Effects of cryogenic treatment on mechanical properties and crystal orientation of 0.25C-0.80Si-1.6Mn steel with extraordinary strength-toughness

Yongli Chen1,2,∗, Yuhua Li1, Xuejiao Zhou3,∗, Fei Tan1 and Yueyue Jiang1,3

1 School of Metallurgical and Materials Engineering, Chongqing University of Science and Technology, Chongqing 401331, People’s Republic of China
2 Chongqing Shanwaishan Blood Purification Technology Co., Ltd, Chongqing 401331, People’s Republic of China
3 State Key Laboratory of Rolling and Automation, Northeastern University, Shenyang 110819, People’s Republic of China

* Authors to whom any correspondence should be addressed.

E-mail: zzj9040203017@126.com (XJ Zhou) and chenyongli@cuqst.edu.cn (YL Chen)

Keywords: dual phase steel, deep cryogenic treatment, crystal orientation, yield ratio, retained austenite, microstructure and mechanical properties

Abstract

Deep cryogenic treatment (DCT) in the phase transition region of austenite (A) to ferrite (F) is a novel process that can efficiently improve the content of martensite (M) and F of dual phase (DP) steel. In this work, microstructure transformation in DP structural steel treated by DCT was investigated in detail. Scanning electron microscopy (SEM) and electron backscatter diffraction (EBSD) were used to characterize the microstructure, alloy distribution, grain size, and orientation for the DP structural steel. The results demonstrated that DCT could effectively improve the yield ratio and mechanical properties of the tested steel. F and its large-angle significantly reduced the influence of fine-grain strengthening on the reduction of yield ratio. This research offered innovative technical support and theory guidelines for producing and studying such extraordinary strength-toughness DP steel with high strength and low yield ratio.

1. Introduction

The low yield ratio with high strength-toughness of steel was regarded as one of the outstanding metallurgical achievements for the modern bulling’s seismic steel application. DP steel with Ferrite (F), Martensite(M), and Retained Austenite (RA) has been paid a lot of attention in recent years. One method of obtaining the DP microstructure is to adjust the carbon distribution of different microstructures in the isothermal heating process, which can generate M and F in the subsequent cooling process [1–6]. Another is to control the cooling routine for austenite’s transformation behaviors to obtain the characteristic microstructure of M and F [7, 8]. Researches of Nouri et al [9] and Jiang et al [10] showed that F and M volume increased with the increase of silicon content. Davies et al [11] found that raising F’s content could enhance the toughness of DP steel. Kim et al [12] proposed that the refinement of M grain size was beneficial to yield strength and toughness in low temperature, but it was detrimental to yield ratio. Lan et al [13] elucidated the relationship between processing, microstructure and mechanical properties, showing that the tensile strength was increased with an increase in the volume fraction of lath bainite (LB) and lath Martensite (LM).

Scholars have been committed to cooling coolant, and the air, water, and oil were always selected as the primary. Due to its low cooling rate, the grain size of the transformed M is usually large. The DP steel with a low concentration gradient alloy and RA has weak tensile strength and toughness. Deep cryogenic treatment (DCT) optimizes the service performance by changing the microstructure irreversibly. It has been well proved that DCT is beneficial to the mechanical properties of tool steels [14], carburized steels [15], and alloy structural steels [16]. The mechanisms of DCT are discussed as well in the researches. It has been confirmed that the transformation of retained austenite into martensite and the precipitation of ultra-fine carbides are the main two reasons that
induce the improvement of performance [17, 18]. We develop a new process that a particular F is prepared in the A + F region, and the distribution of alloying elements between A and F is appropriately controlled. Then the tested steel is cooled by liquid nitrogen to −196 °C to get smaller lath of transitioned martensite and obtain more volume of retained austenite, which is beneficial to increase the strength and toughness of the steel simultaneously. Furthermore, the DP steel produced by the DCT has a lower yield ratio characteristic. As a consequence, this innovative technology is an efficient industrial production method with great potential application value. It is meaningful for the production of high-strength DP steel and is also helpful for developing the construction industry in the future.

2. Materials and methods

2.1. Materials

The chemical composition of the tested steel was 0.25 C, 0.80 Si, 1.6 Mn, 0.045 P, 0.04 S, 0.02 Nb, Cr+Mo≤0.1 (wt.%), and Fe balances. The metallographic specimens were etched by 4% nitric (Vol.%) after being grinded and polished. Leica 2500 M (Leica, Wetzlall, Germany) microscope was used to observe the microstructure of the sample. The microstructure is mainly composed of F and P, as shown in figure 1(a). Tensile properties were tested according to the standard GB/T 228.1-2010 by WAW-1000 (SUST Electric Equipment Co., Ltd, Zhuhai, China) universal tensile testing machine. Mechanical properties are presented in figure 1(b), which indicates that yield strength (YS), tensile strength (TS), total elongation (TEL), and yield ratio (YR) are 567 MPa, 734 MPa, 16%, and 0.76, respectively. Figure 1(c) shows that there are many small-angle grain boundaries (GB ≤ 5°) and a large number of large-angle grain boundaries (GB ≥ 15°) in the experimental steel. This is because the experimental steel produces abundant of small-angle grain boundaries after rolling deformation. Figure 1(d) displays that the experimental steel contains a more large ratio of grains with the average diameter about 2 μm, and contains a relatively low ratio of grains with the average diameter about 5.5 and 11.52 μm.

2.2. Methods

Experimental steels were heated to 1200 °C and held for 900 s to achieve complete austenitization, then the tested steels were cooled to the two-phase region (γ + α) with 750 °C (Process 4), 800 °C (Process 3), 850 °C (Process 2), and 900 °C (Process 1) and held for 900 s, respectively. Afterward, the tested steels were subjected to DCT processes, which were presented in figure 2. The treated specimens were cut along the axis by a wire spark
cutting machine and etched with 4% (Vol.%) nitric acid-alcohol after being polished. Next, metallography was investigated by Leica 2500 M (Leica, Wetzlall, Germany) optical microscope, and microhardness was tested in WMHV-1000 (Shanghai Hongce, Shanghai, China) Microscopic Hardness Tester. Scanning electron microscope coupled with energy disperse spectroscopy (SEM/EDS) (S-3400N, Hitachi, Tokyo, Japan) has been used to observe the microstructure and the alloying element distribution of the tested steel. The selected specimens were electropolished under 25 V constant voltage for 10–30 s. The electron backscatter diffraction (EBSD) was operated in S-3400N to investigate the crystal orientation with scanning step size of 0.3 μm.

3. Results and discussion

3.1. Mechanical properties and microstructure
Table 1 shows the mechanical properties of the four different processes. It is easy to find that the tension strength has been significantly improved and the yield strength has been markedly decreased compared with the original steel. Mechanical properties, such as yield strength, tensile strength, total elongation, and yield ratio for Process 3 are 540 MPa, 863 MPa, 16%, and 0.62, respectively. Thus, the DCT in the austenite-ferrite zone can effectively improve the strength and toughness of the steel. The excellent combination of strength and yield ratio indicates that the steel has an excellent seismic performance. This can offer technical support and theoretical guidance for the production of such extraordinary strength-toughness steel.

The SEM micrographs of the tested steel were shown in figure 3. It indicates that DCT can effectively generate F and M, which is beneficial to improve the tensile strength and reduce the yield ratio.

3.2. Effect of isothermal temperature in the austenite-ferrite zone on microstructure and properties
The microstructures of DCT after isothermal treatment at different temperatures were presented in figure 4. It can be seen that the F + M microstructure was transited at tested temperatures 750 °C–900 °C. With the decrease of temperature, the volume contents of F for the four different processes increased markedly from 5.25% at 900 °C to 55.51% at 750 °C, as shown in figure 4 (e). Further analysis of figure 4, it can be seen that the F content at 900 °C was still small, even there was no significant precipitation. A large amount of M was formed by
DCT, which indicated that $\gamma \rightarrow \alpha$ phase transformation was tiny at 900 °C. More F was formed along the boundaries of austenite at 850 °C and 800 °C. With the decrease of isothermal temperature, a lot of F formed at 750 °C, and the M was significantly reduced. Combining with figures 4(a)–(d), We can safely conclude that as the isothermal temperature decreases in the dual-phase zone, the F content gradually increases at the temperature range between 750 °C and 800 °C.

Microstructure and microhardness at 850 °C under process 3 were shown in figure 5. The average microhardness of F was 247.81 in figures 5(a), (b) and the microhardness of M was 262.16 in figures 5(c), (d), which was more clearly displayed in figure 5(e). We found that M microhardness is greater than the F after DCT, but the difference between them is not significant. It is mainly due to the refinement strengthening of M treated by DCT, which causes the M microhardness is higher than that of the F transited in the dual-phase isothermal treatment. Simultaneously, DCT enhances the precipitation strengthening for F and M, increasing F and M hardness. Under the combined effects of refinement and precipitation strengthening, its microhardness difference is not significant.
The distribution of alloying elements tested by the EDS line scan under process 3 was presented in figure 6. The line scan result showed that when M was generated in the tested steel, the carbon content in the initial position increased. Then the carbon content was redistributed to form a new microstructure.

3.3. Effect of orientation on microstructure and properties
The phase composition of process 3 of EBSD characterization is present in figure 7. It shows crystal structure is mainly composed FCC and BCC. According to the volume calculation, the BCC accounts for 96%, and FCC accounts for 4%. The microstructures composed of BCC crystal structure is mainly M and F. Simultaneously, FCC is mainly RA, which presents M-A island and film-like RA distributed along with the martensite lath shape [19–22]. Figure 7(b) shows the grain size statistical distribution of BCC and FCC. Compared with the grain distribution of the original sample in figure 1(b), it can be seen that after DCT treatment, most of the original grain is reduced to 0.33 μm² from 0.52 μm²; the grain size is reduced by 36.53%. The effect of cryogenic crystal refining is enhanced. According to the Hall–Patch strengthening law [23, 24], the strength and toughness will simultaneously increase.

Figure 8 shows the misorientation of grains and its numerical statistics under process 3. It can be seen from figure 8(a) that there are a large number of adjacent grains with misorientation greater than 15°, and a small amount of grains with misorientation less than 5°. Morris et al and Ghosh et al [25] have shown that large-angle grain boundaries can improve the toughness, and the result of this experiment also conforms to this law. The essence of the large-angle interface that is beneficial to improving the toughness of steel is that the cleavage plane orientation is larger. When the cleavage crack passes through, the large deflection occurs at such interface, which inhibits the propagation of cleavage cracks.

At the same time, we can find from figure 8(b) that in the large-angle state greater than 15°, there are still a large number of medium-angle difference states with the misorientation between 11°–25°, which is contrary to
our traditional belief of large-angle interface which was known that the small-angle grain boundary is generally less than 5 degrees. The large-angle grain boundary is greater than 15 degrees. And there are quite a few grains of misorientation less than 25 degrees in process 3. The statistics of grain misorientation show the phenomenon of ‘bimodal aggregation.’ It may be due to the production of RA, F and M in the quenching treatment in the two-phase zone, and more F of orientation difference $12^\circ - 25^\circ$ was produced during the isothermal process in the two-phase zone. Compared with figures 8(a) and 7(a), it reveals that the grains with a misorientation greater than 15$^\circ$ are the F with polygonal morphology and a BCC crystal structure, which was a transition in the dual phase zone isothermal process, and orientation of M grains is greater than 45$^\circ$.

Figure 9 shows the average misorientation of each phase and the distribution of internal misorientations. It can be seen from figure 9(a) that the average grain orientation of M (green) is worse than that of F (brown and red). At the same time, it can be seen from figure 9(b) that internal misorientations of F are higher than that of martensite. And the two RA forms of the M-A island and the film-like RA presented in figure 7(a) were relatively low. The coordinated effect of RA may cause it. With the uneven distribution of the carbon element during the isothermal process, the carbon concentration in each crystal is different [26], resulting in the increase of the misorientation in difference of the crystal grains. It indicates that the DCT process does not increase M internal misorientation during $\gamma$ to $\alpha$ phase transition. DCT is advantageous to reduce the grain size further. Some investigations [27–29] have shown that fine-grain strengthening improves the strength and toughness of steel but reduces the yield ratio. Furthermore, a large-angle state can reduce the strength of steel and increase its plasticity and toughness, which has been confirmed by many scholars [30–32]. The cryogenic treatment after isothermal treatment in the two-phase zone in this experiment can increase the F content and refine the size of the microstructure and increase the ratio of the large-angle grains. In this study, the strength of the tested steel increased, and their yield ratio reduced. It indicated that the F produced by DCT and its large angle significantly reduce the influence of fine-grain strengthening on the yield ratio reduction.
4. Conclusion

The 0.25C-0.80Si-1.6Mn steel was holding in a two-phase region of 750 °C–900 °C for 900 s then subjected to DCT; the mechanical achievement properties of high strength combined with low yield ratio were obtained. Microstructure and orientation were analyzed in detail. The main conclusions are as follows:

1. Compared with the original steel performance, the deep cryogenic treatment’s mechanical properties of 0.25C-0.80Si-1.6Mn steel were significantly improved. The yield strength was decreased to 540 MPa from 567 MPa, tensile strength was increased to 863 MPa from 734 MPa, and the yield ratio was decreased to 0.62 from 0.76.

2. The microstructures of DCT after isothermal treatment with the decrease of temperature between 750 °C and 900 °C, F volume content gradually increased.

3. After DCT treatment, most of the original grain is reduced to 0.33 μm² from 0.52 μm², the grain size is reduced by 36.53%. DCT process does not increase the internal misorientation of M during γ to α phase transition. Besides, F produced by DCT and its large angle significantly reduce the influence of fine-grain strengthening on reducing yield ratio.

Acknowledgments

This work was supported by the National Natural Science Foundation of China (No. 51704055, 51904052), Science and Technology Research Program of Chongqing Municipal Education Commission (No. KJQN201801501, KJQN201901508), Chongqing Special Postdoctoral Science Foundation, Postdoctoral Science Foundation of China (No. cstc2019jcyj-msxmX0106, cstc2020jcyj-msxmX0476), and Natural Science Foundation of Chongqing, China (No. cstc2019jcyj-msxmX0106, cstc2020jcyj-msxmX0476).

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Conflicts of interest

The authors declare no conflict of interest.

ORCID iDs

Yongli Chen https://orcid.org/0000-0003-2485-6009

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