Tempering Effects on the Stability of Retained Austenite and Mechanical Properties in a Medium Manganese Steel

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Tempering effects on the austenite stability and mechanical properties of 0.2C–5Mn steel were investigated in the temperature range from 100°C to 600°C with 1 hour. It was found that tempering doesn’t result in a significant change of the austenite plus ferrite duplex structure, which was developed in the previous annealing through austenite reverted transformation, whereas significant decreasing of the austenite fraction and carbon concentration was found in the specimens tempered at 200°C and 500°C due to the precipitation of carbides. Correspondingly tempering slightly deteriorates the ductility when the specimens were tempered at 200°C and 500°C without effects on mechanical properties around 400°C. Based on the analysis of relationship between mechanical properties and retained austenite, it was found that the product of tensile strength to total elongation (Rm*A T) was strongly dependent on the product of volume fraction and carbon concentration of retained austenite (f A*C). Furthermore, the optimal mechanical properties with tensile strength 1 000 MPa and total elongation 40% could be obtained after tempering at 400°C with 1 hour, which means that galvanization is feasible in the 0.2C–5Mn steel with ferrite and austenite duplex structure.

KEY WORDS: microstructure; mechanical properties; TRIP effect; austenite fraction; tempering.

1. Introduction

In recent years, application of high strength and ultrahigh strength steels were greatly increased in the automobile industry based on the consideration of not only reduction of fuel consumption and exhaust emission but also the much stricter safety standards issued by governments.1–4) However increased strength generally means decreased ductility of steels, such for the IF steel with strength about 300 MPa and ductility about 45%,5) whereas for the Martensitic steels with strength about 1 500 MPa or more but ductility only about 10%.5) This decreased ductility limits the application of the high strength and ultrahigh strength steel due to their worse formability and crush properties.7) Thus the future automobile strength steel should assume a good combination of both strength and ductility.

It is well know that increasing the austenite fraction with relative stability could increase ductility effectively. So the intercritical annealing was applied to get large amount of austenite due to austenite reverted transformation (ART) from martensite, which was named ART-annealing.8–10) Recently, H. L. Yi, et al. reported the significant results about ferrite/austenite duplex C–Mn steels, which had excellent mechanical properties with ultimate strength 900 MPa and total elongation 28%.11) It was also reported in our previous study that an excellent combination of strength about 1 000 MPa and ductility about 40% could be obtained in the medium manganese steels, which was processed by ART-annealing with relative long time.12–15) During this ART-annealing process, the full martensitic structure evolved into an ultrafine lamellar austenite and ferrite duplex structure with austenite volume fraction of about 30–40%.13) The substantially increased mechanical properties were ascribed to this large fractioned metastable austenite.

It is well known that the galvanization is essential to acquire high quality automobile sheet steels, which need to be processed at 400–500°C. At present time study on the stability of this steel during galvanization process was seldom reported in literature. Furthermore, the thermal stability of the austenite stability at temperature slightly above room temperature was hardly found in literature as well, which needs to be studied for the painting process for the automobile steels. Thus tempering effects on the microstructure and mechanical in temperature large range need to be done for the medium manganese steels.

In this study tempering in the temperature range from 100 to 600°C was carried out on the ART-annealed medium manganese steel (0.2C–5Mn). The microstructure evolution during the following tempering process was measured by scanning electron microscopy (SEM) and x-rays diffractions (XRD). The variation of mechanical properties was measured through tensile test. The purpose of this study is to investigate the tempering effects on the microstructure and mechanical properties of the ART-medium manganese steel.
with ~30% retained austenite.

2. Experimental Procedure

In this study, Fe-0.2C–5Mn steels with the main alloying elements of 0.2% C and 4.72% Mn as presented in Table 1 and other trace elements were designed and prepared by high frequency induction furnace in a vacuum atmosphere. The ingots were homogenized at 1 250°C for 2 hours, forged in between 1 200–850°C into bars with diameter of ø16 mm, and finally cooled in furnace to room temperature. The critical temperatures of Ac1 and Ac3 of the designed steel were calculated based on the THERMAL-CAL software and were presented in Table 1 as well. The forged bars were processed by austenization at 675°C for 6 hours with air cooling to room temperature and then tempering with 1 hour at different temperatures (100°C, 200°C, 300°C, 400°C, 500°C and 600°C) and finally air cooling to room temperature.

Microstructures and austenite fraction of specimens were measured by scanning electron microscopy (SEM, HITACHI S-4300) and x-rays diffraction (XRD, PHILIPS APD-10). Specimens for SEM and XRD measurements were mechanically ground and polished, then etched in 2% nital and electrolytically etched in a solution of 10% chromic acid. Microstructures and austenite fraction of specimens were measured by X-ray diffractometry using Co–Ka radiation. The calculations were based on the integrated intensities of (200)\(\gamma\), (211)\(\gamma\), (220)\(\gamma\), (311)\(\gamma\), and (311)\(\alpha\) diffraction peaks. The volume fraction of retained austenite of each peak, \(V_i\), was calculated from the integrated intensities of ferrite and austenite peaks using the following Eq. (1), and the volume fraction of retained austenite, \(V_{\gamma}\), was the average of \(V_i\).\(^{16}\)

\[
V_i = \frac{1}{1 + G(I_{\alpha}/I_{\gamma})} \quad \text{......................... (1)}
\]

where \(I_{\alpha}\) and \(I_{\gamma}\) are the average integrated intensity obtained at (200)\(\alpha\), (220)\(\gamma\), (311)\(\gamma\), and (211)\(\alpha\), diffraction peaks. G-value for each peak was presented as follow, 2.5 for \(I_{200}\gamma/I_{200}\alpha\) 1.38 for \(I_{200}\gamma/I_{220}\alpha\), 2.02 for \(I_{200}\gamma/I_{311}\gamma\), 1.19 for \(I_{211}\gamma/I_{220}\gamma\), 0.06 for \(I_{211}\alpha/I_{220}\gamma\), and 0.96 for \(I_{211}\alpha/I_{311}\gamma\).

The carbon concentration of austenite was calculated from the integrated intensity of austenite peaks using the following equation.\(^{17}\)

\[
a_{\gamma} = 3.556 + 0.0453X_C + 0.00095X_{Mn} \quad \text{......................... (2)}
\]

where \(X_C\), \(X_{Mn}\) (wt. %) are the carbon and manganese concentration of retained austenite, \(X_{Mn}\) is nearly used 1.5 times of the manganese concentration in steels.\(^{18}\) And \(X_{Mn} = 9\%\) is taken in this calculation because the manganese concentration is about 9% in austenite phase as reported in our previous paper.\(^{14}\) \(a_{\gamma}\) is the lattice parameter, calculated from Eqs. (3)–(4).\(^{19}\)

\[
a_{\gamma} = (\lambda/2 \times \sin \theta) \times \sqrt{h^2 + k^2 + l^2} \quad \text{......................... (3)}
\]

\[
d_{\text{hit}} = \lambda/2 \times \sin \theta \quad \text{......................... (4)}
\]

where \(\lambda\) is the X-ray wavelength of cobalt target, \(\theta\) is the diffraction angle, \(d_{\text{hit}}\) is the interplanar spacing, and \(h, k, l\) are the lattice parameters of (220)\(\gamma\) and (311)\(\gamma\).

Uniaxial tension test was performed on the dog-bone shaped specimens with gauge length of 25 mm and diameter of 5 mm at a strain rate of ~10\(^{-3}\)/s in an Instron machine (WE-300) at room temperature. Based on the tensile test, the tensile strength (Rm), the yield strength (Rp0.2), the total elongation (At), the uniform elongation (Au), reduction of area (Z), and the stress-strain curves were recorded.

3. Results

3.1. Microstructure Evolution during Following Tempering Process

The microstructure of 0.2C–5Mn steel austenitized firstly at 675°C with 30 minutes, then oil quenched and followed by ART-annealing at 650°C with 6 hours was examined by SEM as shown in Fig. 1. It can be seen that the lamellar typed structure with white lath (austenite) and dark lath (ferrite) could be easily distinguished. The thicknesses of both austenite laths and ferrite laths are lower than 0.35 μm, thus this structure is really an ultrafine lamellar duplex structure. In addition, apart from the lath type ferrite and austenite, rod-typed carbides with white color precipitated in the ferrite lathes could be easily observed in Fig. 1 and no carbides could be found in the austenite laths (long and thin white phase).

The microstructure evolution of 0.2C–5Mn steel as a function of tempering temperature was given in Fig. 2 to examine the tempering effects on the microstructure. In the tempering process (Figs. 2(a)–2(f)), it is shown that the microstructure remained lamellar structure, and the microstructure is nearly similar during low temperatures tempering. It also can be seen that much more carbides precipitated in the ferrite lathes in the tempered specimens (Fig. 2) than that in the ART-annealed specimen (Fig. 1). In the tempered specimens, much less amount of carbides could be seen in

Table 1. Chemical composition (wt. %) and Ac1, Ac3 of the steel in this study.

| C   | Mn | P   | S   | N   | Ac1 | Ac3 |
|-----|----|-----|-----|-----|-----|-----|
| 0.2 | 4.72 | 0.008 | 0.002 | 0.003 | 630°C | 720°C |

Fig. 1. Ultrafine lamellar ferrite and austenite duplex microstructure of 0.2C–5Mn steel firstly austenitized at 675°C with 30 minutes, then oil quenched and followed by ART-annealing at 650°C with 6 hours.
the specimens tempered at 400°C and 600°C than others as shown in Fig. 2. It can be seen in Fig. 2(d), the amount of carbides precipitation decreased, and fine lamellar structure was clear during 400°C tempering process. With increasing tempering temperature up to 500°C, the microstructure began to become thick with large quantity of carbides precipitation. However, the specimen tempered at 600°C had smaller amount of carbides.

Comparison with the reported results about microstructure during ART-annealing, the dominant heat treatment in this study is austenization and annealing in the intercritical region (between Ac1 and Ac3), therefore the original microstructure of the steels should consist of austenite laths and ferrite laths, bright-laths should be austenite laths, and black-laths should be ferrite laths. Furthermore, with the following tempering, the phenomena of precipitation or dissolving of carbides and decomposition of austenite should exist and could be observed in the studied steels.

3.2. Variation of Austenite Volume Fraction and Carbon Concentrate during Tempering Process

The volume fraction of retained austenite of the studied steels was measured by XRD and the results were presented in Fig. 3. It can be seen that the initial austenite volume fraction of specimens with different temperatures tempering first decreases from ~38% to ~29% and then increases to ~35% when the tempering temperature lower than 400°C, finally fluctuates between ~28% and ~31%, moreover, the austenite fraction of steel tempered at 500°C is minimum.
Generally, austenite fraction increases with decrease of carbon concentrate. The carbon content $C_\gamma$ of new developed austenite, which was calculated based on the XRD data, was also shown in Fig. 3. It can be seen that carbon content first decreases from 0.666% to 0.657%, then goes up to the maximum 0.676% when the tempering temperatures ranges from 100°C to 400°C. However, with the tempering temperature up to 500°C, the carbon content drops to the minimum 0.536%, and finally increases to 0.641% when the tempering temperature is 600°C.

3.3. Mechanical Properties of Steels Processed under Different Heat Treatment Conditions

Stress-strain curves of the samples under different heat-treatment conditions were shown in Figs. 4(a) and 4(b). From Fig. 4, it can be seen that samples continuously yield and flow curves are characterized by intensive serrations or jerk flow feature, indicating dynamic strain aging and strong localized deformation during tension test, whereas the intensive serrations decrease when the tempering temperature increases, except for the sample tempered at 400°C. In Fig. 4, the inserted figure illustrates the serrated flow behavior observed in the samples tempered at 400°C. In Fig. 4(a), it is found that the ductility of ART-annealed sample or tempered sample at 400°C is better than that of others, and the total elongation is about 40% ± 3%. On the basis of equations $\sigma_\text{T} = (1+\varepsilon_\text{T})\sigma_\text{E}$ and $\varepsilon_\text{T} = \ln(1+\varepsilon_\text{E})$, the engineering stress-strain curves were converted to true stress-strain curves and given in Fig. 4(b). It can be seen from the true stress-strain curves that the ultimate tensile stress of sample tempered at 400°C increases up to 1 340 MPa ± 30 MPa, which is higher than that of other. In addition, the work hardening behavior of the sample tempered at 500°C is fairly different from others, because the large amount of austenite rapidly transformed into martensite due to poor stability of austenite, which decreased the dynamic transformation velocity.

In order to illustrate the effect of tempering on the mechanical properties, the tensile stress (Rm), yield stress (Rp0.2), total elongation (At) and uniform elongation (Au) were measured from the stress-strain curves (Fig. 4) and were presented as a function of tempering temperature in Fig. 5. When tempering temperature is lower than 300°C, the tensile stress first increases from 975 MPa to 1 030 MPa and then drops to 965 MPa, and the yield stress first decreases from 620 MPa to 565 MPa and then go up to 630 MPa. However, with the increasing of tempering temperature, the tensile stress began to go up to 1 020 MPa and lastly decreases to 982 MPa, and the yield stress decreases to 530 MPa and lastly increases to 620 MPa. Furthermore, total elongation (At) and uniform elongation (Au) first decreases and then increase to the maximum with the tempering temperate going up to 400°C. However, total elongation (At) and uniform elongation (Au) drop to the minimum and finally increases during tempering process at 500°C and 600°C. It can be seen from Fig. 5 that tempering deteriorates the ductility significantly at 200°C and 500°C but almost has no effects on the mechanical properties at 300–400°C, which means that galvanization is feasible in the 0.2C–5Mn steel with ferrite and austenite duplex structure.

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![Stress-strain curves](image1.png)

**Fig. 4.** Stress-strain curves of the samples under different heat-treatment conditions (a) Engineering stress-strain curves, (b) true stress-strain curves.

![Mechanical properties](image2.png)

**Fig. 5.** Mechanical properties of steels processed at different conditions (a) tensile and yield stress (Rm/Rp0.2) as a function of tempering temperature and (b) total and uniform elongation as a function of tempering temperature.
4. Discussion

4.1. Precipitation and Dissolving of Carbides and Variation of Austenite Fraction during the Following Tempering Process

As shown in Figs. 1–2, the microstructure evolution of the studied steel was distinctly affected by the different following tempering conditions (i.e., tempered at 100°C–600°C). On the basis of initial austenite phase (~38%) and martensite laths, the carbides dissolved and precipitated in the matrix and the austenite phase continually transformed during the following tempering process. As presented in Fig. 3 that retained austenite fractions of the specimens tempered at different temperatures are ~30% and ~35%, due to large original austenite fraction (~38%) in the as-annealing specimen. This illustrates that the austenite phase changed during the different following tempering processes, with the precipitation and dissolving of carbides, which assumed the carbon transfer by the partial phase transformation from martensite to austenite.

With increasing of tempering temperature from 100°C to 200°C, the dissolved carbides gradually precipitated from the matrix, so the increase of carbides could be seen in Figs. 2(a) and 2(b). In the meantime, the austenite volume fraction and carbon content of the specimens under different heat treatment conditions decreased as shown in Fig. 3. When the tempering temperatures ranged from 300°C to 400°C, the carbides began to decomposed, which resulted in the austenite stabilizing elements such as carbon and manganese entering into the austenite, thus relatively few carbides could be discovered in Figs. 2(c) and 2(d), furthermore, the carbon concentrate increased from 0.656% to 0.676%. However, with increasing the tempering temperature up to 500°C, Fig. 2(e) showed that the amount of carbides increased, ascribed to decomposition of retained austenite by a diffusion mechanism accompanying more precipitation of carbides.24–26) or precipitation of carbides from austenite, which reduced austenite stability, resulting in transformation from austenite to martensite during cooling process,19,27) thus the volume fraction and carbon concentrate of retained austenite drastically decreased to minimum as presented in Fig. 3. Tempering at a temperature of 600°C, the partitioning of carbon into austenite,28) which was due to dissolving of carbides, improved austenite stability, leading to increase of retained austenite and carbon concentrate as shown in Fig. 3.

From Fig. 3, tempering at a temperature of 100–300°C, the carbon concentrate slightly fluctuated, which indicated that the amount of carbides was almost the same. As shown in Figs. 2(a)–2(c), the carbides always kept the fine rod morphology and the dislocation density was nearly unchanged during tempering process. When the tempering temperature up to 400°C, the amount of carbides obviously decreased and less granular carbides appeared instead of rod carbides (Fig. 2(d)), resulting in the increasing of the carbon concentrate in Fig. 3. However, in the microstructure of sample tempered at 500°C (Fig. 2(e)), the rod carbides precipitated again and the size became thicker than that of samples tempered at other temperatures, which led the carbon concentrate to sharp decrease. Finally tempered at 600°C, the dislocation density of carbides gradually decreased and the carbides also became smaller than that tempered at 500°C (shown in Fig. 2(f)).

4.2. Dependence of Mechanical Properties on the Volume Fraction and Stability of Austenite

If the results of Figs. 3 and 5 are compared, it can be seen that the variation of tensile stress and elongation with the changing of tempering temperature is nearly similar, i.e.,
a significant dependence of tensile stress and elongation on the austenite volume fraction. In order to investigate the relationship between the volume fraction and stability of retained austenite and strength and ductility of the studied steels, the tensile stress/yield stress, true tensile stress/yield stress, total elongation/uniform elongation and product of tensile strength to total elongation/uniform elongation were presented as a function of the austenite fraction in Fig. 6. From Fig. 6(a), the tensile stress ranges from 965 MPa to 1030 MPa with the changing of the product of the volume fraction and carbon concentration of retained austenite. Furthermore, with the increase of the product of austenite fraction and carbon concentration, the tensile stress significantly increased as clearly shown in Fig. 6(b). However, the yield stress fluctuates slightly. The dependence of ductility on the product of austenite fraction and carbon concentrate was presented in Fig. 6(c), it can be distinctly seen that both total elongation and uniform elongation increase with the product of austenite fraction and carbon concentrate. The variation trend of plasticity with the changing of the product of austenite fraction and carbon concentrate illustrates that the plasticity of the studied steel is mainly caused by martensitic transformation from the metastable austenite phase during plastic deformation, which is called transformation induced plasticity (TRIP) effect.\(^{29}\) However, the large austenite fraction does not always correspond to high ductility, because the TRIP effect is also relative to the mechanical stability of retained austenite during deformation. Though the volume fraction of retained austenite of the as-annealed steel is \(~38\)%,

The plasticity is not more excellent than that of the steels processed by 300°C and 400°C tempering, which may be importantly ascribe to the decrease of austenite mechanical stability. Furthermore, the tensile strength of the steels processed by tempering at 500°C is higher than that of other, but the ductility is much worse than that of other. Because small volume fraction of retained austenite and low mechanical stability lead to rapid phase transformation from austenite to martensite at the early stage of deformation, the uniform elongation is inferior to that of steels tempered at other temperatures. Figure 6(d) shows the variation of the product of tensile strength to elongation as the tempering temperature. In general, the product linearly increases with increasing of the austenite fraction. It illustrates that TRIP effect is the critical factor of improving the mechanical properties, especially the uniform elongation, which is mainly due to the large amount of austenite with relatively high mechanical stability, which ensures continuous and progressive martensitic transformation from retained austenite.

5. Conclusion

The following heat treatment effects on the microstructure and mechanical properties of 0.2C–5Mn steels were investigated in this study. The results were summarized as follows.

(1) Lamellar duplex structure is nearly remained during the tempering process, but the carbides begin to precipitate in the ferrite lath, especially at 200°C and 500°C, which led to reduction of volume fraction and carbon content of retained austenite. However the large fractioned austenite (\(~35\)% ) was still kept in the studied steel after tempering at 400°C with 1 hour.

(2) Tempering deteriorates the ductility significantly at 200°C and 500°C, but has no effects when tempered at other temperature. After tempering at 400°C with 1 hour, an excellent mechanical property of tensile strength \(~1000\) MPa and total elongation \(~40\)% could be obtained. It is very interesting that the following tempering at 400°C for 1 hour can also obtain large amount of retained austenite, moreover, a balance of strength and ductility.

(3) Based on the analysis of dependence of mechanical properties on retained austenite, it could be found that total elongation was not only dependent on the austenite volume fraction but its carbon concentration. Thus the total elongation was found to be proportional to the product of austenite volume fraction and its concentration.

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