



Article The Phase Transformation in a Low-Carbon 13Cr4Ni Martensitic Stainless Steel during Two-Stage Intercritical Tempering

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Abstract: The microstructure evolution of a low-carbon 13Cr4Ni martensitic stainless steel during two-stage intercritical tempering at 630 °C and 590 °C has been investigated by X-ray diffraction, thermodilatometry, and transmission electron microscopy. It was found that the amount and size of reversed austenite increase remarkably after second-stage tempering at 590 °C. However, there is no remarkable variation in the chemical composition and nucleation site of the reversed austenite during the first- and second-stage tempering. The dynamics of the phase transformation and elements distribution imply that the martensite-to-austenite phase transformation during second-stage tempering is controlled by diffusion. The unstable austenite transformation into martensite during the cooling process of the first-stage tempering induces high density dislocations and inhomogeneous element distribution, which facilitate the nucleation and growth of the reversed austenite in the second-stage intercritical tempering. Additionally, some lathy reversed austenite spheroidizes to granular during second-stage tempering.

Keywords: martensitic stainless steel; reversed austenite; intercritical tempering; phase transformation

1. Introduction

Low-carbon 13Cr4Ni martensitic stainless steel with a typical chemical composition of 0.03%C, 13%Cr, 4%Ni, and 0.5%Mo (in weight) is widely used for hydraulic turbine runners, offshore oil and nuclear industries because of its good mechanical properties and weldability [1–3]. Its good mechanical properties are related to the special microstructure. After quenching and intercritical tempering, some small-size austenite grains are retained to room temperature, which are either referred to as reversed austenite or retained austenite [4–10]. Previous research found that the reversed austenite can be induced to transform into martensite during plastic deformation and improve the good toughness and plasticity of this material [1,2,11–14], namely the transformation-induced plasticity (TRIP) effect [15–19]. Because of the determining role of reversed austenite in enhancing the toughness of 13Cr4Ni steel, many studies have investigated the formation and stability mechanism of reversed austenite during intercritical tempering [10–14,20–22]. At the macroscopic scale, high-temperature X-ray diffraction (XRD) results indicated that martensite gradually transforms into austenite with the extension of the holding time of the intercritical tempering process until the element diffusion stabilizes [8], and the growth of austenite is accompanied by the segregation of Ni [5,7,8]. The amount of reversed austenite retained to room temperature first increases with an increasing tempering temperature and then decreases to zero [3,5,7,8,10]. At the microscopic scale, it has been found that the reversed



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Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). austenite is enriched with Ni and other austenite stabilizing elements. Therefore, most of the researchers believe that the martensite-to-austenite phase transformation during intercritical tempering is controlled by diffusion. Furthermore, the reversed austenite remains at room temperature due to the enrichment of austenite stabilizing elements [5,7,8,10].

Meanwhile, the proper content of reversed austenite is the key to enhancing the plasticity and toughness of 13Cr4Ni steel [13]. Because of the limited amount of reversed austenite after one-stage intercritical tempering [10], further intercritical tempering was further performed after one-stage intercritical tempering to increase the amount of reversed austenite and dramatically optimize the strength-ductility balance [5,23]. As mentioned above, the phase transformation and microstructure evolution of 13Cr4Ni steel during one-stage intercritical tempering are relatively well understood. However, the evolution of the microstructure in two-stage tempering has not been clearly understood, although it has been successfully applied in hydraulic turbine runner castings to satisfy the demanding requirement of strength and ductility [23]. In addition, some research has indicated that not only the amount of reversed austenite but also the mechanical stability of reversed austenite is changed during two-stage tempering [6,13]. The reversed austenite obtained by proper two-stage tempering is more stable than that obtained by one-stage tempering during deformation, which induces a much better strength-ductility balance [11]. Therefore, investigating the phase transformation of 13Cr4Ni steel during two-stage intercritical tempering will provide insights into its excellent mechanical properties.

The heat treatment of 13Cr4Ni steel has some similarities to the quenching, lamellarizing, and tempering (QLT) heat treatment used in 6Ni, 9Ni cryogenic steel, and medium Mn steel [24,25]. However, there are also some essential differences between them. Generally, the lamellarizing heat treatment in QLT is a process of intercritical annealing in the upper range of the two-phase ($\alpha + \gamma$) field and the tempering heat treatment in QLT is an intercritical tempering at a lower temperature within the two-phase field [24,25]. The main effect of lamellarizing is dispersing the reversed austenite embedded in the martensite matrix by changing the nucleation site of reversed austenite from grain boundaries to lath boundaries [24]. Nevertheless, both the first- and second-stage tempering heat treatments are carried out in a lower temperature region within the $\alpha + \gamma$ region in 13Cr4Ni steel [5,23]. According to previous research, the nucleation site of reversed austenite in Fe-13%Cr-6%Ni martensitic stainless steel is determined by tempering temperature rather than the addition of first-stage tempering [8]. Therefore, the second-stage tempering may not vary the nucleation site of reversed austenite.

Although Hubackova et al. investigated the two-stage tempering of Fe-13%Cr-6%Ni steel and proposed a model to illustrate the microstructure evolution during the two-stage tempering processing [26], the phase transformation and growth of the reversed austenite during second-stage intercritical tempering are still controversial. Compared with the one-stage intercritical tempering, the original microstructure of second-stage tempering is different from the quenched martensite. After first-stage tempering, the microstructure of 13Cr4Ni steel is composed of tempered martensite, reversed austenite and quenched martensite [10]. At the same time, the element distribution is not as even as that in the quenched condition. Therefore, the inhomogeneous microstructure and element distribution will influence the nucleation and growth of reversed austenite during second-stage intercritical tempering, which will further affect the mechanical properties of the material.

In this work, the microstructures of 13Cr4Ni stainless steel after one- and two-stage tempering are characterized by XRD and TEM. And the phase transformation and microstructure evolution during the two-stage intercritical tempering are analyzed accordingly. And then, a model is proposed to illustrate the microstructure evolution according to the experimental results. In addition, another paper focusing on the effect of reversed austenite on the tensile properties of the material has been published, which illuminates the TRIP effect of the reversed austenite after different intercritical tempering heat treatments [11].

2. Experiments

The experimental material was first manufactured using an electric furnace and refined in a vacuum oxygen decarburization furnace. And then the molten steel was poured into a casting to obtain some attached test blocks with the size of 70 mm \times 100 mm \times 200 mm. One of the test blocks was used to carry out the experiments in this manuscript. The chemical composition of the experimental steel is 0.037 C, 0.28 Si, 0.75 Mn, 12.7 Cr, 4.1 Ni, 0.53 Mo (in weight percent) and Fe balance. Before the 1050 °C air quenching and intercritical tempering, the material was first annealed at 1150 °C for 20 h to eliminate the casting microsegregation. Two kinds of samples with dissimilar reversed austenite were prepared by different intercritical tempering heat treatments [23]. Sample 1 was tempered at the intercritical temperature 630 °C for 2 h. Sample 2 was tempered at 630 °C for 2 h firstly and then tempered at 590 $^{\circ}$ C for 2 h again. Previous research reveals that the A_{c1} temperature of the quenched material is about 580 °C [5]. Therefore, the quenched martensite partially transforms into austenite when the material is tempered at 630 °C and 590 °C. Additionally, some austenite retransforms into martensite during the cooling process of the intercritical tempering at 630 °C because of its limited thermal stability. In order to investigate the nucleation and growth and the thermal stability of reversed austenite during the secondary tempering stage, different cooling temperatures are designed. In the first-stage tempering at 630 °C of two-stage tempering, it will be cooled from 630 °C to different temperatures by air cooling, and then proceeded with the second-stage tempering at 590 °C. However, almost all the austenite remains at room temperature when the tempering temperature is 590 °C [5,8,10]. Consequently, the microstructure of sample 1 is composed of tempered martensite, reversed austenite, and quenched martensite, while only tempered martensite and reversed austenite are included in sample 2.

The XRD samples were electro-polished to remove the strain-affected layer after mechanical grounding. The amount of reversed austenite was calculated by comparing the diffraction intensities of ferrite and austenite peaks [10]. The thin foil sample for TEM observation were ground firstly and then polished using the twin-jet electro-polishing method with a solution of 6% nital at -22 °C. Tecnai F30 transmission electron microscope equipped with electron dispersion spectroscopy (EDS) detector and high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) detector was used to carry out the TEM observation. The dilatometric experiments were performed using Linseis RITA L78 induction heating dilatometric machine.

3. Results

3.1. The Microstructure after 630 °C One-Stage Intercritical Tempering

The XRD spectrum of the quenched low-carbon 13Cr4Ni stainless steel is shown in Figure 1, in which no austenite diffraction peaks can be observed, which indicates that the observed austenite after intercritical tempering is created by tempering rather than quenching. The XRD spectra of sample 1 and sample 2 are shown in Figure 2, which indicates that approximately 11% reversed austenite was obtained at room temperature after tempering at 630 °C. Figure 3a is a high-angle annular dark-field (HAADF) image of sample 1, showing alternating bright laths with dark laths. The HAADF image is sensitive to element distribution and the brightness of the image is approximately proportional to the element number squared. Therefore, the bright laths are expected to have higher concentrations of some heavy elements. The selected area diffraction pattern of it indicates an FCC structure of it (see the inserted picture in Figure 3a). Other than for the reversed austenite at martensite lath boundaries, little austenite grain can be observed at the prior austenite grain boundaries. These phenomena are consistent with the observation in 13%Cr-6%Ni steel [8]. It is reported that the morphology and nucleation site of the reversed austenite influenced the temperature of intercritical tempering. When the temperature of intercritical tempering is slightly higher than the A_1 temperature, the reversed austenite is lathy and lying at martensite lath boundaries. While with the temperature of intercritical tempering increasing, the reversed austenite is embedded at prior austenite or martensite packet

boundaries, and its morphology becomes granular [8]. In this study, the temperature of one-stage tempering is 630 °C, which is not high enough to induce the austenite nucleating at prior austenite or martensite packet boundaries.



Figure 1. The XRD spectrum of the 13Cr4Ni stainless steel after air quenching at 1050 °C.



Figure 2. The XRD spectra of samples 1 and 2 calibrated by silicon.

Figure 3b shows the distributions of the main elements along the red line in Figure 3a measured by EDS line scanning with 0.5 nm spot size and 2 nm step size. A parabolic distribution of Ni is observed along the scanning line. And the bright reversed austenite lath has a higher concentration of Ni (~8%) than the average concentration of Ni (4.1%) in the material. That is why the reversed austenite in the HAADF image is brighter than the matrix. In addition, the concentration of Ni in the surrounded martensite lath is a little higher than that in the matrix, showing a gradient distribution of Ni. The other elements, such as Cr, Si, Mn, and Mo are uniformly distributed. Thus, after tempering at 630 °C for 2 h, the reversed austenite is mainly lathy and embedded at the martensite lath boundaries with about 100 nm in width.



Figure 3. The HAADF image and EDS results of sample 1: (**a**) HAADF image; (**b**) EDS results of the elements distribution along the red line in (**a**).

3.2. The Microstructure after 630 °C + 590 °C Two-Stage Intercritical Tempering

Two-stage intercritical tempering at 630 °C and 590 °C increases the amount of reversed austenite to about 21% according to the XRD spectrum in Figure 2. Figure 4a displays one of the HAADF images of sample 2, in which lathy reversed austenite is dispersed into the martensite matrix. Figure 4b shows the main elements' distribution along the red line in Figure 4a measured by EDS in TEM, also indicating the enrichment of Ni in the reversed austenite in sample 2, and the highest Ni concentration is approximately 11%, which is similar to that in the reversed austenite in sample 1. The width of the lathy reversed austenite in this figure is about 170 nm, which is significantly larger than that in sample 1. Figure 5a is another HAADF image of the two-stage tempered sample 2. In contrast to Figure 4a, the reversed austenite in Figure 5a is granular and the diameter of the granular reversed austenite increases to about 350 nm. The segregation of Ni in the granular reversed austenite is also confirmed by the EDS result shown in Figure 5b. From Figures 4 and 5, it can be found that the size of the reversed austenite dramatically increases and, simultaneously, granular reversed austenite appears at the martensite lath boundaries during second-stage intercritical tempering.

Carbon is another austenite stabilizing element, which has a stronger effect than Ni. Therefore, it may also segregate into reversed austenite. Unfortunately, it is very difficult to accurately measure the concentration of carbon using EDS in TEM. The higher concentration of carbon in austenite would increase the lattice parameter of austenite. Therefore, the lattice parameters of reversed austenite in sample 1 and sample 2 are measured by XRD to evaluate the difference in the concentration of carbon between them. The XRD spectra of sample 1 and sample 2 with Si calibration are given in Figure 2. The lattice parameters of reversed austenite in sample 2 are calculated to be 3.5854 Å and 3.5860 Å, respectively. Because the other elements in the reversed austenite in the two samples are almost identical, the 0.0006 Å difference in the lattice parameter of reversed austenite in sample 1 and sample 2 is supposed to be caused by the difference in the concentration of carbon. Based on the relationship between the lattice parameter of austenite and the chemical composition given by Dyson et al. [27], the concentration of carbon in reversed austenite in sample 1 is only 0.018% higher than that in the reversed austenite in sample 1.



Figure 4. The HAADF images and EDS results of sample 2: (**a**) HAADF image of the area containing lathy austenite; and (**b**) EDS results of the elements distribution along the red line in (**a**).



Figure 5. The HAADF images and EDS results of sample 2: (**a**) HAADF image of the area containing granular austenite; (**b**) EDS results of the elements distribution along the red line in (**a**).

4. Discussion

As mentioned above, the initial microstructure of the second-stage intercritical tempering is different from that of first-stage intercritical tempering. The dash line in Figure 6 records the dilatometric curve of the material during intercritical tempering at 630 °C for 2 h. The intercritical tempering can be divided into heating, isothermal holding, and the cooling process. A remarkable shrinkage of the dilatometric curve, indicating austenization, is observed during the isothermal holding process. In addition, during the cooling process, the dilatometric curve first decreases linearly, and then deviates from the linear relationship at ~175 °C. This means that some martensite transforms into austenite during the heating and isothermal holding process. However, the austenite partially transforms back into martensite during the cooling process due to its limited stability [5,8,10]. Therefore, the material is composed of tempered martensite, reversed austenite, and quenched martensite after tempering at 630 °C. Since the A_{c1} temperature of the quenched material is approx-

imately 580 °C, only a small amount of martensite transforms into austenite during the following heating process of 590 °C second-stage tempering, it is difficult to accurately observe by dilatometric experiment. Therefore, in the dilatometric experiment the material is reheated to 1050 °C to investigate its austenization process, which is similar to the austenization during second-stage intercritical tempering. The solid line in Figure 6 displays the dilatometric curve of this process. Similarly to the quenched material, the austenization process of the tempered sample also splits into two steps. It can be clearly identified using the first-order derivative of the dilatometric curve in the transformation range (as shown in Figure 6 by the dot line). This phenomenon implies that, similarly to the martensite-to-austenite phase transformation is also controlled by diffusion during second-stage tempering [28,29]. The similar chemical compositions of the lathy austenite in sample 1 and sample 2 also indicate that the mechanism of martensite-to-austenite phase transformation during second-stage tempering.



Figure 6. The dilatometric curve of the material during intercritical tempering at 630 °C and then reheated to 1050 °C for normalization.

Despite the fact that the martensite-to-austenite phase transformation is also controlled by diffusion during second-stage intercritical tempering, there are some obvious dissimilarities, i.e., size and morphology, between the reversed austenite in sample 1 and sample 2. It can be observed that the size of reversed austenite in sample 2 is two times larger than that in sample 1 (Figures 3 and 4), although the temperature of the second-stage tempering is 40 °C lower. This phenomenon means that the growth rate of reversed austenite in second-stage tempering is larger than that in first-stage tempering. In order to investigate the factors influencing the growth of reversed austenite in second-stage tempering, a series of heat treatments are designed. In these heat treatments, the samples are cooled to different temperatures from 630 °C during first-stage tempering, and then reheated to 590 °C to conduct the second-stage tempering. According to the dashed line shown in Figure 6, the M_s and M_f temperatures of the sample tempered at 630 °C are about 200 °C and 50 °C, respectively. Therefore, when the final cooling temperatures of first-stage tempering are 150 °C, 70 °C, and 20 °C, the amount of austenite that transforms into martensite gradually increases, namely by 5%, 12%, and 21%, respectively. However, when the final cooling temperature is 300 °C, there is no martensite transformation. It was found that the amount

of reversed austenite gradually increases with the decrease in the final cooling temperature from ~5% to ~21%. The more new martensite is generated in first-stage tempering, the more reversed austenite is generated in second-stage tempering. This phenomenon illustrates that the martensite transformation during the cooling process of first-stage tempering influences the nucleation and growth of the reversed austenite during second-stage tempering. Partial austenite transforms back into martensite during the cooling process of first-stage tempering, inducing Ni- and C-enriched martensite surrounding the reversed austenite after first-stage tempering (as shown in Figure 3). During the heating process of the secondstage tempering, the new martensite containing higher amounts of Ni and C first transforms into austenite and reversed austenite grows rapidly because of its higher concentration of austenizing elements. In addition, high-density dislocations were introduced into the reversed austenite and surrounding matrix to accommodate the volume changed by the martensite transformation at first-stage tempering (Figure 6). Therefore, the dislocations, especially those in austenite, accelerate the diffusion of interstitial and substantial elements during the diffusional phase transformation in the second-stage intercritical tempering. Thus, the reversed austenite can grow rapidly, as shown in Figures 4a and 5a.

The other difference between the one-stage and two-stage tempered samples is that some granular reversed austenite appears at the martensite lath boundaries after two-stage intercritical tempering. As mentioned above, some researchers have suggested that the reversed austenite prefers to nucleate at the martensite lath boundaries with lathy morphology when the tempering temperature is low [8]. The observation of the sample tempered at 630 °C validates this conclusion. However, some granular austenite grains have been observed at the martensite lath boundaries in sample 2 after two-stage tempering at a lower temperature (Figure 5a). Moreover, the TEM dark filed image indicates that the granular austenite grains lying in one martensite block always have the same grain orientation. Figure 7 shows one of the TEM images of the two-stage tempered sample 2, which displays the spheroidizing process of reversed austenite in sample 2. In this picture, a granular reversed austenite grain has been successfully spheroidized from a lathy reversed austenite (as indicated by the solid line arrow). At the same time, some necking phenomena are present in the lathy austenite (as indicated by the dash line arrow). Therefore, it is reasonable to suppose that the granular austenite at the martensite lath boundaries is spheroidized from the lathy austenite rather than newly nucleating during second-stage intercritical tempering. The similar granular reversed austenite is also observed in the one-stage tempered material when the isothermal holding time is extended to 5 h [10]. This further confirms the spheroidizing process of lathy austenite during tempering.

Based on the above observation and analysis, the evolution of the microstructure in the investigated steel during two-stage intercritical tempering is summarized in the sketch shown in Figure 8. The quenched martensite laths in a block in the normalized 13Cr4Ni stainless steel have a similar grain orientation and a lot of dislocations (Figure 8a). During the heating and the isothermal holding process of the tempering at 630 $^{\circ}$ C, the reversed austenite nucleates at the martensite lath boundaries and gradually grows into the martensite laths (Figure 8b) by the mechanism suggested by Nakada [8]. The austenite stabilizing elements, such as C and Ni, segregate in the reversed austenite by diffusion, indicated by the grey color in Figure 8b. When the cooling process of the first-stage tempering begins, some austenite transforms back into martensite because of its low thermal stability. The martensitic phase transformation, reserving the enrichment of austenitestabilizing elements in the martensite, induces a lot of dislocations to the reversed austenite and quenched martensite to accommodate the volume change (Figure 8c). When the heating and isothermal holding process of the second-stage tempering begins, part of the new quenched martensite first retransforms into austenite and grows rapidly because of the enhanced diffusion ways provided by dislocations and initial higher concentrations of austenite-stabilizing elements (Figure 8d). Therefore, the growth rate of reversed austenite in second-stage tempering is much higher than that in the first-stage tempering, although the temperature of second-stage tempering is lower. During the isothermal holding at





Figure 7. The TEM image of two-stage tempered sample 2 containing the spheroidizing process of the reversed austenite.



Figure 8. The sketch of the microstructure evolution of 13Cr4Ni martensitic steel during two-stage tempering: (**a**) the quenched martensite laths at the room temperature; (**b**) reversed austenite forming in the heating and isothermal holding process of the tempering at 630 °C; (**c**) the microstructure during the cooling of the tempering at 630 °C; (**d**) the microstructure in the heating and isothermal holding process of the second-stage tempering at 590 °C; and (**e**) spheroidization of the reversed austenite during isothermal holding at 590 °C.

The different size and morphology of the reversed austenite in sample 1 and sample 2 have essential influence on its mechanical stability and finally affect the mechanical properties of the 13Cr4Ni martensitic steel, which is discussed in another paper [11].

5. Conclusions

- 1. Lathy reversed austenite at martensite lath boundaries is found both in sample 1 and sample 2. Meanwhile, only sample 2 has some granular austenite at the martensite lath boundaries.
- 2. The martensite-to-austenite phase transformation during the second-stage intercritical tempering is also controlled by diffusion. The chemical compositions of the reversed austenite in sample 2 are similar to that in sample 1. Both of them are rich in austenite-stabilizing elements.
- 3. The size of the reversed austenite dramatically increases and, simultaneously, granular reversed austenite forms during the second-stage intercritical tempering. The martensitic transformation of austenite during the cooling process of the first-stage tempering induces high-density dislocations and the distribution of inhomogeneous elements, which further enhance the growth of reversed austenite in second-stage intercritical tempering.

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