Observation of Crack Evolution in Nickel steel of different Thermal Treatment

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Abstract—Formation and propagation of crack in quenching+tempering(QT) and quenching+ intercritical quenching+ tempering (QLT) treated Fe-9%Ni-C alloys were investigated by means of in-situ scanning electron microscope (SEM) and transmission electron microscopy (TEM).The sample treated by QLT showed better cryogenic toughness than QT treatment. High content of retained and reversed austenite in QLT-treated alloy could be tested which located not only on the high angle grain boundaries such as prior austenite boundary and packet boundary but also within the laths which was stable during loading because of protection of harder phase. Retained and reversed austenite in QT treated alloy mainly located at high angle grain boundaries and was weak in stability during loading, could not improve cryogenic toughness of the alloy comparatively.

Keywords—QLT treatment; QT treatment; In-situ tensile test; Retained and reversed austenite

I. INTRODUCTION

In recent years, clean and efficient resource such as natural gas rather than coal and oil--the conventional means is of particular concern, because of sustainable development and harmony between human society and environment. The natural gas is usually liquefied at -196°C and stored in tanker and isothermal tempered at 540°C for 1h finally cooled in water, the other was quenched at 800°C and isothermal tempered at 540°C for 1h finally cooled in water, the other was quenched at 800°C and isothermal tempered at 540°C for 1h finally cooled in water.

II. EXPERIMENTAL PROCEDURES

The steel used in this paper was melted in vacuum induction furnace in Central Iron and Steel Research Institute of China. Table 1 listed its chemical compositions. The ingot was forged into 80mm×80mm×100mm slab, and then homogenized at 1150°C for 1h, hot-rolled to 15mm thickness plate in the range of 1150°C-800°C. One plate was quenched at 800°C and isothermal tempered at 540°C for 1h finally cooled in water, the other was quenched at 800°C firstly, reheated to 650°C for 1h and quenched in water, finally isothermal tempered at 540°C for 1h and cooled in water.

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>S</th>
<th>Ni</th>
<th>P</th>
<th>N</th>
<th>Mn</th>
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<tr>
<td></td>
<td>0.1</td>
<td>0.01</td>
<td>9.1</td>
<td>0.0047</td>
<td>0.0031</td>
<td>0.4</td>
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</table>

The cylinder samples with diameter of 8mm were machined from the plates for tensile testing, and three standard impact samples with size of 10mm×10mm×55mm were machined for Charpy-V notch (CVN) impact testing at -196°C. The content of retained and reversed austenite was estimated by X-ray diffraction (XRD) comparing the
integrated peak intensities of the (002)α and (220)γ phase[7,8]. The samples were polished and etched in 7% nital solution, and the fracture propagation during tensile testing was observed by JSM-5800 in-situ scanning electron microscope (SEM) and JEM-2000 transmission electron microscope (TEM).

III. RESULTS AND DISCUSSION

The mechanical properties of QT and QLT treated specimen together with the volume fraction of retained and reversed austenite were shown in table 2. Table 2 shows that the yield strength of the specimen treated by QT is a little higher than that of QLT, the tensile strength of the specimen treated by QT was lower than that of QLT treated specimen, and the elongation of the specimen treated by QT was much lower than that of QLT treated specimen. The reason of that is closely related to the microstructure. The microstructures of two specimen are comprised of martensite matrix and different volumes of austenite. The martensite could enhance the yield strength, and the austenite could improve the plasticity. However, cryogenic toughness of the specimen treated by QLT was much higher than that of QT treated specimen, which is because the volume fraction of retained and reversed austenite was about 3 times of that of QT treated specimen. It was reported that cryogenic toughness was significantly improved by retained and reversed austenite [9,10].

<table>
<thead>
<tr>
<th>Heat treatment</th>
<th>Volume fraction</th>
<th>Yield strength</th>
<th>Tensile strength</th>
<th>Elongation</th>
<th>Akv(-196℃)/J</th>
</tr>
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<tbody>
<tr>
<td>QT</td>
<td>2.6%</td>
<td>735</td>
<td>755</td>
<td>20</td>
<td>130</td>
</tr>
<tr>
<td>QLT</td>
<td>11%</td>
<td>708</td>
<td>778</td>
<td>24</td>
<td>186</td>
</tr>
</tbody>
</table>

IV. IN-SITU SEM OF FRACTURE IN QT-TREATED SAMPLE

Microstructure consisting of lath martensite with size about 10-20μm in the sample subjected to QT treatment before tension was shown in Fig.1. It was showed that retained and reversed austenite in QT treated sample mainly located at high angle grain boundaries.

Fig.1. Microstructure of QT-treated sample before tension

At the very beginning of the tension, stress concentration formed near the notch, indicated as arrow in Fig.2a. With deformation increased, stress concentration increased and finally led to the formation of the fracture, whose direction was parallel to the maximal shear stress, 45°to tensile direction. Distinct deformation band emerged at the tip, the original irregular laths gradually rotated to the direction which was parallel to tensile direction. This was in accordance to the report[11]. Retained and reversed austenite along the grain boundary coordinated with the matrix which led to greater deformation giving rise to transformation. In contrast, retained and reversed austenite between laths was protected by adjacent laths, deformed slightly and it could alleviate the deformation. That was why retained and reversed austenite between laths showed better stability.
The propagation of the fracture was shown in Fig.3. During loading, the crack propagated along a curve course. When the loading force increased from 100N to 250N, the crack extended under radial force especially forming micro-pore in the weak zone at the tip of the crack. The formation and propagation of the micro-crack released the stress, was beneficial to slide and to form zigzag shape propagation path in shear deformation. It contained shear crack and congestion of micro-pore mechanism, which was ductile fracture, as shown in Fig.3a-c. Meanwhile, the crack extended along the direction of plastic strain concentration, 30°to the tensile direction. Morphology of the fracture tip was indicated in Fig.3d-f. The laths in certain area turned to the tensile direction, which was perpendicular to the propagation of the crack. It was difficult to rotate further, almost extended across the lath.

![Fig.2. Microstructural evolution of QT-treated sample during tension.](image)

![Fig.3. Propagation of crack during tension of QT-treated sample.](image)

The inclusions were always regarded as the crack source, nevertheless, during tension they always ruptured, formed cavity at the junction of inclusion and matrix. As the loading force increased, the cavity extended but not formed crack seen as arrow in Fig.4a. The size of the inclusion was about 3μm, affected the plasticity and the toughness. Besides, a small quantity of micro-crack formed within lath martensite and either extended through the lath or connected with main crack. This verified that the nucleation of the crack did not originate from defects of surface, but the concentration of stress.

![Fig.4. Inclusions in QT-treated sample during tension.](image)

V. IN-SITU SEM OF FRACTURE IN QLT-TREATED SAMPLE

Microstructure of sample treated by QLT before tensile test could be seen in Fig.5a. It was showed that high content of retained and reversed austenite in QLT-treated sample was located not only on the high angle grain boundaries such as prior austenite boundary and packet boundary but also within the laths. The evolution of microstructure at tip of the crack was shown in Fig.5b-d. It was indicated that the deformation behavior was similar to QT-treated sample, so were the formation of crack and the propagation. Basically it extended through laths.
plasticity greatly, resist extension of the crack indirectly. hindering the propagation of the crack, which could enhance the retained and reversed austenite showed indirect effect on toughing depended on its stability. According to the results, enhance the cryogenic toughness of 9Ni steel, the effect of the retained and reversed austenite was, it could contribute to TRIP effect during deformation process, no matter how stable austenite which enhanced the plastic of the matrix and the that the main toughing reason was the retained and reversed the value is 5 times of shear plastic deformation. We believed absorb a large amount of energy in the state of stress-strain, is reported [15] that austenite transformed into martensite can which can absorb more energy than the direct propagation. It reversed austenite can hinder the propagation of the crack as to alleviate stress concentration at crack tip and to inhibit extension of crack. It is reported [15] that austenite transformed into martensite can absorb a large amount of energy in the state of stress-strain, the value is 5 times of shear plastic deformation. We believed that the main toughing reason was the retained and reversed austenite which enhanced the plastic of the matrix and the TRIP effect during deformation process, no matter how stable the retained and reversed austenite was, it could contribute to enhance the cryogenic toughness of 9Ni steel, the effect of toughing depended on its stability. According to the results, the retained and reversed austenite showed indirect effect on hindering the propagation of the crack, which could enhance plasticity greatly, resist extension of the crack indirectly.

VI. SUMMARY

The content, stability and distribution of the retained and reversed austenite played vital role in improving cryogenic toughness (-196°C) of 9%Ni steel. The retained and reversed austenite which was stable at low temperature and distributed not only along high angle grain boundaries such as prior austenite grain boundary and packet boundary but also within lath martensite can enhance cryogenic toughness.

The inclusions were always regarded as the crack source, and the crack mainly propagate across lath martensite. The volume fraction of retained and reversed austenite in QLT-treated sample is much higher than that in QT-treated sample, which improve the strength and the toughness.

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