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Fatigue fracture behavior of high-strength low-alloy steel for flexible marine riser in the high cycle fatigue regime

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Abstract

The relationship between microstructure, stress ratio, and fatigue fracture behavior of high-strength steel for flexible marine risers was investigated by microstructure characterization and fatigue test. The microstructure characteristic, S-N curve, fatigue fracture morphology, and fatigue crack morphology after quenching and tempering treatment and cyclic heat treatment, respectively, were evaluated. The results revealed that after cyclic heat treatment, the microstructure was refined considerably, the grain boundary density and the content of high-angle grain boundary increased, which inhibited the fatigue crack propagation and improved the fatigue strength. The average stress increased by increasing the stress ratio, which promoted the crack initiation and propagation; consequently, the fatigue strength and fatigue life of the tested steel decreased.

1. Introduction

A flexible marine riser is a pipeline connecting subsea wellhead and offshore platform to transmit oil and gas, which is key equipment in offshore oil and gas exploitation. A flexible marine riser is a type of composite pipe composed of a multi-layer structure containing an inner carcass layer, sealing layer, armor layer, and outer sheath layer [1–3]. Armor layer is the core layer of a flexible marine riser, which largely determines the service performance of the flexible riser. Therefore, high-strength low-alloy steel is used as the armor layer to ensure the mechanical properties of the flexible marine riser [4, 5]. The flexible marine riser is subjected to the effect of gravity and the impact of the ocean current or wave during its service [6, 7]. As a result, the high strength steel for flexible riser bears the coupling effect of axial tension and transverse repeated bending, resulting in the fatigue fracture failure. According to engineering experience, flexible marine risers are subject to large tensile stress and transverse cyclic bending stress for a long time, which can easily cause excessive local stress of the armor layer metal and fatigue failure [8, 9]. Thus, it is necessary to study the fatigue fracture behavior of high strength steel for the flexible marine riser to improve its fatigue strength and service life, and also to improve the reliability and safety of flexible riser in service.

At present, finite element simulation and numerical analysis are often used to study the mechanical characteristics and fatigue properties of high strength steels for flexible risers [10–13]. Zhang [14] investigated the impacts of axisymmetric loads and the axial stress distribution of the armor layer through theoretical and numerical analyses. Based on the theory of elasticity, O’Halloran [15] developed an analytical frictional contact model and further established an analytical fretting fatigue life prediction model of the pressure armor layer for flexible risers. Vaz [16] used finite element analysis to study the critical instability load and failure modes of armor layer steels for flexible risers. According to the results of the above study, the research on fatigue fracture behavior of high strength steels for flexible marine risers is scarce, especially on the fatigue test method.

To date, several studies have been performed on the high cycle fatigue behavior of high strength steels. Li investigated the high cycle fatigue behaviors of medium carbon pearlitic wheel steels [17]. It was found that the fatigue fractures were mainly originated from the surface matrix of specimens and the fatigue limit was slightly
improved by the grain size refinement, but was insensitive to inclusions. The fatigue fracture behavior of the as-quenched martensitic steel was studied by means of the EBSD orientation analysis [18]. The results suggested that the initial fatigue cracks along block boundaries were caused by plastic strain incompatibility between adjacent martensite blocks. Sadek [19] investigated the very high cycle fatigue behavior of automobile steel and analyzed the effect of microstructure on the fatigue fracture mechanism. Qi [20] studied the high cycle fatigue behavior of low carbon medium manganese high strength steel. The results suggested that the retained austenite in tempered martensitic microstructure significantly improved the fatigue life. The fatigue crack propagation process of low carbon martensitic steel was evaluated by Ueki [21]. In this work, the influence of microstructural inhomogeneity on fatigue crack propagation was described. Although the fatigue property of high strength steel has been extensively studied, there are few studies on crack propagation and fatigue fracture behavior of high-strength martensitic steel handled by twice quenching and tempering (QTQT treatment). For high-strength steel, cyclic heat treatment can make the microstructure uniform and refined, which will improve the comprehensive mechanical properties of high-strength steel. In order to realize the lightweight of marine riser, cyclic heat treatment (QTQT treatment) is adopted in this work to improve the strength of marine riser. Moreover, the influence of microstructure and stress ratio on fatigue fracture behavior of QTQT treated steels for flexible marine riser is barely reported. Therefore, studies on the effect of microstructure and stress ratio on fatigue fracture behavior of high strength steels for flexible riser subjected to different heat treatments are required, and also the evaluation of its influential mechanism.

In this study, the fatigue test of high strength steels for flexible marine riser was performed using a high-frequency fatigue testing machine. The stress-fatigue life (S-N) curve of the tested steel was measured and the fatigue fracture and fatigue crack were characterized and analyzed, respectively. Additionally, the effects of microstructure refinement and stress ratio on the fatigue properties of the tested steel were evaluated aiming to elucidate the influential mechanism.

2. Experimental section

2.1. Material

The chemical composition of the tested steel (modified API 5 L X100 steel) is shown in table 1. The tested steel was melted into ingots in a vacuum induction furnace and then rolled and heat-treated in the laboratory. The rolling and heat treatment process is shown in figure 1. According to figure 1, the tested steel was water-cooled to 850 °C after hot rolling in a high-temperature zone, followed by air-cooling to room temperature. The total deformation amount of hot rolling was 88% and the thickness of the hot-rolled plate was 12 mm, then the hot-rolled plate was cold rolled to 4 mm at room temperature. After cold rolling, the tested steel was treated by water quenching and tempering (QT), and cyclic heat treatment (twice quenching and tempering, QTQT), respectively. The parameters of the two heat treatment processes are shown in figure 1. For the QT treatment, the
quenching and tempering temperatures were 940 °C and 580 °C, respectively. In this study, the QTQT is a heat treatment process including the quenching and tempering treatment once more on the basis of the QT treatment. To reduce the austenite grain size, the second quenching temperature was lower than the first quenching temperature. The QT temperatures of the second QT treatment were 920 °C and 600 °C, respectively. Finally, fatigue tests were performed on the tested steel fabricated by the two different heat treatment processes.

2.2. Tensile test and fatigue test
The room-temperature tensile test was carried out on a universal tensile testing machine (WDW-300). According to the standard of ISO 6892-1:2019, the tensile specimen was a rectangular cross-section specimen with a thickness of 3 mm. The length and width of the parallel section were 60 mm and 12.5 mm, respectively. The total length of the tensile specimen was 150 mm and the tensile rate was 2 mm min\(^{-1}\). Three parallel specimens were used for each heat treatment process to reduce the experimental error. The length direction of the tensile specimen was parallel to the longitudinal direction of the steel plate. The tensile stress was applied along the length direction of the specimen during tensile test.

The fatigue test was performed on a high-frequency fatigue testing machine (GPS 100) at room temperature, according to the standard of ISO 1099-2017. Owing to the limited thickness of the tested steel plate, the fatigue specimen is a funnel-shaped sheet specimen, and its dimension is shown in figure 2. The specimen length direction was along the rolling direction of the tested steel. The vibration frequency of the fatigue test was \(\sim 105\) Hz. The stress ratio \((R)\) was 0.1 for the QT steel, and 0.1 and 0.3 for the QTQT steel, respectively. In the fatigue test, the conditional fatigue strength at \(10^7\) cycles was measured using the staircase method. The conditional fatigue strength was calculated by the following equation:

\[
\sigma_{R(N)} = \frac{1}{m} \sum_{i=1}^{n} u_i / \sigma_i
\]

where \(m\) is the total number of effective tests, \(n\) is the number of stress levels, \(\sigma_i\) is the stress level of grade \(i\) (stress amplitude, MPa), and \(u_i\) is the number of tests at the stress level of \(i\).

Through the group method, the S-N curves were measured by five levels of stress level. The conditional fatigue strength calculated by equation (1) was placed in the S-N curve as the lowest point (lowest stress level). Besides, three to six specimens were used for other four levels of stress level, and the number of specimens at each stress level increased by decreasing the stress level to reduce the discreteness of data. Finally, the measured S-N curve was piecewise linear fitted by using Origin software, and the corresponding curve was drawn according to the fitting equation.

2.3. Microstructure characterization
The morphology of microstructure, fatigue fracture morphology, and fatigue crack morphology were evaluated using an optical microscope (OM) and scanning electron microscope (SEM), respectively. The etching reagent used for microstructure observation is nitric acid alcohol solution with volume fraction of 4%. The microstructure characteristics of the tested steel were investigated by electron back-scattered diffraction (EBSD) and samples were mechanically polished and then electrolytic polished in perchloric acid alcohol solution for EBSD observation. The post-processing of EBSD data was completed by the analysis software Channel 5 of SEM. The fine structures of the microstructure were analyzed by transmission electron microscope (TEM) and energy-dispersive x-ray spectroscopy (EDS). TEM samples were electro-polished using a solution of 8% perchloric acid and alcohol.
3. Results and discussion

3.1. Microstructures and mechanical properties

Figure 3 shows the microstructure of tested steels subjected to different heat treatment processes; figures 3(a) and (b) are the OM images and figures 3(c) and (d) are the SEM images. The OM and SEM images revealed that...
The microstructure of the tested steels was tempered martensite after different heat treatment processes. The prior austenite grain boundary of the tested steel was clearly visible under the OM. When comparing the grain size of the prior austenite after different heat treatments, the grain size of the prior austenite after the QTQT treatment (19.2 μm) was much smaller than that obtained by QT treatment (12.5 μm). Figures 3(e) and (f) are TEM images of the microstructure showing that after the QT and QTQT treatments, the tempered martensite still maintained the lath shape; moreover, many precipitated particles appeared in the microstructure (marked by the red arrow). After the QTQT treatment, the width of tempered martensite lath and the size of precipitated particles decreased significantly, which was measured by Image-Pro Plus (IPP) software. The average width of martensite lath decreased from 285 nm (QT treatment) to 185 nm (QTQT treatment), indicating that the microstructure was effectively refined by the QTQT treatment. The holding temperature of the second quenching was lower than the first quenching, and thus, the prior austenite grain size was relatively smaller after second quenching. Because of the microstructure heredity, the martensite lath formed by the subsequent transformation was also relatively small. The precipitates in the microstructure after different heat treatments were analyzed by EDS and the results are shown in figures 3(g) and (h), respectively. Figure 3(g) shows that the coarse precipitates formed by QT treatment were the composite precipitation of Cr, Mo, and Nb. The morphology of precipitated phase was long strip, and its average length was about 159 nm. Figure 3(h) shows that the finely dispersed precipitates formed by QTQT treatment were the composite precipitation of (Nb, Ti)C. After QTQT heat treatment, besides forming precipitates of Cr and Mo, a large amount of Nb and Ti precipitated particles were also generated. The size of precipitates decreased obviously, most of them were spherical particles and evenly distributed in the substrate, and the average diameter of precipitates was 38 nm. The microstructure characteristics of different heat treatment processes demonstrated that the grain size clearly decreased and the microstructure became finer and more uniform after the QTQT treatment.

The mechanical properties of the tested steels subjected to different heat treatments are shown in table 2. The yield strength of the tested steel after the QTQT treatment was ∼30 MPa higher than that of the QT treatment, indicating that the strength of the tested steel significantly increased because of the microstructure refinement.

### Table 2. Mechanical properties of tested steels after different heat treatments.

| Specimen | Yield strength, MPa | Tensile strength, MPa | Total elongation, % |
|----------|---------------------|-----------------------|-------------------|
| QT       | 796                 | 876                   | 15.9              |
| QTQT     | 825                 | 902                   | 17.7              |

3.2. Effect of the microstructure refinement on the fatigue fracture behavior

Under the condition of stress ratio $R = 0.1$, the S-N curves of the tested steel subjected to different heat treatments were measured, as shown in figure 4. After the QT and QTQT treatments, the fatigue strength ($\sigma_{0.1(10^6)}$) of the tested steels was 291.6 MPa and 307.1 MPa, respectively. Figure 4 demonstrates that the fatigue strength of the tested steel significantly increased after the QTQT treatment, thus, the fatigue strength of the tested steel was significantly improved by refining the microstructure. According to the S-N curves, under the...
same stress level, the number of cycles (N) for QT treated steel were scattered, while the number of cycles for the QTQT treatment was more compact, indicating that the data dispersion after the QTQT treatment was reduced. At the same stress level, the tested steel subjected to QTQT treatment appeared more cycles, indicating that the tested steel after the cyclic heat treatment exhibited a longer fatigue life.

The fatigue fracture morphology of the tested steel after the QT treatment is shown in figure 5. Figure 5(a) is the macroscopic fracture morphology, showing the characteristics of each region of the fatigue fracture. The macroscopic morphology of the fatigue fracture was divided into fatigue crack source region, fatigue crack propagation region, and instantaneous fracture region, which corresponded to the three stages of fatigue failure, i.e., crack formation, crack propagation, and instantaneous fracture. Figure 5(b) shows that a number of stripes in the crack source region were divergent from the crack source in the interior, demonstrating that the fatigue cracks initiated on the specimen surface. In the process of fatigue, the stress concentration was formed on the surface of the specimen, which became the initiation position of the fatigue crack. After the formation of fatigue micro-crack, the crack expanded from the specimen surface to the interior, resulting in the fracture failure of the tested steel. Figure 5(c) shows that the crack propagation region was relatively flat and several secondary cracks (as indicated by the red arrow) appeared in this region. Secondary cracks effectively released the stress concentrated at the main crack tip, consumed energy, and delayed the propagation of the fatigue crack [20, 22]. As shown in figure 5(d), the instantaneous fracture region presented a static load fracture morphology, and the surface was rough and granular. The instantaneous fracture region was uneven with fracture steps and few small and shallow dimples. The cross-linking effect between the dispersed shallow dimples was not evident, indicating poor plasticity and toughness in this region [23].

Figure 6 shows the fatigue fracture morphology of the tested steel subjected to the QTQT treatment. Figure 6 reveals that the fatigue fracture morphology of the tested steel after the QTQT treatment was similar to that obtained using the QT treatment. The fatigue cracks were all initiated on the surface of the specimen and propagated inward in a divergent direction. No inclusions or coarse precipitated particles were found at the crack source, so the crack originated from the interior microstructure near the surface, which belongs to non-inclusion induced crack initiation. The cyclic stress made the microstructure extrusion and intrusion repeatedly at the boundaries, which led to the formation of microcracks [24]. The crack propagation region and secondary cracks were formed in the process of propagating into the interior of the specimen. When the fatigue crack

![Fatigue fracture morphology of the tested steel after QT treatment: (a) macroscopic fracture morphology; (b) crack source region; (c) crack propagation region; and (d) instantaneous fracture region.](image)
propagated to a certain extent, instantaneous fracture occurred, resulting in failure. Some small and shallow dimples also appeared in the instantaneous fracture region, which demonstrated a small number of dimples. The fatigue crack tip morphology of the tested steel subjected to different heat treatments is shown in figure 7. In the microstructure of the QT treatment, the fatigue cracks were relatively straight, and the cracks mainly propagated forward in the form of transgranular propagation. However, after the QTQT treatment, the deflection of fatigue cracks was larger, revealing that the microstructure presented a stronger inhibition effect on the crack propagation after the cyclic heat treatment, hindering the crack propagation. According to the previous research results [25], when the fatigue crack was initiated and propagated in the martensite structure, the fatigue crack alternated its path along the lathy boundaries and passed through the laths perpendicularly. Therefore, the lathy boundaries became the resistance to crack propagation and caused crack deflection. The QTQT treatment refined the grain size and improved the uniformity of microstructure, enhancing the

**Figure 6.** Fatigue fracture morphology of the tested steel after QTQT treatment: (a) macroscopic fracture morphology; (b) crack source region; (c) crack propagation region; (d) instantaneous fracture region.

**Figure 7.** Fatigue crack tip morphology of tested steel after different heat treatments: (a) QT; (b) QTQT.
microstructure resistance to the initiation and propagation of fatigue crack [26, 27], and effectively reducing the propagation rate of fatigue crack, and thus significantly improving the fatigue resistance of the tested steel.

The grain boundary characteristics of the tested steels were analyzed by EBSD technology and the results are shown in figure 8. Figures 8(a) and (c) show the distribution characteristics of grain boundaries. The blue lines represent the high-angle grain boundaries (≥15°) and the red lines represent the low-angle grain boundaries (2°–15°). Martensitic lath boundaries, sub-grain boundaries, CSL boundaries all contribute to the misorientation angle [28, 29]. Martensitic lath boundaries and sub-grain boundaries belong to the high-angle grain boundaries, and CSL boundaries belongs to the low-angle grain boundaries. Figures 8(b) and (d) are the statistical histograms of grain boundary distribution corresponding to figures 8(a) and (c), respectively. Figure 8 shows that the grain boundary distribution of the tested steel became dense and uniform after the QTQT treatment, indicating that the microstructure was refined and the grain boundary density significantly increased. High density grain boundaries played a significant role in hindering the dislocation slip during deformation, which effectively improved the effect of dislocation strengthening and inhibited the initiation and propagation of cracks [30]. This work confirmed previous studies on fatigue behavior of martensitic steels, that is, martensite packet and block boundaries were efficient microstructural barriers for crack growth [31]. After the QTQT treatment, the grain boundary density and the proportion of high-angle grain boundary for tested steel increased. The proportion of high-angle grain boundaries increased from 31.5% (QT) to 35.2% (QTQT), indicating that the grain size of the tested steel was refined. The high-angle grain boundary exhibited a satisfactory inhibition effect on dislocation motion and crack propagation, which was helpful to reduce the fatigue crack propagation rate and prolong the fatigue crack propagation life [32, 33]. Therefore, after the QTQT treatment, the refinement of the microstructure increased the fatigue strength significantly, prolonging its fatigue life.

The schematic illustration of fatigue crack propagation under different heat treatment processes is shown in figure 9. High-angle grain boundaries in high strength steel can effectively prevent crack propagation. Both the interface of the martensite block and the prior austenite grain boundary in tempered martensite belonged to the high-angle grain boundary and were the main factors hindering the crack propagation [34, 35]. In the process of fatigue crack propagation, prior austenite grain boundaries were effective barriers against plastic deformation and crack propagation [35]. When the crack passed through the interface of martensite block and prior austenite grain boundary, the crack deflected substantially, which would hinder the rapid propagation of cracks.
Figure 9(a) shows that the prior austenite grains of the tested steel were relatively coarse after QT treatment, and fewer martensite blocks were generated in the prior austenite. Therefore, the cracks passed through the less high-angle grain boundaries in the process of crack propagation, leading to less deflection of cracks and resulting in relatively flat cracks and less resistance to crack propagation. As shown in figure 9(b), after the QTQT treatment, the prior austenite grain was significantly refined, and the density of martensite blocks greatly increased. Consequently, fatigue cracks passed through more high-angle grain boundaries and received greater resistance in the process of propagation, producing more tortuous cracks. The refinement of the prior austenite grain effectively inhibited the crack propagation and prolonged the fatigue crack propagation life, as a result, the fatigue strength of tested steel was improved.

3.3. Effect of stress ratio (R) on the fatigue fracture behavior

Figure 10 shows the S–N curves of the tested steel after QTQT treatment at different stress ratios. The fatigue strength ($S_{0.1 \times 10^7}$) of $R = 0.1$ after cyclic heat treatment was 307.1 MPa. When $R = 0.3$, the fatigue strength of the tested steel decreased significantly and the fatigue strength ($S_{0.3 \times 10^7}$) was 210.8 MPa. By increasing the stress ratio, the average stress of the tested steel increased under the condition of $R = 0.3$. Additionally, the larger the stress ratio, the smaller stress intensity factor amplitude ($\Delta K_{th}$) corresponding to the fatigue crack propagation, and the higher the driving force of crack propagation, resulting in a high fatigue crack propagation rate [36, 37]. Therefore, a high $R$ reduces the crack propagation life. Thus, the high strength steel for the flexible marine riser exhibited a relatively low fatigue strength under the condition of $R = 0.3$.

Figure 11 shows the fatigue fracture morphology of the tested steel after the QTQT treatment under the condition of $R = 0.3$. Compared with the fracture morphology of $R = 0.1$, the fatigue fracture morphology characteristics were not significantly different. The difference was that the number of secondary cracks in the crack propagation region decreased. As shown by the red arrow in figure 11(c), a small number of secondary cracks appeared in the fatigue crack propagation region, demonstrating that the increase of stress ratio...
significantly hindered the formation of secondary cracks. The energy of fatigue crack propagation was not consumed effectively with the reduction of secondary cracks [38], resulting in a large local stress at the crack tip. Consequently, the resistance of fatigue crack propagation decreased.

Figure 12 shows the main crack morphology of QTQT steel at different stress ratios. When comparing the fatigue crack morphology at different stress ratios, the deflection of fatigue cracks was larger, and more secondary cracks appeared on the main crack under the condition of $R = 0.1$. The tortuous secondary cracks effectively released the stress at the main crack tip during the fatigue crack propagation process, hindered the main crack propagation, and consequently, improved the fatigue strength of the tested steel. Depending on the
heat treatment and strength condition of tempered martensitic steels, high cycle fatigue cracks were prone to initiate in the interior under the premise of inclusion. However, all fatigue cracks initiated on the surface of the specimen in this test due to the absence of coarse inclusions. Under the action of fatigue alternating stress, the fatigue crack was initiated on the surface by a mechanism of extrusion and intrusion [39]. Figure 12 showed that the deflection degree of fatigue crack is relatively small under the condition of \( R = 0.3 \). It indicated that the resistance to initiation and propagation of fatigue crack was relatively small, which was not conducive to improve the fatigue life.

4. Conclusions

In this study, stress-fatigue life (S-N) curves of high strength steels for flexible marine risers were obtained by a high cycle fatigue test. The effects of microstructure refinement and stress ratio on the fatigue properties of the tested steels were studied by analyzing the fatigue fracture and crack morphology under different experimental conditions. The main conclusions are the following:

1. Under the condition of stress ratio \( R = 0.1 \), the fatigue strength (expressed in stress amplitude) of the tested steel after the QT and QTQT treatments were 291.6 MPa and 307.1 MPa, respectively. For the tested steel after cyclic heat treatment, when the stress ratio changed from 0.1 to 0.3, the fatigue strength decreased significantly to 210.8 MPa.

2. The QTQT treatment effectively reduced the size of the prior austenite grain, refined the microstructure, attained a more finely and dispersedly distribution of the precipitates (Nb, Ti)C, and increased the content of high-angle grain boundary in the microstructure, which significantly inhibited the initiation and propagation of fatigue cracks; consequently, the fatigue strength and fatigue life of the tested steel were improved.

3. Under the condition of stress ratio \( R = 0.3 \), the number of secondary cracks decreased, which promoted the stress concentration at the crack tip and increased the crack propagation rate. The higher stress ratio also increased the average stress and the driving force of crack propagation. Therefore, the high strength steel for flexible marine riser exhibited a relatively lower fatigue strength.

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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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