

Research Article

Effects of Cryogenic Treatment and Tempering on Mechanical Properties and Microstructure of 0.25C-0.80Si-1.6Mn Steel

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The effects of mechanical strength and yield ratio on the seismic steel are significant. This paper investigated the mechanical strength and yield ratio of medium carbon seismic steel by deep cryogenic treatment at -60°C and -110°C for 60~120 s and tempering at 350°C for 30 minutes. The microstructure, misorientation, grain boundary, and size were characterized by electron backscattering diffraction (EBSD), scanning electron microscopy (SEM), and energy dispersive spectrometry (EDS). The result shows that the tensile strength and reduction of the cross-sectional area of steel were increased by 170 MPa and 9% and yield ratio and elongation were decreased by 0.13 and 9% due to the existence of the large-angle grain boundary. The present study confirms that the mechanical properties of medium seismic steel can be improved effectively by the deep cryogenic treatment and tempering.

1. Introduction

Ultralow temperature treatment was first used in the 1930s to improve the mechanical properties of metal materials. It was divided into cold treatment (CT) and deep cryogenic treatment (DCT). DCT improves the mechanical properties of metal materials by promoting the transformation of residual austenite (RA) to martensite (M), increasing the precipitation of dispersed carbides, and accelerating the removal of residual strain. This kind of treatment was mostly used for the treatment of tool steels [1–7]. More recently, Molinari et al. [8–10] studied the cryogenic treatment on the hardness performance of high-speed steel. The results indicated that cryogenic treatment had an excellent effect on the improvement of toughness and hardness. In the follow-up study, they took advantage of the conventional heat treatment and CT to demonstrate that the white layer and the precipitation of secondary carbides could significantly improve the wear resistance of the steel [11, 12]. Besides, Chen et al. [13] pointed out that the CT and secondary tempering could effectively improve the tensile strength, yield strength, elongation, and fracture toughness by

refining the martensite substructure, promoting the transformation of retained austenite, and strengthening the precipitation of carbides in steel but decreasing the hardness. Overall, these studies focus on the wear resistance, toughness, and hardness of tool steel.

The main purpose of the present work is to investigate the relationship of yield strength ratio, strength, and microstructure transformation of 0.25C-0.80Si-1.6Mn steel under the treatment of DCT and tempering. It was confirmed that this novel procedure was an effective technology to improve the mechanical properties of the seismic steel, and this research is meaningful to promote the development of seismic steel in the construction industry.

2. Experimental Materials and Methods

2.1. Experimental Materials. The steel with the chemical composition (wt%) of 0.25C-0.80Si-1.6Mn-0.045P-0.04S was selected as the object in the present study. Based on the GB/T 228.1-2010, the size of the tensile specimen is shown in Figure 1. It has presented that the total length was 140 mm, diameter of clamping end was $\phi 8$ mm, and test rod diameter was $\phi 5$ mm; the size and shape of the metallographic sample

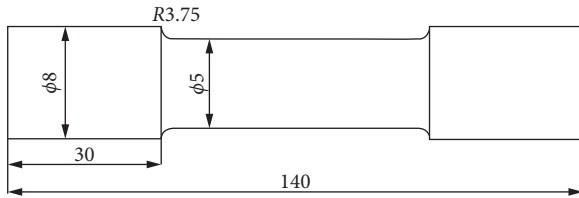


FIGURE 1: Schematic diagram of tensile specimen.

were designed as a cylinder with a diameter of 5 and a height of 5 to observe the microstructure.

Afterwards, the microstructure of initial steel was observed by using the optical microscope (OM) of Leica 2500M and electron backscattering diffraction. Some valuable results can be seen from Figure 2(a). The results show that the microstructure of initial steel was ferrite (red arrow) and pearlite (black arrow).

From Figures 2(b) and 2(c), the large-angle grain boundary (white curve) occupied a large proportion, accounting for about 81.5%, and the low-angle grain boundary (green curve) and the subgrain boundary (red curve) accounted for a small proportion, about 16.5% and 2%, respectively, and the average grain size of initial steel was $5.27 \mu\text{m}$ in Figure 2(d). Moreover, the original mechanical properties of steel were tested by using the high-temperature tensile testing machine. It can be concluded that the values of tensile strength, yield strength, total elongation, reduction of cross-section area, and yield ratio are 734 MPa, 567 MPa, 16%, 33%, and 0.76, respectively.

2.2. Experimental Methods. The steel samples were austenitized at 950°C for 30 minutes in the furnace and cooled with different temperatures in the low-temperature equipment. The technological process graph is shown in Figure 3. Then, the samples were treated by process 1 at -60°C and process 2 at -110°C , respectively.

Afterwards, the sample treated by process 1 was grinded, polished, and then etched by the 4% nitric acid alcohol, and the steel treated by process 2 was electropolished under 25 V constant voltage for 10~30 s. The electron backscatter diffraction (EBSD) was operated to investigate the microstructure and grain orientation with 2000 magnification. Finally, the tensile strength, yield strength, elongation, and other mechanical properties of the samples were measured with the testing equipment, and the results were analyzed with MTEX [14], SEM, and EBSD.

3. Experimental Results

3.1. Mechanical Properties. The mechanical properties of steel samples cooled at -60°C and -110°C for 60~120 s and tempered at 350°C for 30 minutes are shown in Figure 4. It can be seen from Figure 4 that the yield strength increased from 734 MPa to 904 MPa and tensile strength raised from 567 MPa to 571 MPa, respectively. The yield ratio was decreased from 0.76 at original temperature to 0.62 and 0.63 at -60°C and -110°C , respectively. As shown in Figure 4(c), compared with original temperature, the reduction of cross-

sectional area was raised from 33% to 31% and 42% at -60°C and -110°C , elongation was decreased sharply from 16% to 7% and 5% at -60°C and -110°C , respectively. Based on the results, it can be summarized that the deep cryogenic treatment at -110°C is better than the cryogenic treatment at -60°C on the improvement of tensile strength, yield strength, and yield ratio. The reason for the improvement of the steel performance by the deep cryogenic treatment at -110°C is that the cooling temperature over -80°C [15], which can promote the transformation of microstructure and dissolution of elements [16].

3.2. Effect of -60°C Insulation and 350°C Tempering on Microstructure. To investigate the effect of deep cryogenic treatment at -60°C and tempering at 350°C on the microstructure of steel, SEM and OM were utilized to observe the microstructure of steel, and the results are presented in Figure 5. The microstructure apparently involves ferrite and tempered troostite. The scan arrow and fluctuation of alloy contents of steel are indicated in Figures 5(c) and 5(d). From Figure 5(c), it can be concluded that the fluctuation of carbon is the largest, and the other fluctuation of chemical elements changes very little. Especially at the positions of 21.4 nm and 23.0 nm along the scanning direction, the fluctuation of carbon was particularly obvious. It indicates carbon has migrated and diffused at these positions during the treatment; hence, the phenomenon of fluctuation of alloy elements also presents that the tempered troostite was emerged at these positions.

In addition, some useful information can be obtained from Figures 6(a) and 6(b). It shows that the misorientation angle of the grain boundary was mainly distributed in the range of $20^\circ\sim 60^\circ$, and compared with the grain size of initial steel, the average grain size was decreased from $5.27 \mu\text{m}$ of initial grain size to $4.07 \mu\text{m}$, in which the large-grain boundary accounted for 83.5%, the low-angle grain boundary accounted for 14.48%, and the subgrain boundary accounted for 2.02%. Besides, the phenomenon has also indicated that the migration and dissolution of elements during the transformation of microstructure are strongly affected by the content of carbon. When the carbon was migrated and dissolved under the cryogenic treatment, the transformation of ferrite, martensite, and other microstructures in the steel can be compromised as better situation. Thereby, the mechanical properties of steel can be improved significantly.

3.3. EBSD Analysis under -110°C Insulation and 350°C Tempering. The EBSD analysis of microstructure for the steel with deep cryogenic treatment at -110°C and tempering at 350°C was operated with MTEX, and the results are shown in Figure 7. Figure 7(a) clearly illustrates that the main phase of steel are body-centered cubic (BCC) and face-centered cubic (FCC). Obviously, the result clearly indicates that the microstructures of the sample are ferrite and troostite. From Figure 7(a), it is meaningful to note that the FCC accounts for 25%, RA accounts for 1.2%, and BCC accounts for 73.8%. Figures 7(b) and 7(c) provide the IPF of FCC and BCC of steel. The result demonstrates that the

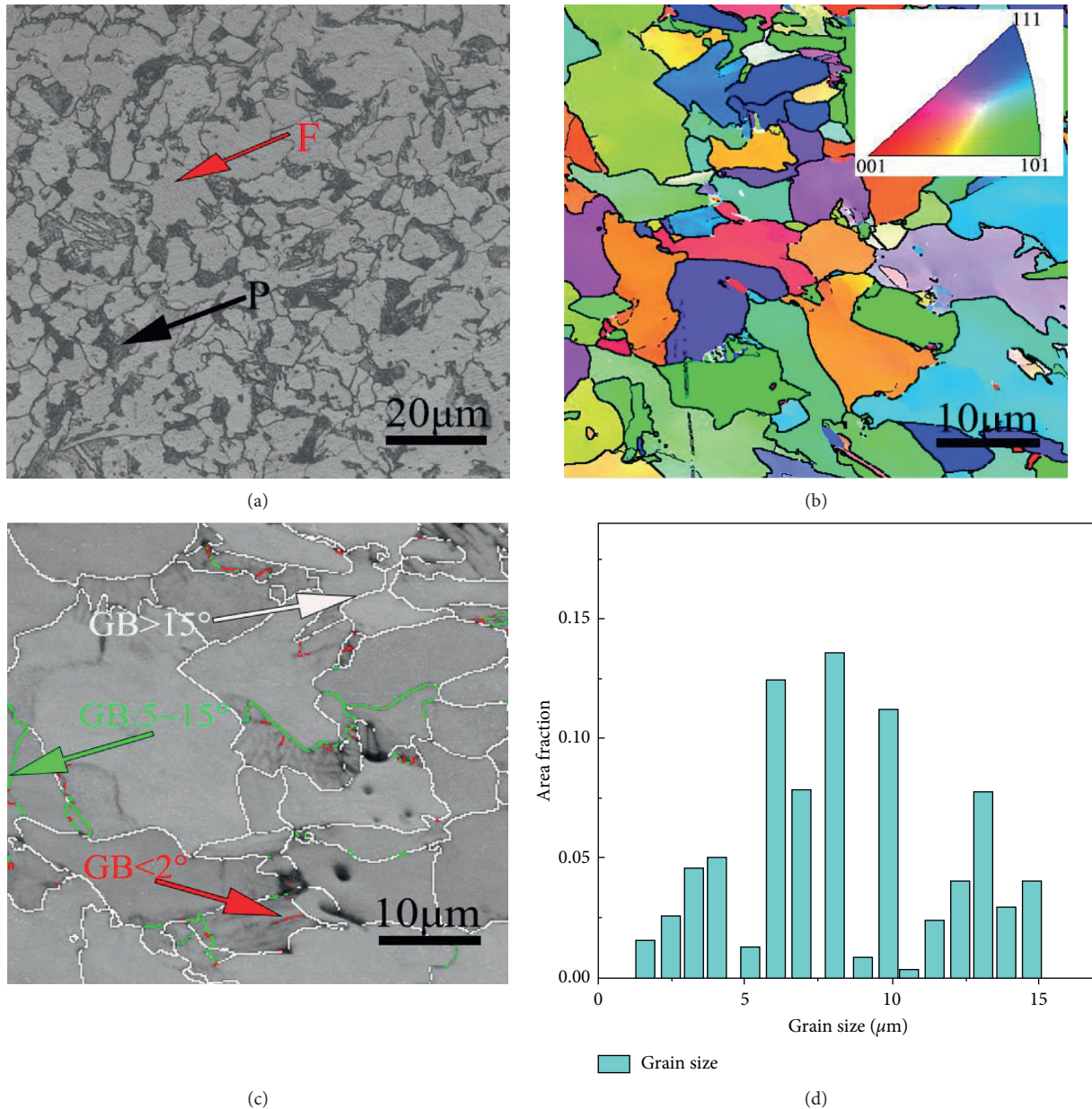


FIGURE 2: Microstructure and grain boundary of initial steel. (a) OM microstructure of initial steel, (b) IPF of initial steel, (c) grain boundary of initial steel, and (d) grain size of initial steel.

orientation of grains is to focus on [001]. Figures 7(d) and 8 show the different grain boundaries and misorientation of sample. Figure 7(d) indicates that the low-angle grain boundary accounts for 13.22%, large-angle grain boundary accounts for 86.78%, and subgrain boundary accounted for 4.2%.

Based on the perspective of recrystallization, most of the grains have been recrystallized. It is clear to see in Figure 8 that the grain size of steel was refined with the deep cryogenic treatment at -110°C and tempering. The average size of grain is $3.71\ \mu\text{m}$. As a consequence, due to the low-angle grain boundary accounting for a small proportion in the microstructure of steel [17], the plasticity and engineering strength of steel were affected significantly.

4. Discussion

Based on the results, it is easy to note that the deep cryogenic treatment and tempering have a greater influence on the mechanical properties of the steel samples, such as the yield strength ratio, elongation, and tensile strength. The value of large-grain boundary, low-angle grain boundary, subgrain boundary, and average grain size was 83.5%, 14.48%, 2.02%, and $4.07\ \mu\text{m}$, respectively, under process 1. When steel was treated by process 2, average grain size is $3.71\ \mu\text{m}$, and the large-angle boundary of the steel samples accounts for 86.78%, the low-angle boundary accounts for 13.22%, and the subgrain boundary accounts for 4.2% in the tested steel samples. Compared with the factors of initial steel in

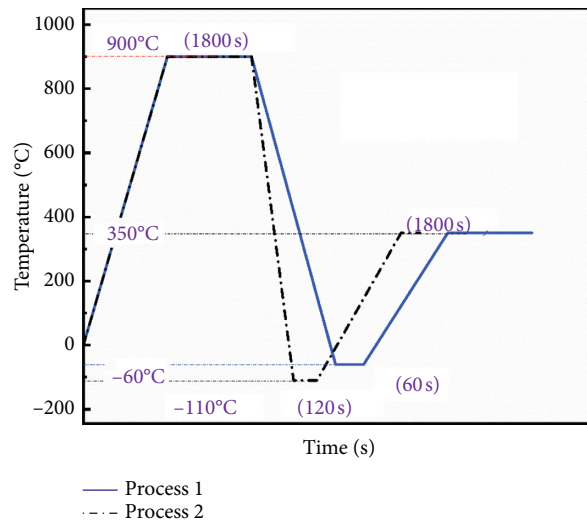


FIGURE 3: Process graph of cryogenic treatment and tempering.

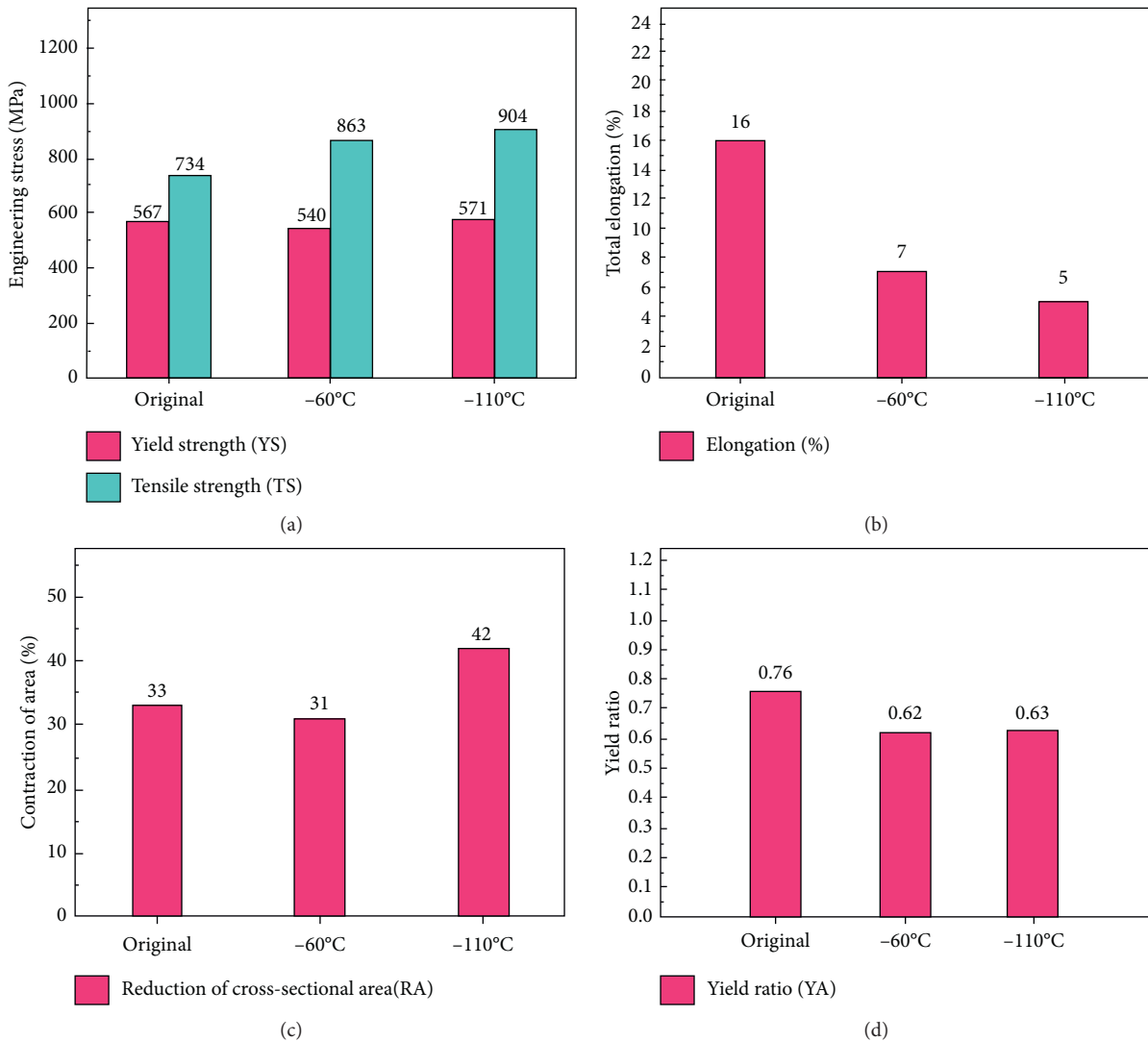


FIGURE 4: Mechanical properties under different processes. (a) Tensile strength and yield strength, (b) total elongation, (c) contraction of area, and (d) yield ratio.

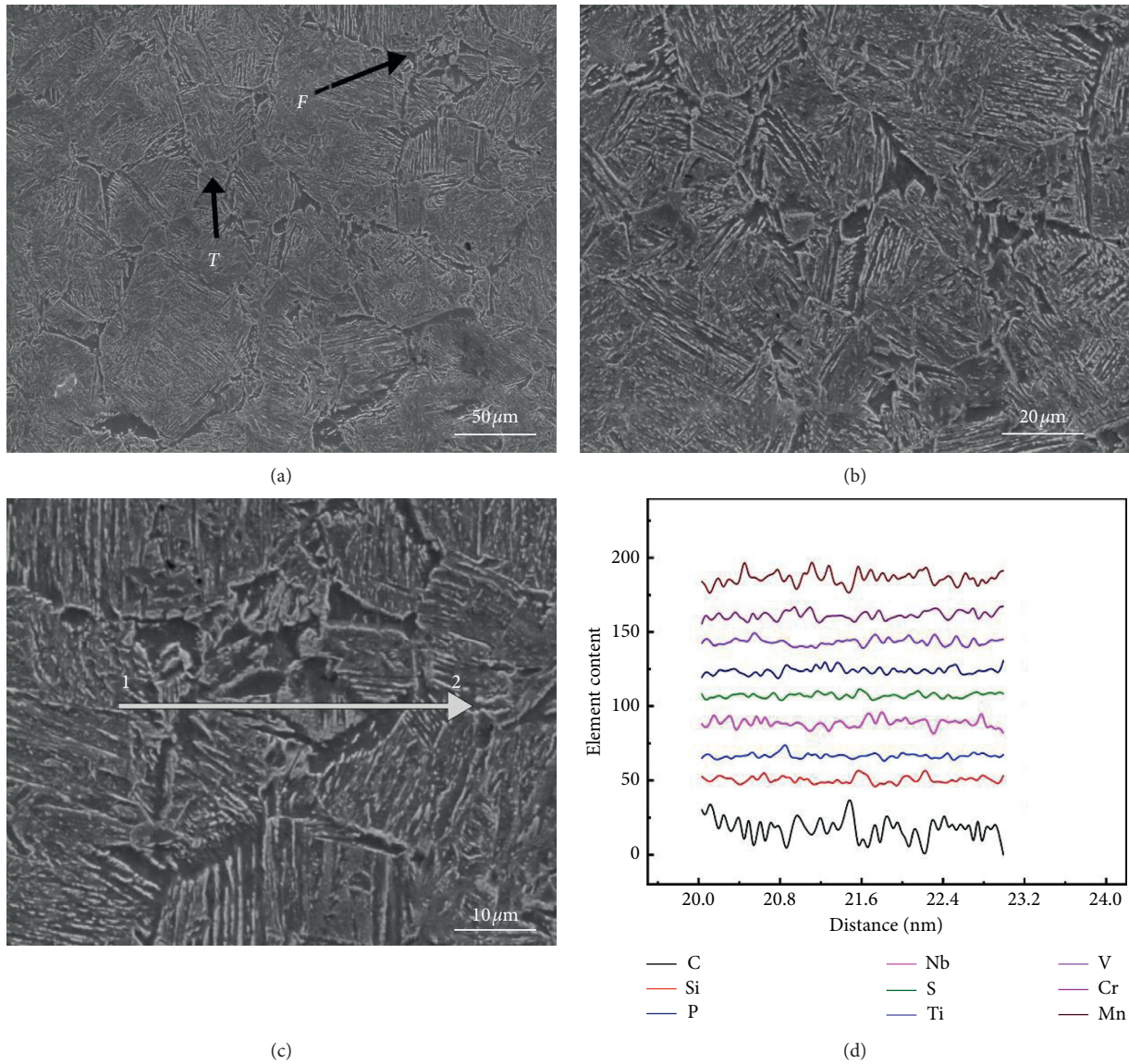


FIGURE 5: DCT at -60°C and tempering at 350°C . (a) Image of SEM, (b) image of OM, (c) line scanning position, and (d) fluctuating of element by line scanning.

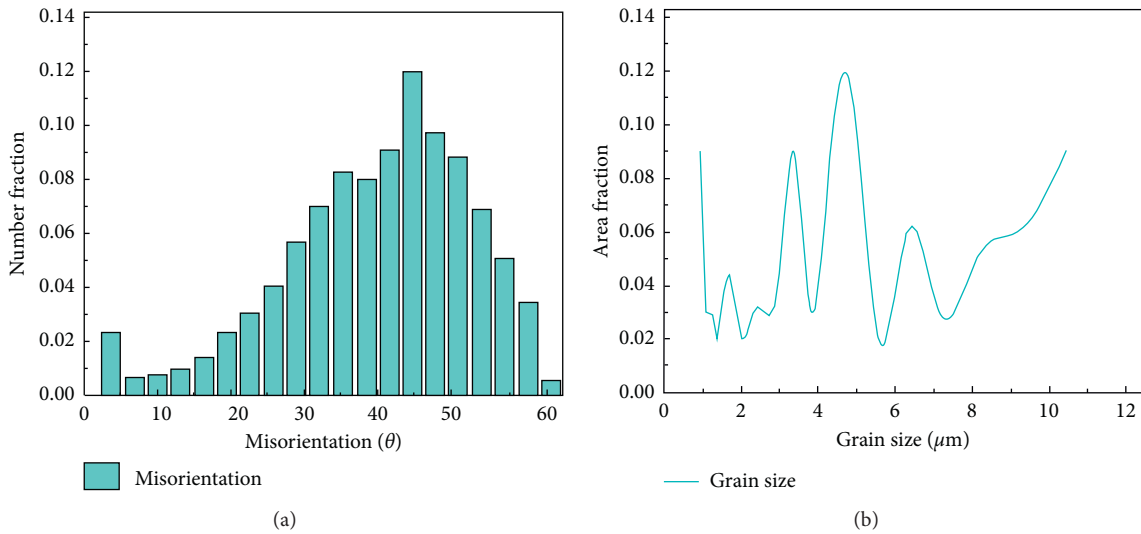


FIGURE 6: DCT at -60°C and tempering at 350°C . (a) Misorientation histogram and (b) grain size curve.

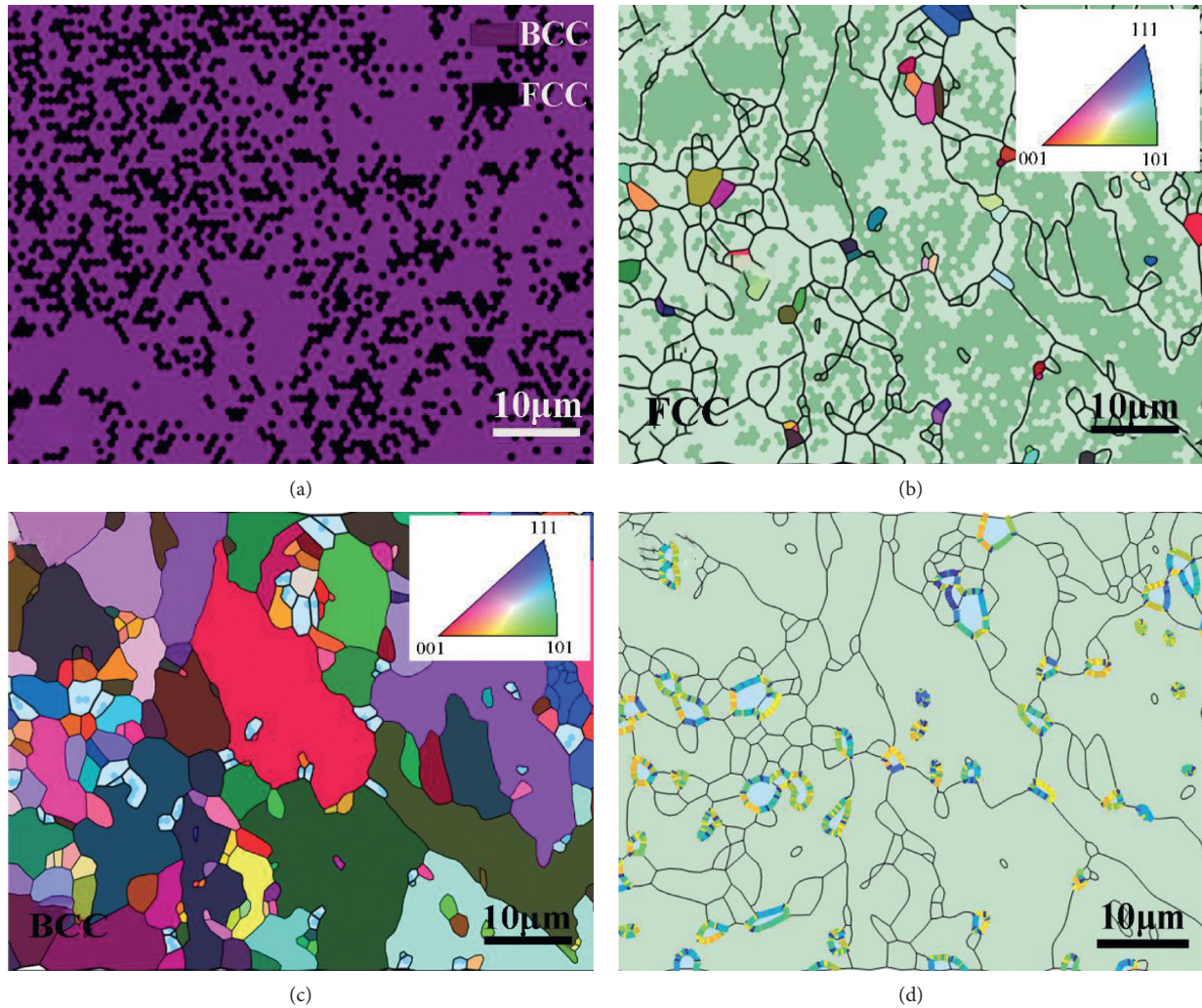


FIGURE 7: DCT at -110°C and tempering at 350°C . (a) Image of phase, (b) misorientation image of FCC, (c) misorientation image of BCC, and (d) image of grain boundary.

Figure 2, deep cryogenic treatment and tempering can reduce the low-angle grain boundary and increase the large-angle grain boundary significantly. The large-angle grain boundary was increased by 2%~5.18%, the low-angle grain boundary was decreased by 2.02%~3.32%, and the average grain size was decreased by 1.2~1.57 μm . Obviously, the large-angle grain boundary and low-angle grain boundary can be affected significantly by the deep cryogenic treatment and tempering. The data in Figure 4 clearly show that the tensile strength, yield strength, and yield ratio of the steel were improved apparently, which are consistent with the original assumption. One unexpected finding was that the elongation was decreased from 16% to 5% apparently under the cryogenic treatment and tempering. The inconsistency may be due to the large-angle grain boundary of steel accounts for a large proportion, and the uniformity of grain was affected by the cryogenic treatment and tempering. Therefore, the elongation of the steel was reduced. The previous study showed that the low temperature could improve the yield ratio, toughness, plasticity, and other

properties of steel by refining the grain size and optimizing the martensite and other microstructure [18–20]. Further study indicates that the microstructure of steel can be effectively homogenized with the thermal treatment of tempering at medium temperature, which can enhance the impact toughness and yield ratio of steel [21–23].

So far, the cryogenic treatment is mainly used for processing tool steel, and little attention has been paid to the seismic steel. The present work combines advantages of cryogenic treatment with tempering to explore the effect on the mechanical properties, such as tensile strength, elongation, yield ratio, and yield strength for the medium carbon seismic steel. The results indicated that the treatment in the present work can significantly improve the mechanical properties of steel, except the elongation. If this problem can be solved effectively, the procedure can be benefited to improve the mechanical properties of medium carbon seismic steel in future. Therefore, further study can focus on the improvement of elongation for steel with deep cryogenic treatment.

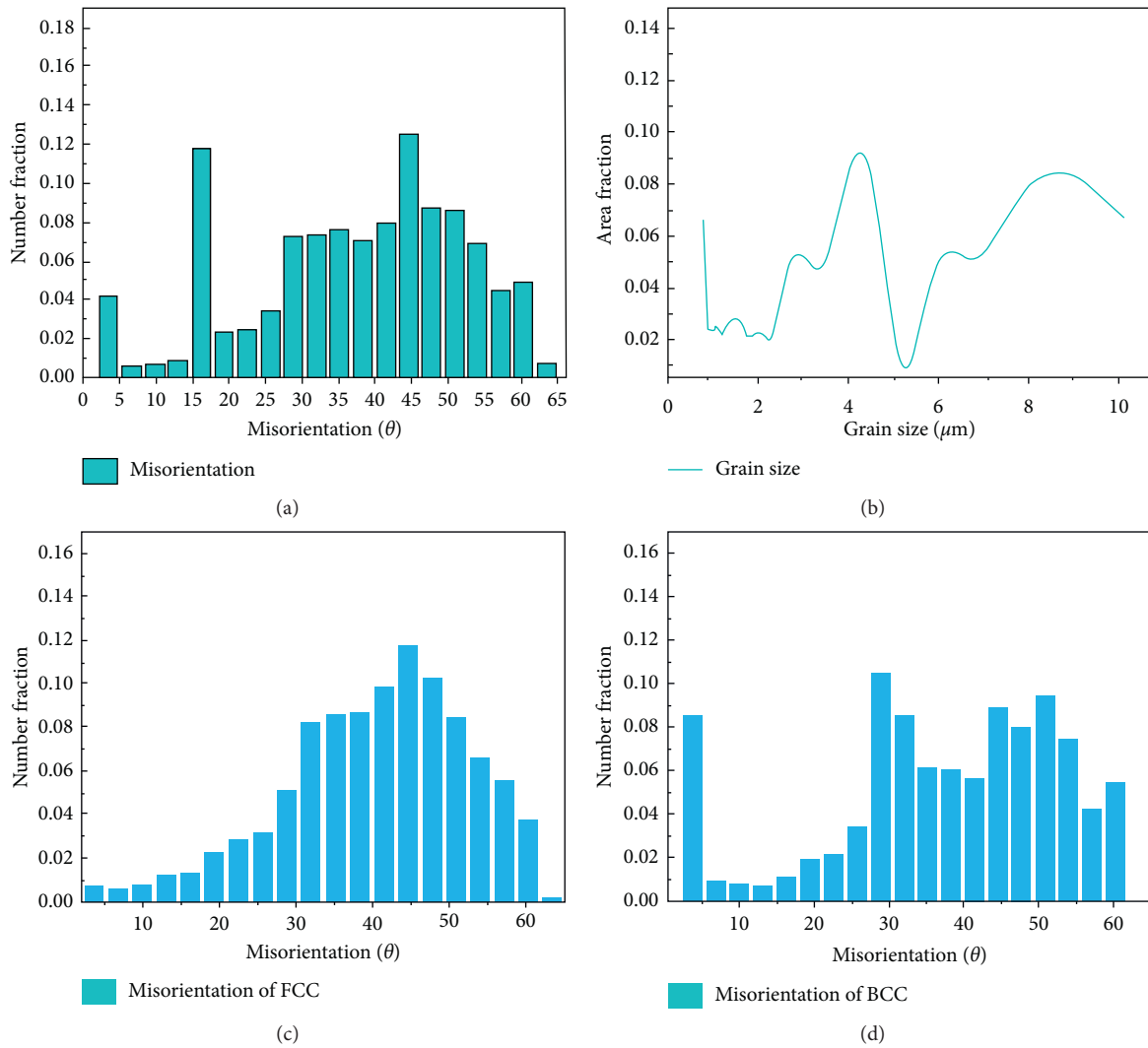


FIGURE 8: DCT at -110°C and tempering at 350°C . (a) Misorientation histogram of grain, (b) image of grain size, (c) misorientation histogram of FCC, and (d) misorientation histogram of BCC.

5. Conclusions

The 0.25C-0.80Si-1.6Mn steel was operated with deep cryogenic treatment at -60°C and -110°C and tempering at 350°C . EBSD, SEM, and EDS were used to analyze the mechanical properties and microstructure. The conclusions are as follows:

- (1) Compared with the original performance, the mechanical properties of 0.25C-0.80Si-1.6Mn steel were significantly improved by the deep cryogenic treatment at -110°C and tempering. The tensile strength and yield strength were increased from 734 MPa and 567 MPa to 904 MPa and 571 MPa, respectively. The yield ratio was decreased from 0.76 to 0.63.
- (2) The deep cryogenic treatment at -110°C and tempering at 350°C can effectively reduce the average grain size to $3.71\ \mu\text{m}$. The process is benefit to the fine grain strengthening of steel.

- (3) The performance of the steel samples under the cryogenic treatment at -60°C is not as good as the cryogenic treatment at -110°C . The phenomenon indicates that the deep cryogenic temperature should be selected below -60°C .

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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