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Effect of Final Cooling Temperature on Microstructure and Low-Temperature Toughness of Mn-Series Ultra-Low Carbon HSLA Steel Postprint

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Abstract

The effects of final cooling temperature (550, 450, and 350 ℃) on the microstructure and low-temperature toughness of Mn-series ultra-low carbon high-strength low-alloy steel were investigated. The mechanical property test results indicated that at a final cooling temperature of 450 ℃, the experimental steel achieved a favorable strength-toughness combination, with a yield strength of 775 MPa and a ductile-brittle transition temperature of -55 ℃. Microstructural observation and crystallographic characterization results revealed that as the final cooling temperature decreased, the microstructure gradually transformed from granular bainite to lath bainite and lath martensite; at a final cooling temperature of 450 ℃, the microstructure was dominated by lath bainite, most lath packets contained three different lath blocks, the effective grain size was minimized, and the proportion of high-angle grain boundaries reached a maximum. Observation of cleavage crack propagation paths demonstrated that bainitic lath blocks with high-angle grain boundaries exerted a significant hindering effect on cleavage crack propagation; therefore, the refinement of lath block size and the increase in high-angle grain boundary proportion constitute the primary reasons for the improvement in low-temperature toughness.

Full Text

Preamble

Effect of Finish Cooling Temperature on Microstructure and Low Temperature Toughness of Mn-Series Ultra-Low Carbon HSLA Steel

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Abstract

This study investigates the effect of finish cooling temperature (550, 450, and 350 °C) on the microstructure and low temperature toughness of Mn-series ultra-low carbon high strength low alloyed (HSLA) steel. Mechanical testing results indicate that the steel achieves an optimal combination of strength and toughness at a finish cooling temperature of 450 °C, with a yield strength of 775 MPa and a ductile-brittle transition temperature of -55 °C. Microstructural observation and crystallographic characterization reveal that as the finish cooling temperature decreases, the microstructure gradually transforms from granular bainite to lath bainite and lath martensite. At 450 °C, the microstructure is dominated by lath bainite, with most packets containing three different blocks, resulting in the smallest effective grain size and the highest proportion of high-angle grain boundaries. Observations of cleavage crack propagation paths demonstrate that bainitic blocks with high-angle grain boundaries significantly impede cleavage crack propagation. Therefore, the refinement of block size and increased proportion of high-angle grain boundaries are the primary reasons for the improved low temperature toughness.

Keywords: ultra-low carbon HSLA steel, bainite microstructure, low temperature toughness, block, crystallographic feature

Introduction

Ultra-low carbon high strength low alloyed (ULC-HSLA) steels have been widely applied in engineering machinery, bridge construction, shipbuilding, oil pipelines, and aerospace applications due to their low cost, excellent strength-toughness balance, and superior weldability. These steels have received particular attention for marine engineering applications, and the related mechanism research and new steel development have long been a focus of international scholars. In recent years, to meet the demands of deep-sea and polar development, these steel plates are required to possess not only high strength but also good low temperature toughness.

Research has shown that the low temperature toughness of steel is closely related to microstructural parameters such as microstructure type, size, and high-angle grain boundaries. In industrial production, ULC-HSLA steel is rapidly cooled after rolling to a specific finish cooling temperature (FCT) before being coiled or air-cooled to room temperature. This cooling regime inevitably affects the transformation products and consequently the final mechanical properties of the steel plate. It is generally believed that selecting a lower finish cooling temperature promotes the formation of fine low-temperature microstructures (such as bainite and martensite), which is beneficial for improving mechanical properties. However, the choice of finish cooling temperature is also limited by plate thickness, production line cooling capacity, and coiling capability. Therefore, it is necessary to systematically investigate the effect of cooling regimes on microstructural transformation and low temperature toughness of ULC-HSLA steel and explore the underlying mechanisms. This understanding will help establish appropriate post-rolling cooling processes to achieve the goal of high strength and high toughness.

In this work, Mn-series ULC-HSLA steel was used as the research material. Different cooling regimes were controlled to obtain granular bainite, lath bainite, and lath martensite microstructures. The mechanical properties, particularly low temperature toughness, of these different microstructures were studied. Through crystallographic characterization of the transformation products and analysis of the microstructural evolution process, the influence of cooling regimes on the transformation process and resulting microstructure was investigated.

Experimental Procedures

The experimental steel was melted in a medium-frequency induction furnace and cast into ingots, which were then forged into 80 mm × 80 mm × 120 mm billets with a finish forging temperature of 950 °C. The main chemical composition of the experimental steel (mass fraction, %) was: C 0.03-0.05, Mn 1.5-3.0, Si 0.6-0.9, Cr 0.3-0.5, Ti 0.02-0.03, Fe balance. The critical temperatures Ac_1 and Ac_3 were measured as 758 °C and 880 °C, respectively, using a quenching dilatometer. The martensite transformation start and finish temperatures (M and M_f) were 380 °C and 315 °C, respectively.

The rolling process and cooling regime for the experimental steel plates were as follows: The billets were heated at 1200 °C for 12 hours, then rolled in five passes on a rolling mill with roll diameter of 375 mm and length of 375 mm. The starting rolling temperature was 1050 °C, the finish rolling temperature was 850-900 °C, and the deformation of each pass was controlled at 20%, resulting in a final plate thickness of 25 mm. After rolling, the plates were immediately waterquenched to 550, 450, and 350 °C (the finish cooling temperature), respectively, and then air-cooled to room temperature. These samples were designated as FCT-550, FCT-450, and FCT-350. To precisely control the finish cooling temperature, finite element simulation was first used to calculate the cooling curves at different positions in the 25 mm plate during water quenching. During cooling, the finish cooling temperature was controlled by combining the cooling curves with real-time measurement using a Raytek infrared thermometer.

Tensile tests were conducted on an INSTRON 1205 testing machine using standard tensile specimens (GBT228-2002) with an original gauge length of 25 mm and diameter of 5 mm, sampled from the middle of the plate along the rolling direction. Impact tests were performed using standard V-notch Charpy impact specimens (10 mm × 10 mm × 55 mm) sampled from the middle of the plate along the rolling direction with the notch perpendicular to the rolling plane, at temperatures of -80, -60, -40, -30, -20, -10, 0, and 20 °C.

Microstructural observation was performed using a Nikon Eclipse LV-100 optical microscope (OM) and a JSM-6301F scanning electron microscope (SEM). Quantitative analysis of microstructures was conducted using Photoshop 7.0 software. The etchant for metallographic samples was 2% nitric acid alcohol solution (volume fraction). A JEM-200CX transmission electron microscope (TEM) was used to observe the microstructure. TEM samples were prepared by mechanically grinding thin foils to 30–40 m, punching into 3 mm diameter discs, and then thinning to foil using a twin-jet electropolishing instrument with an electrolyte of ethanol + 5% perchloric acid (volume fraction) at -20 to -30 °C and a current of 30–50 mA.

Electron backscatter diffraction (EBSD) analysis was conducted using an S-3400N SEM at 15 kV with a scanning step size of 0.2–0.5 m. The obtained orientation imaging maps, pole figures, misorientation distribution maps, and effective grain size data were processed using HKL Channel 5 software. EBSD samples were first mechanically polished and then electropolished at room temperature to avoid interference from the strain layer caused by mechanical polishing on electron backscatter signals. The electropolishing solution was 10% perchloric acid alcohol solution (volume fraction) at 20 V for 10–15 s. To observe the relationship between cleavage crack propagation path and microstructure, the fracture surfaces of impact specimens tested at -80 °C were nickel-plated, cross-sectioned by wire cutting, and then examined using SEM and EBSD to observe the microstructure near the fracture cross-section.

Results and Discussion

2.1 Mechanical Properties and Microstructural Observation

The room temperature tensile properties of the ULC-HSLA steel obtained at different finish cooling temperatures are shown in Table 1 . The yield strength is above 740 MPa for all conditions and increases slightly with decreasing finish cooling temperature. The yield ratio of all samples is less than 0.8, which is beneficial for improving the safe service performance of the material. However, the impact toughness of the experimental steels shows significant differences among the three finish cooling temperatures.

Figure 1 [Figure 1: see original paper] shows the ductile-brittle transition curves of the experimental steel at different finish cooling temperatures. At a finish cooling temperature of 450 °C, the steel exhibits the best low temperature impact performance, with an upper-shelf energy (USE) of approximately 192 J and a ductile-brittle transition temperature (DBTT) as low as -55 °C. Moreover, the impact energy at -80 °C remains at 40 J. In contrast, at finish cooling temperatures of 550 and 350 °C, the DBTT values are -15 and -32 °C, respectively, and the impact energies at -80 °C are both approximately 20 J.

The SEM and TEM images of the ULC-HSLA steel microstructures obtained at different finish cooling temperatures are shown in Figure 2 [Figure 2: see original paper]. The finish cooling temperature has a significant effect on the microstructure. At 550 °C, the microstructure consists mainly of granular bainite (BG), with martensite/austenite (M/A) islands of about 1 m distributed within the matrix (Figure 2b). At 450 °C, the microstructure is dominated by lath bainite, with clear bainitic lath interfaces and fine M/A islands discontinuously distributed between the laths. Several bainitic laths are arranged in parallel to form a bainitic block (Figure 2c), with lath widths of 0.2–0.3 m (Figure 2d). At 350 °C, the microstructure consists mainly of lath martensite with a small amount of lath bainite (Figure 2e). Research has shown that lath bainite and lath martensite have similar crystallographic morphology in low-carbon steel, with several blocks existing within prior austenite grains, each composed of parallel laths. TEM images show that due to the lower transformation temperature of martensite compared to bainite, martensite laths are finer (0.1–0.2 m wide) and have straighter interfaces (Figure 2f).

The differences in mechanical properties, particularly low temperature toughness, of the ULC-HSLA steel at different finish cooling temperatures are closely related to the microstructure. At 550 °C, the presence of relatively coarse M/A islands, which are hard and can easily become crack nucleation sites or cause significant stress concentration in the adjacent matrix to induce cracking, is detrimental to impact toughness. At 450 and 350 °C, the microstructures are dominated by lath bainite and lath martensite, respectively. Although both have similar yield strengths, the lath bainite microstructure exhibits a lower DBTT. It is generally believed that in low-carbon/ultra-low carbon bainitic or martensitic microstructures, packet/block boundaries act as high-angle grain boundaries (misorientation $> 15°$) that can hinder cleavage crack propagation. Previous experiments have shown that the DBTT of steel decreases with refinement of packet/block size. However, SEM and TEM observations cannot effectively distinguish between bainitic and martensitic packets/blocks, so EBSD technology was employed for further analysis.

2.2 Crystallographic Characterization

Studies by Morito et al. and Furuhara et al. have shown that in low-carbon steels (0.0026%C–0.38%C), lath martensite maintains a K-S orientation relationship with the parent austenite. Their research on 0.15%C-Fe-Ni steel also demonstrated that bainite has similar crystallographic characteristics to lath martensite, maintaining a K-S relationship with the parent austenite. In this work, granular bainite, lath bainite, and lath martensite microstructures were obtained through different cooling regimes. All three have a bcc structure and maintain a K-S relationship with the fcc austenite parent phase, which can be expressed as:

{011}bcc   {111}fcc, [$\bar{1}$11]bcc   [$\bar{1}$01]fcc

Due to crystallographic symmetry, there are 24 crystallographically equivalent variants in the bcc system that maintain the K-S relationship. These variants are numbered V1–V24, as shown in Table 2 .

Figure 3 [Figure 3: see original paper] shows the EBSD analysis results for the ULC-HSLA steel at a finish cooling temperature of 550 °C. Figure 3a is an orientation map of the granular bainite microstructure, where black lines indicate boundaries with misorientation > 10° and white lines outline a prior austenite grain boundary. Variants from Table 2 are marked with V and numbers. Figure 3b shows the {001} pole figure of variants transformed from the austenite grain circled in Figure 3a, where symbols indicate variants calculated according to the K-S relationship. The measured variant orientations agree well with the K-S calculations. It can be seen that the prior austenite grain contains only one packet (a group of laths with the same habit plane), and most of the area is occupied by two specific variant combinations (V8&V11). These two variant combinations have the same <011> rotation axis and a misorientation of 10.53° between them. Kitahara et al. observed similar phenomena in lath martensite microstructures and termed this specific variant combination a sub-block.

To analyze the misorientation between different variants (packets/blocks), a boundary map was introduced, as shown in Figure 4a [Figure 4: see original paper]. The misorientation between points along line AB and point A, as well as point-to-point misorientations, are shown in Figures 4b and 4c, respectively. In Figure 4a, boundaries with misorientation > 15° are marked with red thick lines, those with 10°–15° misorientation with blue thick lines, and those with 2°–10° misorientation with yellow thin lines. Line AB passes through V8&V11 and V9, indicating that the misorientation between the V8&V11 variant combination is less than 15°, forming sub-block boundaries that are difficult to hinder cleavage crack propagation during fracture, thus being detrimental to toughness.

Figure 5 [Figure 5: see original paper] shows the EBSD analysis results for the ULC-HSLA steel at a finish cooling temperature of 450 °C. Figure 5a is an orientation map of the lath bainite microstructure, where black lines indicate boundaries with misorientation > 10° and white lines outline a prior austenite grain boundary. Variants from Table 2 are marked with V and numbers. Figure 5b shows the {001} pole figure of variants transformed from the austenite grain circled in Figure 5a. It can be seen that the prior austenite grain contains only one packet, but this packet includes three blocks corresponding to V1&V4, V2&V5, and V3&V6 variants. The three blocks are distributed alternately, refining the block size.

The boundary map for the 450 °C finish cooling temperature sample is shown in Figure 6a [Figure 6: see original paper]. The misorientation between points along line AB and point A, as well as point-to-point misorientations, are shown in Figures 6b and 6c, respectively. Line AB passes through V1&V4, V2&V5, and V3&V6. The misorientation between these blocks is greater than 15°, forming high-angle grain boundaries that can hinder cleavage crack propagation and improve low temperature toughness.

Additionally, in the sample with a finish cooling temperature of 450 °C, a prior austenite grain can contain multiple packets, with each packet composed of several blocks, as shown in Figure 7 [Figure 7: see original paper]. Figures 7a and 7b show the SEM image and EBSD orientation map, respectively, where white lines outline a prior austenite grain and blue lines mark packet boundaries. Figure 7c shows the {001} pole figure of variants transformed from the austenite grain circled in Figure 7a. It can be seen that there are five packets within one prior austenite grain, among which packets P1, P2, and P3 each consist of three different blocks, with all block interfaces being high-angle grain boundaries, thus benefiting low temperature toughness improvement.

Figure 8 [Figure 8: see original paper] shows the EBSD analysis results for the ULC-HSLA steel at a finish cooling temperature of 350 °C. Figure 8a is an orientation map of the lath martensite microstructure, where black lines indicate boundaries with misorientation $> 10°$, white lines indicate prior austenite grain boundaries, and blue lines indicate martensite packet boundaries. Figure 8b shows the {001} pole figure of variants transformed from the austenite grain circled in Figure 8a. It can be seen that the prior austenite grain contains four packets (P1, P2, P3, and P4), but P1, P3, and P4 each contain only one block, while P2 contains two blocks.

Based on the crystallographic characterization, the following conclusions can be drawn: (1) At a finish cooling temperature of 550 °C, the ULC-HSLA steel microstructure is dominated by granular bainite, where a prior austenite grain generally contains only one packet, which is dominated by a single block that may consist of sub-blocks with low-angle boundaries. (2) At 450 °C, the microstructure is dominated by lath bainite, where a prior austenite grain may contain only one packet, but this packet consists of three different blocks distributed alternately. Additionally, a prior austenite grain may contain several packets, with most packets composed of three different blocks. (3) At 350 °C, the microstructure is dominated by lath martensite, where a prior austenite grain may contain several packets, but most packets contain only a single block.

The crystallographic features of ULC-HSLA steel determine its proportion of high-angle grain boundaries and substructural dimensions. Statistical analysis using HKL Channel 5 software indicates that the proportions of high-angle grain boundaries are 15.8%, 20.4%, and 14.3% at finish cooling temperatures of 550, 450, and 350 °C, respectively. The effective grain sizes (with misorientation $>$ 15°) are 7.8, 4.3, and 5.3 m, respectively.

During cooling, different finish cooling temperatures determine different transformation temperature ranges, which in turn affect the austenite transformation process and transformation products. Studies by Morito et al. and Kawata et al. have shown that when the transformation temperature is high and the driving force is relatively small, the transformed microstructure within the same prior austenite grain exhibits obvious variant selection. After variant formation, rapid growth leads to most of the prior austenite grain being dominated by a single variant, resulting in coarse packets/blocks. When the transformation temperature decreases, the driving force increases, variant selection is weakened, and more variants can appear in the same prior austenite grain. You et al. have suggested that during the transformation of austenite to bainite, a relatively sufficient and slow transformation process can promote the formation of more variants distributed alternately, which is beneficial for refining packet/block size. When the transformation temperature is lowered to the martensite region, the martensite variants nucleate and grow within a very short time. Moreover, because the strength of the austenite parent phase in ULC-HSLA steel is lower than that in traditional medium/high carbon or high alloy steels, it cannot provide sufficient stress constraint on the nucleation and growth process of martensite variants, resulting in martensite packets composed of single blocks.

2.3 Analysis of Low Temperature Toughness Improvement Mechanism

Research has shown that the ductile-brittle transition temperature (DBTT) of steel is primarily determined by the effective grain size controlling toughness; the smaller the effective grain size, the lower the DBTT. The relationship between DBTT and cleavage fracture unit size d can be expressed as:

$$T = F(\text{chem}) + F(s) - K_y d^{-1/2}$$

where T is the ductile-brittle transition temperature, $F(\text{chem})$ and $F(s)$ are functions related to composition and strength, respectively, and K_y is a material constant. Since the three samples in this work have the same composition and similar strength, the difference in DBTT is inferred to be mainly caused by variations in effective grain size. Previous studies have shown that martensite packets are the effective grains controlling low temperature toughness in martensitic steels, and the size of cleavage fracture units is related to packet width.

To analyze the relationship between microstructure and cleavage fracture units in ULC-HSLA steel, cross-sections beneath the impact fracture surfaces at -80 °C were examined. Figure 9a [Figure 9: see original paper] shows the relationship between granular bainite microstructure and cleavage crack propagation in the FCT-550 sample. The cleavage crack propagation path length is essentially consistent with the prior austenite grain size. Figure 9b shows the relationship between lath martensite microstructure and cleavage crack propagation in the FCT-350 sample. Most cleavage cracks traverse entire martensite packets until they encounter prior austenite grain boundaries, with unit crack propagation lengths reaching up to 30 m.

Figure 10 [Figure 10: see original paper] shows the SEM image and EBSD analysis results of the cross-section beneath the -80 °C impact fracture surface for the FCT-450 sample. Within a single packet, block boundaries impede cleavage crack propagation, significantly reducing the unit crack propagation length. Crystallographic analysis reveals that at a finish cooling temperature of 450 °C, the microstructure is dominated by lath bainite, with most packets composed of three different blocks. The block size is refined, and these block interfaces are generally high-angle grain boundaries. When cleavage cracks encounter these high-angle grain boundaries during propagation, their direction changes, absorbing considerable impact energy and thus reducing DBTT to achieve optimal low temperature toughness.

Conclusions

1. The strength and toughness of Mn-series ultra-low carbon HSLA steel are closely related to the finish cooling temperature. At a finish cooling temperature of 450 °C, the experimental steel achieves an excellent combination of strength and toughness: yield strength of 775 MPa, room temperature impact energy of 192 J, impact energy of 40 J at -80 °C, and particularly significant improvement in low temperature toughness with the ductile-brittle transition temperature decreasing to -55 °C.
2. As the finish cooling temperature decreases, the microstructure gradually transforms from granular bainite to lath bainite and lath martensite. Crystallographic characterization reveals that at a finish cooling temperature of 450 °C, the microstructure is dominated by lath bainite, with most packets composed of three different blocks, resulting in the smallest effective grain size and the maximum proportion of high-angle grain boundaries, thereby improving low temperature toughness.

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