolled, cold-rolled, and continuously annealed specimens as well as the measurements of mechanical properties of the two experimental steels.

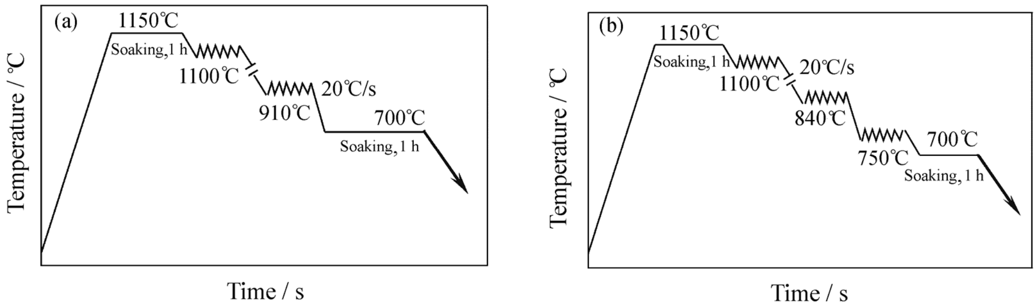

Fig. 1. Processing diagrams of ULC-BH steels hot-rolled in the conventional austenite region (a) and in the ferrite region (b).

The specimens were grounded, mechanically polished after line cutting, and etched with a 4vol% nital solution. Microstructures of the two experimental steels were observed by using optical microscopy. The 3-mm diameter thin foils for transmission electron microscopy examination were punched from 50 μm thick slices, and jet-thinned using a MTP-1A Twin Jet Electropolisher with a solution of 5vol% perchloric acid in ethanol at –30ºC at an operating voltage of 30-40 V. The morphology, size, and distribution of the precipitates as well as the dislocation configuration were observed using JEM-2000FX transmission electron microscopy operated at 120 kV. Bulk texture measurements were carried out on the quarter-planes of continuously annealed specimens using a Simens D5000 X-ray diffractometer with Mo target. Mechanical properties of the experimental steels were examined using an MTS testing machine in accordance with the GB/T228-2002 national standard for tensile tests. The bake hardenability measurement of the ultra-low carbon steels was in accordance with JIS-G3135 standards. The baking hardening (BH) value was dependent on the difference of flow stress at 2% prestrain and upper yield stress after a stoving treatment of 170ºC for 20 min. The crosshead speed of 5 mm/min was used in the tensile tests.

3. Results and discussion

3.1. Microstructure

Fig. 2 shows the optical microstructures of No.1 and No.2 specimens. It can be seen that microstructures of the Ti+Nb bearing ULC-BH hot-rolled specimens at room temperature are mainly irregular polygonal ferrite grains. The ferrite grains are coarse and inhomogeneous. The average grain size is obtained using the linear average intercept method. The average grain sizes in the plate and longitudinal planes of No.1 specimen are 16 and 20 μm, respectively.

The average grain size in the plate plane of No.2 specimen is 26 μm, the grains in the longitudinal plane are elongated with only a small amount of equiaxed grains due to the ferrite region rolling with lubrication. The average grain size of No.2 specimen is bigger than that of No.1 specimen. For No.1 specimen, the phase transformation was accelerated by laminar cooling after finish rolling, which caused the ferrite grains to have no time to grow up, therefore relatively finer hot-rolled grains were obtained. The ferrite region rolling steel was laminar cooled to 840ºC after three passes, and then warm rolled with lubrication. The coarser ferrite grains were obtained through the subsequent simulated coiling process.

Fig. 3 shows the recrystallization microstructures of the Ti+Nb bearing ULC-BH steel after high temperature continuous annealing treatment and the equiaxed ferrite grains are formed. The average grain sizes in the plate planes of No.1 and No.2 continuously annealed specimens are 14 and 18 μm, respectively. The ferrite grains of No.2 specimen hot rolled in the ferrite region with lubrication are coarser than those of No.1 specimen hot rolled in the austenite region rolling after cold rolling and continuous annealing, which may be attributed to the higher deformation stored energy obtained through cold rolling after the ferrite region rolling with lubrication, therefore, the recrystallization and grain growth are promoted.

3.2. Precipitates

The morphology and energy dispersive spectrums of precipitates in the hot-rolled, cold-rolled, and continuously annealed specimens of Ti+Nb ULC-BH steel are shown in Figs. 4-6.

It can be seen that the precipitates are fine dispersed in the hot-rolled, cold-rolled, and continuously annealed specimens and are mainly complex carbonitrides (Ti, Nb)(CN). The morphology of the precipitates are cuboidal, irregular oval-shaped, and short rod-like with the size of about 20-60 nm. Moreover, fine dispersed NbC particles with the size in the range of few nanometers up to 20-30 nm are observed in the No.1 and No.2 continuously annealed specimens.

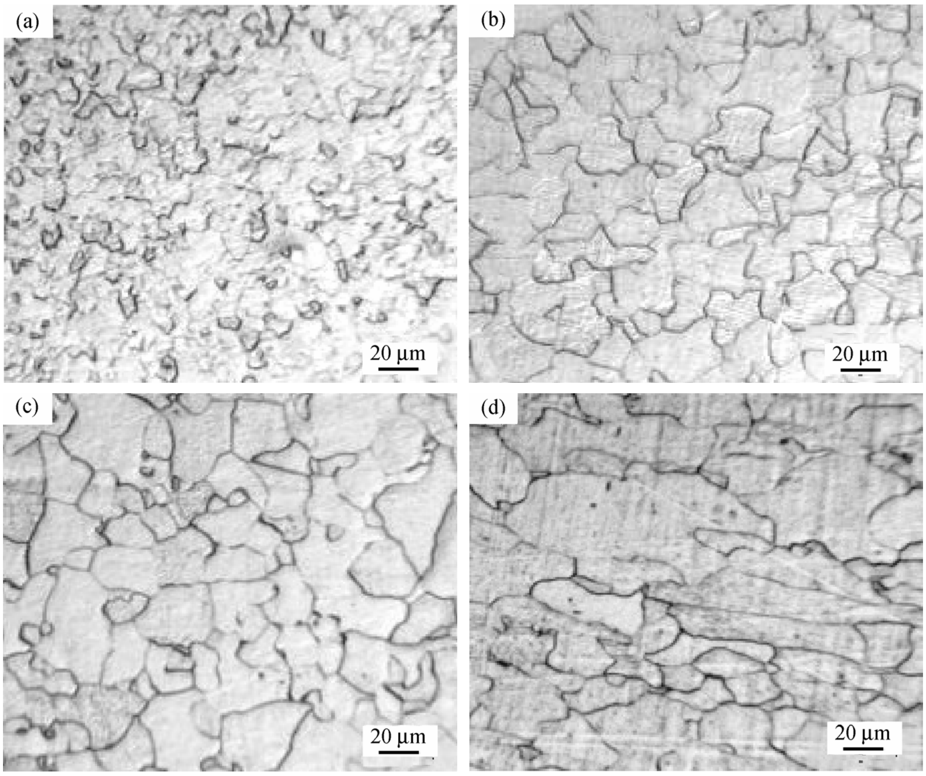

Fig. 2. Microstructures of the hot-rolled ULC-BH specimens: (a) plate plane of No.1 specimen; (b) longitudinal plane of No.1 specimen; (c) plate plane of No.2 specimen; (d) longitudinal plane of No.2 specimen.

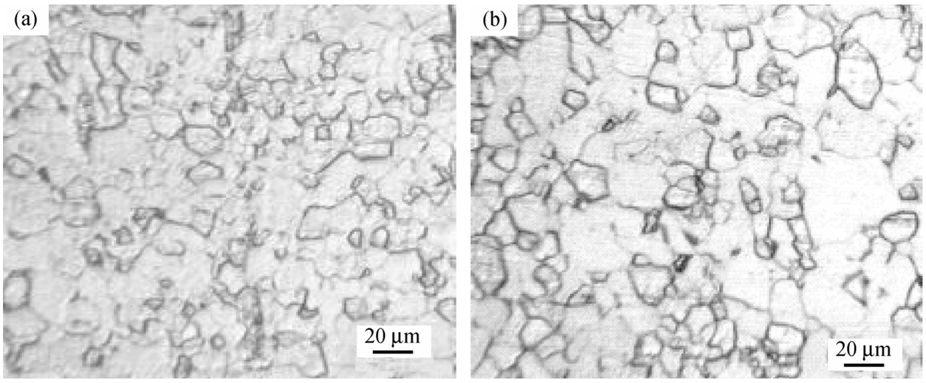

Fig. 3. Microstructures of the continuously annealed ULC-BH specimens: (a) plate plane of No.1 specimen; (b) plate plane of No.2 specimen.

It can be seen from Fig. 7 that the precipitates are more densely distributed in the No.2 specimen hot rolled in the ferrite region than in the No.1 specimen hot rolled in the conventional austenite region. The sizes and morphologies of the precipitates in No.2 specimen are similar to those in No.1 specimen. Through ImageTool software, the average sizes of the precipitates in No.1 and No.2 specimens are approximately 12 and 14 nm, respectively.

The dislocation configurations at and nearby the grain boundaries of continuously annealed specimen are shown in Fig. 8. The dislocations dispersively distribute and tangle at and nearby the grain boundaries. The precipitates are dispersed at the grain boundary and among the dislocations with the size in the range of 20-50 nm.

3.3. Mechanical properties

Mechanical properties of the two continuously annealed specimens are shown in Table 2. It can be seen that the yield strength (σ_s), ultimate tensile strength (σ_b), *r* value, and *n* value of the No.2 specimen hot-rolled in the ferrite region are similar to those of the No.1 specimen hot-rolled in the conventional austenite region. Nevertheless, the elongation rate and

BH value of No.2 specimen are higher than those of No.1 specimen.

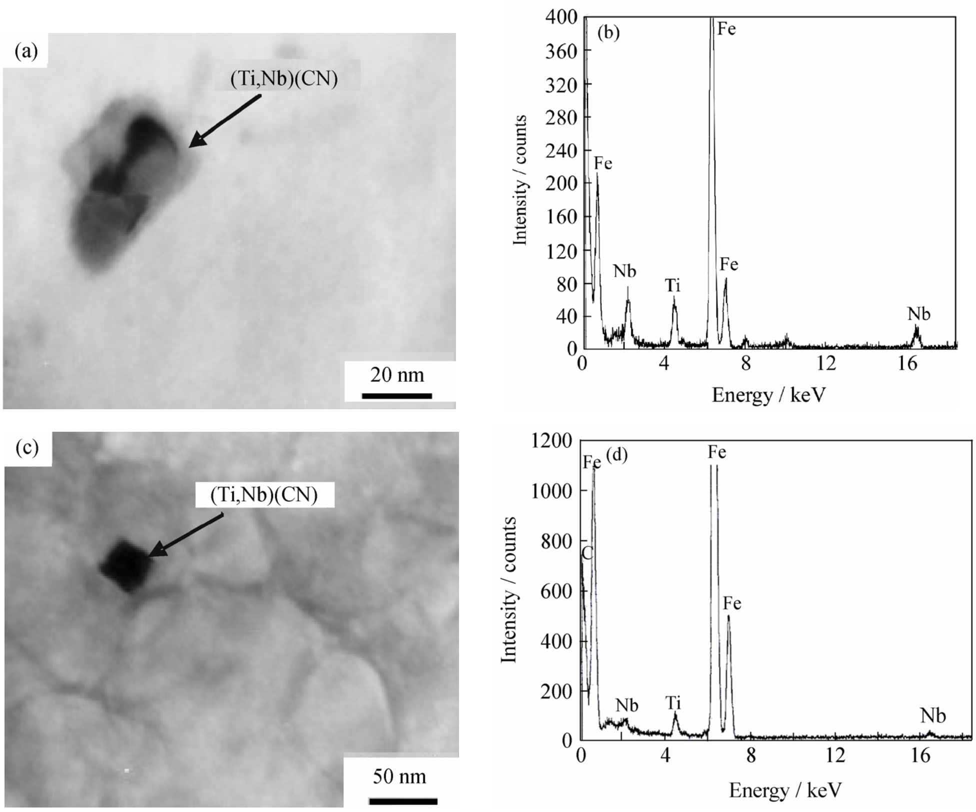

Fig. 4. (Ti, Nb)(CN) precipitate of the hot-rolled ULC-BH steel specimens: (a), (c) precipitate morphologies of No.1 and No.2 specimens, respectively; (b), (d) precipitate EDS energy spectra of No.1 and No.2 specimens, respectively.

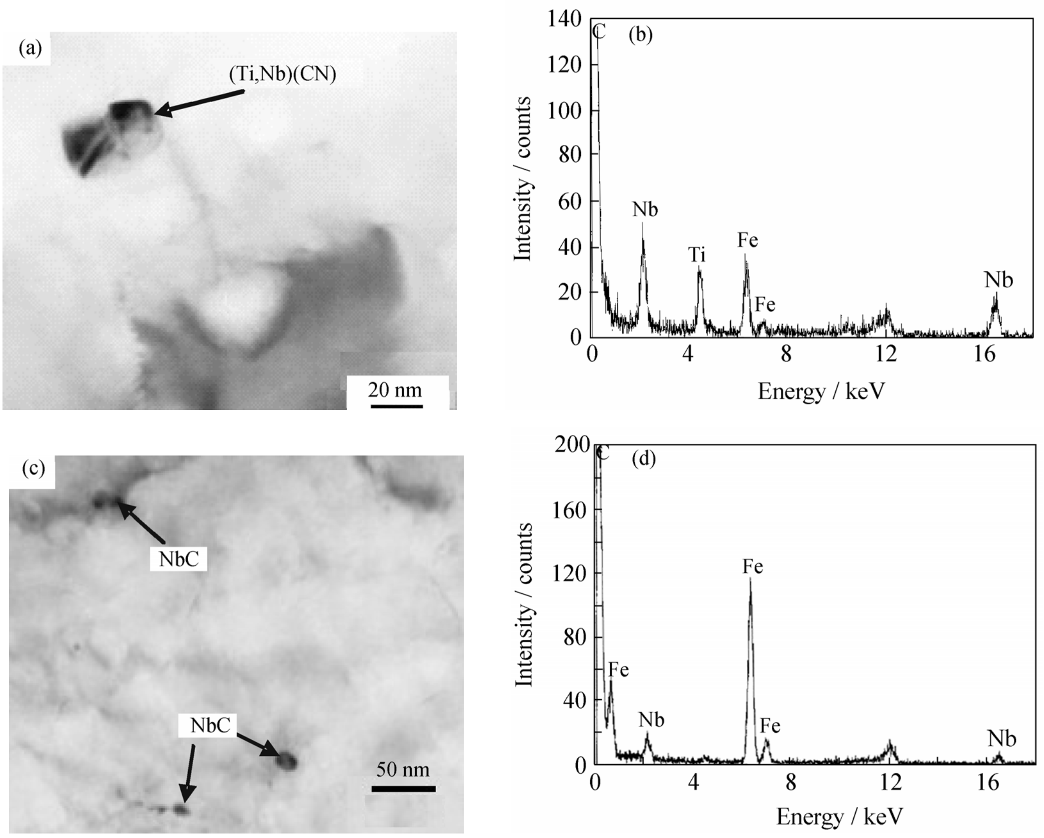

Fig. 5. (Ti, Nb)(CN) and NbC precipitates of the cold-rolled ULC-BH steel specimens: (a), (c) precipitate morphologies of No.1 and No.2 specimens, respectively; (b), (d) precipitate EDS energy spectra of No.1 and No.2 specimens, respectively.

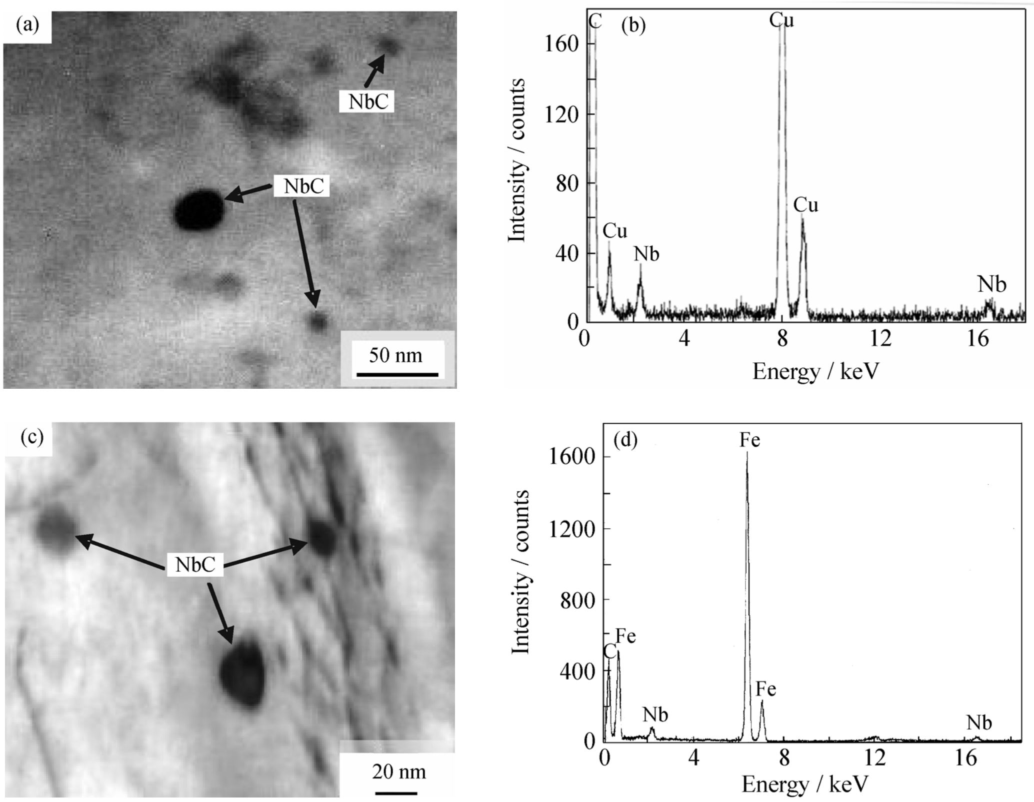

Fig. 6. NbC precipitates of the continuously annealed ULC-BH steel specimens: (a), (c) precipitate morphologies of No.1 and No.2 specimen, respectively; (b), (d) precipitate EDS energy spectra of No.1 and No.2 specimens, respectively.

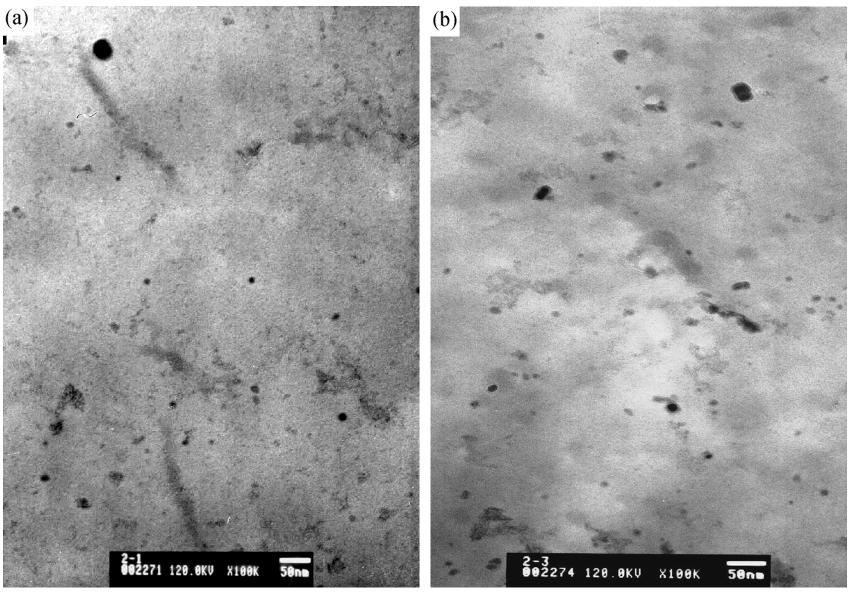

Fig. 7. Morphology and distribution of the precipitates in carbon replicas: (a) No.1 hot-rolled specimen; (b) No.2 hot-rolled specimen.

The elongation rate of the No.2 specimen hot rolled in the ferrite region is higher than that of the No.1 specimen hot rolled in the austenite region. This may be attributed to the beneficial effect of coarser ferrite grains and more interstitial carbon atoms of No.2 annealed specimen on its elongation rate. The number of grain boundaries and complex slip regions decrease with the increase in ferrite grain size. Moreover, the interstitial carbon atoms segregate to grain boundaries and dislocation sites, which reinforce the strength of grain boundaries and pin the dislocations to prevent the activation of the slip system. Therefore, it is beneficial to the strain hardening and uniform deformation of the specimen during tensile testing.

The bake hardenability of the ULC-BH steel depends mainly on mobile dislocations and free interstitial solute carbon content. It can be seen from Figs. 6-8 that NbC precipitates are more densely distributed in No.2 specimen than those in No.1 specimen, and the sizes of NbC precipitates and dislocation density are similar for the two experimental steels. For the conventional austenite region rolling, the finishing temperature is 910ºC, more NbC particles precipitate during the laminar cooling and coiling processes after hot rolling. While for the ferrite region rolling, a large amount of NbC particles precipitate along grain boundaries, subgrain boundaries, deformation bands, and dislocation tangle sites within grain interiors during the hot rolling process. The cause may be attributed to the lower hot rolling temperature and mechanism of deformation induced precipitation. NbC precipitates of the two ULC-BH experimental steels start to grow up during the high temperature coiling process after laminar cooling. Through the subsequent cold rolling process and high temperature continuous annealing cycles, denser NbC particles precipitated in No.2 specimen dissolve in large quantities, sparser NbC particles precipitated in No.1 specimen dissolve less. Therefore, more solute carbon atoms will be obtained for No.2 specimen. This is favorable to the bake hardenability resulting in a higher BH value of the No.2 specimen hot rolled in the ferrite region than that of the No.1 specimen hot rolled in the austenite region.

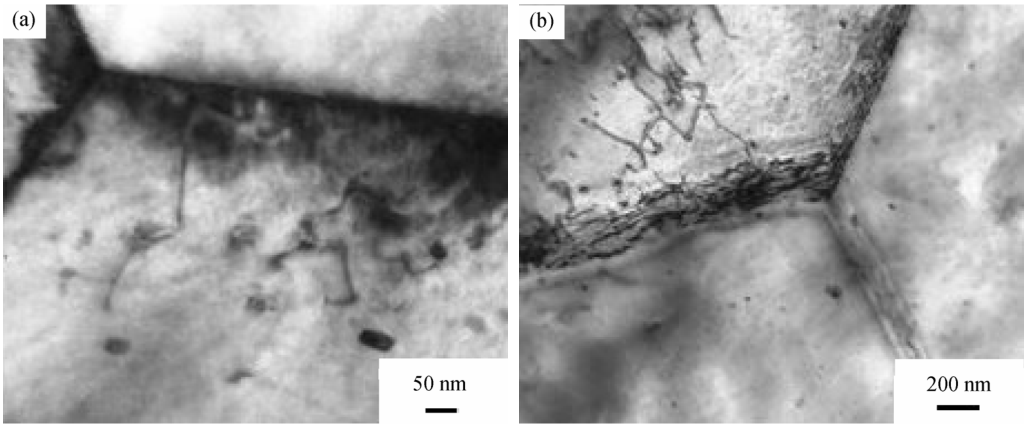

Fig. 8. Morphologies of precipitates and dislocations along the grain boundaries: (a) No.1 continuously annealed specimen; (b) No.2 continuously annealed specimen.

Table 2. Mechanical properties and BH values of ULC-BH steels

Specimen	σ_s / MPa	σ_b / MPa	A_{50} / %	r	n	BH / MPa
1	246	361	41	1.76	0.28	30.3
2	243	364	51	1.74	0.29	49.4

3.4. Texture analysis

Bulk textures of ULC-BH annealed specimens are determined from the {110}, {200}, and {211} incomplete pole figures and a series expansion technique is employed to calculate the orientation distribution function (ODF). In this paper, the φ_2=45° sections of Bunge's Euler space are used to display the textures. It can be seen from the φ_2=45° ODF sections of annealed textures in Fig. 9 that the maximum orientation distribution intensity of No.1 specimen is 9.7, which is similar to the maximum value (9.4) of No.2 specimen.

The surface texture and r value of hot strips are influenced by the lubrication state of the ferrite region rolling. Excellent lubrication condition during hot rolling is crucial to the improvement of the deep drawability of strips after cold rolling and continuous annealing.

Coarse and sparse second phase particles and fine homogeneous ferrite grains are important for ULC-BH steels to obtain fully developed {111} texture and excellent forming performance [15]. The densely distributed fine precipitates and coarse ferrite grains in the No.2 specimen hot-rolled in the ferrite region are unfavorable to the growth of recrystallized grains and the full development of the recrystallization {111} texture during the continuous annealing process. This may be attributed to the disadvantageous effect of fine densely distributed second phase particles and more interstitial solute carbon atoms on the motion of dislocations and ferrite grain boundaries, which decrease the {111} texture component of cold-rolled textures and retard the recrystallization and growth of recrystallized grains during the continuous annealing process.

The densely distributed fine precipitates and coarse ferrite grains of No.2 specimen are unfavorable to the development of {111} texture. Nevertheless, the lubrication condition during the ferrite region rolling is beneficial to the development of {111} texture. In the experiment, the deep drawing performance of No.2 specimen is not improved considerably. This may be the comprehensive results of the two influencing factors. Moreover, the heterogeneous microstructure induced by the deformed grains in the ferrite region rolling may be also unfavorable to the deep drawability. All these need to be further studied in the future research.

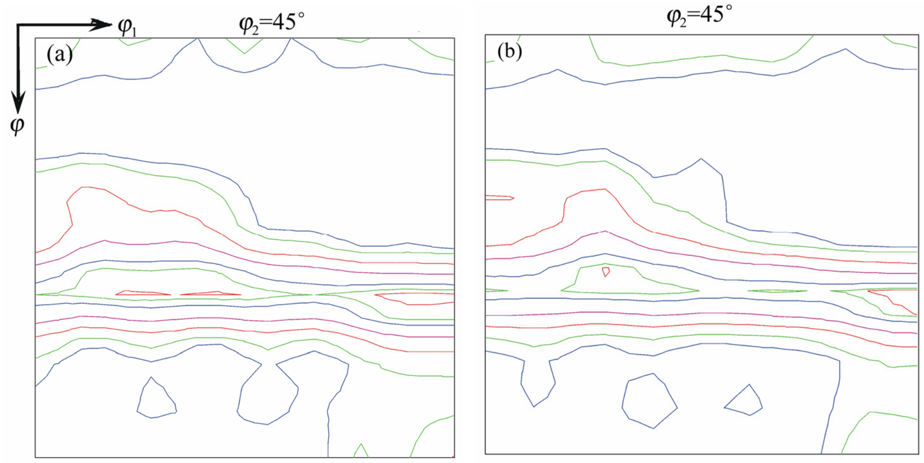

Fig. 9. ODF plots (φ_2=45°) of the main rolling and recrystallization texture components (levels: 1.0, 2.0, 4.0, 6.0, 8.0, 9.0): (a) No.1 continuously annealed specimen (maximum=9.7 times random); (b) No.2 continuously annealed specimen (maximum=9.4 times random).

4. Conclusions

(1) The yield strength, ultimate tensile strength, *r* value, and *n* value of the No.2 specimen hot-rolled in the ferrite region through subsequent cold rolling and continuous annealing processes are 243 MPa, 364 MPa, 1.74, and 0.29, respectively, which are similar to those of the No.1 specimen hot-rolled in the conventional austenite region. The BH value and elongation rate of No.2 specimen are 49.4 MPa and 51%, which are higher than those of No.1 specimen.

(2) The deep drawing performance of the No.2 specimen hot-rolled in the ferrite region with lubrication is not improved considerably. This may be the comprehensive results of the two influencing factors. One factor is the unfavorable effect of densely distributed fine precipitates and coarse ferrite grains in No.2 specimen on the development of {111} texture, the other factor is the beneficial influence of lubrication condition during the ferrite region rolling on the development of {111} texture.

(3) Through the experimental research, a result can be obtained that hot rolling of Ti+Nb bearing ULC-BH steel in the ferrite region with lubrication is feasible. The industrial trial production of Ti+Nb bearing ULC-BH steel hot-rolled in the ferrite region can be considered in the future research.

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