In situ observation of high temperature microstructure and reverted austenite evolution in 13Cr supermartensitic stainless steel

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Abstract

In situ observation of high-temperature microstructure in 13Cr supermartensitic stainless steel was investigated by the high temperature laser scanning confocal microscopy (LSCM), and the effects of tempering temperature and tempering cooling rate on the morphology and contents of reverted austenite were characterized via transmission electron microscopy (TEM), x-ray diffraction (XRD) and electron backscattered diffraction (EBSD). The results indicate that only $\alpha$ phase appeared in the XRD diffraction pattern after quenching at 1050°C, there was no retained austenite in the quenched microstructure. The martensite transformation starting temperature ($M_s$) was around 177.6°C according to the results of LSCM. The amount of reverted austenite increased first and then decreased with the tempering temperature increasing, and it reached a maximum value when the sample tempered at 620°C. With the cooling rate increasing, the amount of reverted austenite showed a sharp decrease after tempering at 620°C. The reverted austenite precipitated on martensite laths, blocks and prior austenite grain boundaries, while the misorientation angles ranged from 0° to 60° in the tempered martensite structure.

1. Introduction

The application of stainless steel is widely used in people’s daily life. There is no doubt that stainless steels play a vital role in the development of the class in steel materials. There are ferritic, austenitic and martensitic microstructures in stainless steels. The microstructures can be acquired by modifying the chemical composition and heat treatment process. The different classes of stainless steels have different properties [1]. Supermartensitic stainless steel was firstly proposed to improve the weldability of Hydrogenerator in the early 1950s. Compared with traditional martensite stainless steel, supermartensitic stainless steel has higher strength, toughness, excellent corrosion resistance and weldability [2–5]. The advantages make it an alternative to the expensive duplex stainless steels and austenitic stainless steels commonly used onshore and offshore industry [4, 6–9].

The excellent mechanical properties are strongly relied on the heat treatments in the supermartensitic stainless steel. The heat treatments have a significant influence on the strength and low temperature toughness, and the parameters of heat treatment will induce significant changes in mechanical properties [6, 7, 10–12]. The mechanical behavior is subjected by the TRIP effect and the fraction of stabilized austenite [8, 13, 14]. It should be emphasized that the main influencing factor of mechanical properties is the reverted austenite in 13Cr supermartensitic stainless steel. Therefore, the amount, morphology and distribution of the reverted austenite are of great significance to study in the work.

In order to acquire the desired reverted austenite microstructure, the austenitizing, quenching and the subsequent tempering after cooling to room temperature are essential steps. Previous studies have been done to optimize the reverted austenite by the ex situ technique [15]. It has some limitations to the research of the
reverted austenite after heat treatment. It was adopted for researching the reverted austenite by the in situ techniques, for instance, the dilatometry, high temperature x-ray diffraction and the magnetic techniques [3, 16–18]. However, the technique of dilatometry can not directly observe the phase transformation. The high-temperature x-ray diffraction technique can not accurately identify the results when the sample has texture or different grain size. The magnetic techniques have limitations for analyzing the supermartensitic stainless steel mainly used at temperatures below 100 °C [18, 19].

The high-temperature laser scanning confocal microscopy, providing an effective means for dynamic microstructure observation has been developed and utilized for the phase transformation. It can observe the changes of microstructure by the in situ technique during heating and cooling. The technique uses specially image processing software to determine the in situ phase transformation, which acquires more accurate temperature as well as close consistent the experimental results with computational simulations [20, 21]. In the present work, in situ observation of the microstructure was studied by the HTLSCM during quenching and tempering in 13Cr supermartensitic stainless steel. Meanwhile, the amount, morphology and distribution of reverted austenite were investigated combined with XRD, EBSD and TEM technique. Additionally, the influencing factors on the reverted austenite stability were analyzed in 13Cr supermartensitic stainless steel. The work will provide the theoretical and production guidance for the heat treatment process of 13Cr supermartensitic stainless steel.

2. Materials and methods

The experimental materials were adopted by a commercial super 13Cr martensitic stainless steel on the market, and the chemical composition was shown in table 1. The raw materials homogenized and then hot rolled. The samples were cut by the wire electrical discharge machining with 8 mm diameter and 3 mm thickness as an in situ observation test.

The samples were ground, polished and cleaned by ultrasonic, and then in situ observation was carried out with high-temperature laser scanning confocal microscope VL2000DX-SVF18ST. The obvious advantage is that the grain size can be detected at different times by the instrument. The heating stage of sample was initially evacuated to 1.33 × 10⁻² Pa and then argon was passed into the sample chamber. The samples heated to 1050 °C with a rate of 5 °C/s and held for 30 min, followed by cooling at a rate of 1 °C/s, 5 °C/s, 10 °C/s and 20 °C/s, which was to explore the cooling rate effect on martensite transformation. Then the sample tempered at 550 °C, 600 °C, 620 °C, 650 °C, 700 °C and cooled at different cooling rate. The heat treatment process chart is given in figure 1. The high-temperature microstructure of the samples was continuously recorded during the heat treatment process. The microstructure under the appropriate conditions was selected for the phase transformation analysis.

The treated samples were conducted by the D8 Advance x-ray diffractometer to analyze the phase components in different heat treatment. The XRD was used to determine the volume fraction of the reverted austenite. The samples were scanned in the angle range of 40° to 100° with a speed of 0.02 °s step interval with Cu Kα radiation. The volume fraction of reverted austenite was quantitatively measured by the integrated intensities. It was determined from the integrated intensities with Kα radiation. The microstructure under the appropriate conditions was selected for the phase transformation, which acquires more accurate temperature as well as close consistent the experimental results with computational simulations [20, 21]. In the present work, in situ observation of the microstructure was studied by the HTLSCM during quenching and tempering in 13Cr supermartensitic stainless steel. Meanwhile, the amount, morphology and distribution of reverted austenite were investigated combined with XRD, EBSD and TEM technique. Additionally, the influencing factors on the reverted austenite stability were analyzed in 13Cr supermartensitic stainless steel. The work will provide the theoretical and production guidance for the heat treatment process of 13Cr supermartensitic stainless steel.

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electrolyte of 6% perchloric acid solution with the voltage of 40 V and a current density of 70 A cm$^{-2}$. JEOL 2100 transmission electron microscope operating at 200 kV was carried out to investigate the reverted austenite microstructure.

3. Results and discussion

3.1. In-situ observation and analysis of quenching

The real-time dynamic observation of the martensite transformation of 13Cr supermartensitic stainless steel quenching at 1050 °C with a 5 °C/s cooling rate to room temperature is shown in figure 2. The evolution of martensite shear is discussed in this work. It can be seen that the prior austenite grains were still clearly visible from 1050 °C decreasing to 177 °C. When the temperature reached to 176.6 °C, the prior austenite grains begin to transform into martensite. Therefore, we can define the martensite starting transformation temperature is 176.6 °C. Subsequently, the large amount of martensite transformed instantaneously in figures 2(d) and (e). The typical characteristic was the shear relief of martensite transformation, and then the lath-plate occurred in the intergranular and ran through the whole prior austenite grains. There were several packets, blocks and laths within a parent austenite grain [24]. It was the lath martensite that observed in the quenched samples. When the formed martensite middle ridge penetrated towards the interior of the austenite grain, and the subsequent martensite lath nucleation was found to be slightly slow. This is because the resistance becomes larger to the surrounding area of formed martensite, which makes the martensite nucleation difficult.

The XRD pattern of 13Cr supermartensitic stainless steel quenched at 1050 °C for different cooling rate is presented in figure 3. The most interesting finding was that the diffraction peak only appears α-Fe at different cooling rates, and there was no γ-Fe diffraction peak in the XRD pattern. It can be manifested that the quenching microstructure is martensite even at slow cooling rate in the 13Cr supermartensitic stainless steel. The steel has a better hardenability due to the chemical composition of the Cr and Ni element content. It guaranteed that the reverted austenite transformed from martensite matrix when the specimens have the heat treatment of quenching and tempering, rather than austenite retained in the microstructure of the quenching process. Therefore, it is guaranteed that the microstructure obtained after quenching is only the martensite with lathe-plate characteristics, and there is no retained austenite in the microstructure.

3.2. In-situ observation and analysis of tempering

3.2.1. Effect of tempering time on reverted austenite

The reverted austenite transformed from martensite matrix when the quenched specimens during tempering process. The reverted austenite is different from residual austenite in that it is produced during the heating and holding process in the quenched sample.

The in situ observations of reverted austenite evolution during tempering at 600 °C for 60 min were indicated in figure 4. It supposed that the tempering time was 0 min when the tempering temperature arrived at 600 °C, and the end of the tempering holding time is 60 min. The tempering time has a great effect on the reverted austenite in our previous work [12]. There was an obvious change in the microstructure between martensite and reverted austenite. The laths of the martensite were still clearly visible. The surface relief in the
Martensite became blurred with the tempering time increasing. It can be inferred that the reverted austenite transformed in the tempered martensite matrix. As the tempering time increased, the martensite surface relief became flat that marked the circle in the figure. It indirectly proves that the formation of the reverted austenite occurs by diffusion mechanism, not a shear mechanism. Therefore, the reverted austenite transformed from martensite is a diffusion mechanism [25]. The longer tempering time the sample has, the more amount of reverted austenite will be acquired.

Figure 2. In-situ observation of martensite transformation process in 13Cr supermartensitic stainless steel (a) 1050 °C, (b)177.6 °C, (c)176.6 °C, (d)168.1 °C, (e)156.2 °C.

Figure 3. X-ray diffraction pattern of 13Cr supermartensitic stainless steel quenching at different cooling rate.
3.2.2. Effect of tempering temperature on reverted austenite

It is well known that the amount of reverted austenite determines the mechanical properties of 13Cr supermartensitic stainless steel \[10, 11, 26\]. The content of reverted austenite measured by XRD at different tempering temperatures is shown in figure 5. Figure 5(a) is the x-ray diffraction pattern of the specimens tempering for 1 h at different temperatures with 1 °C/s cooling rate of 13Cr super martensitic stainless steel. It can be seen that the samples have very little reverted austenite when the sample tempered at 550 °C and 700 °C. There were α phase and γ phase when the sample tempered at 600 °C, 620 °C and 650 °C. It indicates that the reverted austenite nucleated and transformed on the martensite matrix tempering at a range of temperature. The amount of reverted austenite was measured according to the integral intensity of the diffraction peak, and the result is indicated in figure 5(b). It can be seen that the amount of reverted austenite increased when the specimens tempered at the temperature in the range of 550 °C–620 °C. The specimen tempered at 620 °C had the maximum value, and the highest content of reverted austenite was 21%. With the tempering temperature increasing, the amount of the reverted austenite gradually decreased in the samples. This is because the reverted austenite transforms into martensite at a certain tempering temperature range. The content of the reverted

Figure 4. In situ observation of reverted austenite transformed at 600 °C: (a) 0 min, (b) 10 min, (c) 30 min, (d) 60 min.

Figure 5. X-ray diffraction pattern tempered at different temperature with 1 °C/s cooling rate (a) and (b) reverted austenite content at different tempering temperatures.
austenite was a small amount at a low tempering temperature, and it didn’t reach the reverted austenite formation temperature ($A_s$). Otherwise, when the tempering temperature increased to 700 °C, although the austenite content increases at high temperature, the reverted austenite is unstable and then the reverted austenite transformed to martensite during the subsequent cooling process at ambient temperature. The formation of reverted austenite is controlled by the diffusion of Ni element. It has been indicated that the stability of the reverted austenite will increase when the Ni element distributed from the surrounding martensite matrix [11, 15, 23]. As the tempering temperature increased, the solute atomic diffusion rate increase and the consequent solute redistributes in the microstructure, the Ni element concentration in the nucleation positions from the tempered matrix was reduced. It caused an increase in the $M_s$ and led to the decrease of austenite stability that cannot stabilize the $\gamma$ phase on cooling. As a result, the stability of reverted austenite deteriorates during the subsequent cooling process, resulting in the formation of fresh martensite [27].

3.2.3. Effect of cooling rate on the content of reverted austenite

Figure 6 is the in situ observation of the sample tempered at 600 °C with 1 °C/s cooling rate. Figure 6(a) is an in situ observation microstructure holding 60 min tempered at 600 °C, and figure 6(b) is the microstructure after cooling to 100 °C with 1 °C/s rate. It can be found that the martensite laths are still clearly visible by comparing with the pictures. However, there are still some differences between the pictures. As can be seen from the marked black circle in the figure, the surface relief in the microstructure became deepening when the sample tempered at 600 °C with a cooling rate to 100 °C. The reverted austenite mainly nucleated at the prior austenite grain boundary, packet and the martensite lath in the previous study [12, 28]. It can be deduced that reverted austenite transformed into new martensite during the cooling process. The result is consistent with the previous research [12].

The sample tempered at 620 °C with different cooling rates was adopted to explore the stability of the reverted austenite in the steel. The content of reverted austenite was analyzed by x-ray diffraction after the samples tempering at different cooling rate, as shown in figure 7. The intensity of the (111) crystal face pattern peak of the $\gamma$ phase decreased with the increase of cooling rate in the figure 7(a). The intensity of the $\gamma$ phase was very small when the cooling rate was 10 °C/s. The amount of reverted austenite was acquired by different tempering cooling rate in the 13Cr supermartensitic stainless steel according to the equations (1) and (2). The content of reverted austenite declines with the increase of cooling rate, as shown in figure 7(b). It is notable that the specimen has more amount of reverted austenite at a slow cooling rate than fast cooling rate. The reverted austenite content reached the maximum when the sample tempered at 620 °C with 1 °C/s cooling rate. It manifests that the reverted austenite becomes unstable in the fast-cooled sample. It can be explained that higher thermal stress and the concentration of quenched-in vacancies cause the reverted austenite unstable with the higher cooling rate in the microstructure [23]. Therefore, the reverted austenite starts to decompose and retransform into fresh martensite.

3.3. Characterization and distribution of reverted austenite

Figure 8 shows the crystallographic orientation maps of the specimens tempered at 600 °C. Figure 8(a) shows the corresponding orientation image map, the orientation of each indexed diffraction pattern is assigned a unique color. It can be seen that different colors represent different crystal orientation from the figure. The structure can be easily recognized since identical orientations are represented by the same color gradient [13]. The variants in martensite or packet in a prior austenite grain can be seen from the IPF map. Figure 8(b) is the grain boundary
distribution map, where the misorientation angle larger than 15° depicted as thick black lines are high angle grain boundaries. The low angle grain boundaries with misorientations angle ranged from 2° to 15° marked by a red line. The high-angle grain boundary mainly consists of the prior grain austenite boundary and the packet boundary of the martensite [29]. The low angle grain boundary is mainly composed of block and sub-block in the martensite. The phase distribution map of the martensite and reverted austenite of the specimen is shown in figure 8(c), where the reverted austenite and martensite are colored by red and blue respectively. It can be seen that the morphology and distribution of the reverted austenite in the map. It was apparent that the reverted austenite distributed on the martensite matrix from the figure. It reveals that reverted austenite tranforms from the martensite, and the nucleated position may be on the martensite of blocks, packet boundaries and sometimes on prior austenite grain boundaries [30]. Figure 8(d) displays that the histogram of the misorientation angles

Figure 7. X-ray diffraction pattern and reverted austenite content at different cooling rates (a) x-ray diffraction pattern at different cooling rates, (b) Reverted austenite content at different cooling rates.

Figure 8. EBSD pattern of crystal orientation distribution of Super 13Cr during tempering at 600 °C (a) the corresponding orientation image map, (b) grain boundary map, (c) phase distribution map, (d) the misorientation angles distribution.
distribution in the microstructure. It is apparent that the misorientation angles distribute at the range of 2°–15° and 50°–60°. The range of the orientation angles correspond to the low angle grain boundaries and large angle grain boundaries. In a word, the misorientation angles of the martensite ranges from 0 to 60°, the results are universal in low carbon steels [31]. The result is consistent with previous research that the reverted austenite is concentrated in the martensite with the misorientation range of 0–60° [28, 30].

The TEM microstructure tempered at 600 °C for 1 h of the 13Cr supermartensitic stainless steel is shown in figure 9. The microstructure of the samples mainly consists of tempered martensite and reverted austenite. As shown in figure 9(a) by the black arrow marked, the reverted austenite transformed from the martensite matrix as a shape of acicular-like, sometimes it precipitated at the laths or high angle grain boundaries. The dislocations provide conditions for the nucleation of the reverted austenite in the martensite matrix [6]. It guaranteed that 13Cr supermartensitic stainless steel achieved high strength and excellent toughness due to the microstructural characteristics. The reverted austenite marked the black arrow precipitated between the laths. It reveals that the growth kinetics of the reverted austenite is dominated by the diffusion of Ni in the microstructure [32, 33].

4. Conclusions

There are several conclusions obtained by HTLSCM, XRD, TEM and EBSD techniques in the present work. The following conclusions can be drawn:

1. The microstructure was martensite of 13Cr supermartensitic stainless steel quenched at different cooling rates, and there was no retained austenite. The martensite starting transformation temperature was about 177.6 °C when the sample quenched at 1050 °C with 5 °C/s cooling rate.

2. The amount of reverted austenite firstly increased and then decreased with the increasing of tempering temperature. The content of reverted austenite reached to the maximum value when the sample tempered at 620 °C. The reverted austenite became unstable and transformed into new martensite when the tempering temperature exceeded 620 °C.

3. The amount of reverted austenite decrease with increasing of cooling rate when the sample tempered at same temperature. The higher thermal stress and quenched-in vacancies make reverted austenite unstable with higher cooling rate, and it caused the reverted austenite re-transformation into new martensite.

4. The reverted austenite distributed at the martensite matrix with the range of 0–60° misorientation angle in the microstructure. The acicular-like shape of reverted austenite distributed between the martensite laths, and the dislocation in the martensite matrix provided the nucleation position.

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