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Effect of tempering on microstructure and mechanical properties of 3Mn-Si-Ni martensitic steel

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Abstract

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Keywords: Martensitic steel; tempering martensitic embrittlement; carbides; impact toughness.

1 Introduction

It is well known that the tempering of martensitic steel is complicated, accompanying with carbides precipitation and recovery of dislocation structures in carbon-supersaturated martensitic laths^[1, 2]. This complicated process generally includes three stages^[1, 3]. At the first stage, transition carbides such as epsilon (ϵ) carbide^[4] and eta (η) carbide^[5] precipitate in martensitic crystals typically when the tempering temperature is between 100 and 200 °C. At the second stage, the transformation of residual austenite to a mixture of cementite and ferrite occurs at temperatures typically between 200 and 300 °C^[1, 3]. At the third stage, transition carbides are replaced by cementite (θ), usually when the tempering temperature extends to ferrite-to-austenite transformation start temperature (A_{c1})^[1, 3].

High strength martensitic steels, produced by quenching and tempering, are susceptible to embrittlement after tempering at certain temperatures ^[6, 7]. This phenomenon is called as tempered martensite embrittlement (TME), which has been attributed to the following reasons. Firstly, TME relates to the sequence of carbide precipitation ^[1, 8, 9]. Klinger *et al.* ^[8] found that embrittlement was concurrent with the formation of platelet cementite (which replaced ϵ carbide). Secondly, TME is ascribed to the decomposition of residual austenite between martensitic laths ^[1, 9, 10]. Horn *et al.* ^[9] reported a severe embrittlement in AISI4340 steel when film residual austenite between martensitic laths thermally decomposed to cementite and ferrite after tempering at ~ 275 °C. In addition, mechanically induced transformation to martensite during loading also contributed to this embrittlement ^[1, 9]. Thirdly, segregation of impurity elements (such as phosphorus) along prior austenite grain boundaries during austenitization facilitates embrittlement fracture in an intergranular mode ^[9, 11, 12]. TME has been traditionally determined as the reason of a sharp decrease in Charpy V-notch impact energy at ambient temperature and an increase in the ductile-to-brittle transition temperature after tempering at different temperatures. In addition, it has been historically associated with brittle fracture ^[9, 10, 13 - 17], such as intergranular fracture ^[13, 15, 17], cleavage fracture ^[10, 18], quasi-cleavage ^[14], or martensite translath fracture ^[19-21].

At present, most of developed high-strength martensitic steels (such as examples listed in Table 1) are very expensive, because nickel (Ni), chromium (Cr) and molybdenum (Mo) were added as major alloying elements to increase austenite hardenability ^[22, 23]. Alternatively, manganese (Mn) is much cheaper than Ni and Mo, and it has similar effects on the austenite-to-ferrite transformation ^[24, 25]. Noticeably, Bolton *et al.* ^[25] studied the substitution of Mn for Ni using a series of low-carbon Fe-Mn alloys. They found that Mn could only partially substitute for Ni because Fe-Mn alloys were brittle when Ni was absent. Based on the above reasons, a steel containing 2.83 wt. % Mn (much higher than the Mn content in Table 1), 1.2 wt. %Ni (less than the Ni content in Table 1) and 1.2 wt. %Si has been designed by present authors ^[26]. The tensile strength and V notch impact toughness of this designed steel after tempering at 230 °C were 1550 MPa and 91.5 J, respectively ^[26]. Compared with mechanical properties listed in Table 1, the impact toughness greatly increased with little decrease in tensile strength. Importantly, the cost of raw material has been greatly reduced due to substitution of Mn for Ni and Mo.

With regards to fundamental aspect, this paper systematically studied the evolution of microstructures, tensile properties and impact energy with tempering temperature in this designed 3Mn-Si-Ni martensitic steel. Furthermore, the mechanism of tempering embrittlement was discussed based on microstructure characterization and simulated equilibrium phase diagram using Thermo-Calc.

Table 1. The chemical compositions (wt. %) and optimal mechanical properties of commercial high-strength martensitic steels ^[22].

	C	Si	Mn	Cr	Ni	Mo	Tensile strength (R_m)/MPa	Impact toughness (Akv)/J
30CrMnSiNi2A	0.26~0.33	0.90~1.20	1.00~1.30	0.90~1.20	1.40~1.80	--	≥ 1600	≥ 60
AMS6434	0.31~0.38	0.20~0.35	0.60~0.80	0.65~0.90	1.65~2.00	0.30~0.40		
4340	0.38~0.43	0.20~0.35	0.60~0.80	0.70~0.90	1.65~2.00	0.20~0.30	1980	20
300M	0.41~0.46	1.45~1.80	0.65~0.90	0.65~0.95	1.60~2.00	0.30~0.40	2050	24.4

2 Experimental

2.1 Material

The studied steel (3Mn-Si-Ni steel) was a high strength and low alloy martensitic steel and its chemical composition is listed in Table 2. The steel was received as hot-rolled bar in fully annealed condition. Before heat-treatment, as-received material was hot forged to 50 mm thicknesses, slow-cooled to 680 °C, and annealed at this temperature in order to achieve good machinability.

Table 2. Chemical compositions of the 3Mn-Si-Ni steel (wt. %).

C	Si	Mn	Ni	Ti	Cr	Mo	Cu	P	S	Fe
0.19	1.19	2.83	1.24	0.049	0.031	<0.01	0.013	<0.016	<0.0045	Bal.

2.2 Continuous cooling transformation diagram

The continuous cooling transformation (CCT) diagram was determined using dilatometry tests, metallographic examinations and Vickers hardness tests. A Formastor-Digital deformation dilatometer was used to measure ferrite-to-austenite transformation start (A_{c1}) and finish (A_{c3}) temperatures, as well as bainite transformation start temperature and martensite transformation start temperature (M_s). The cylindrical specimen ($\phi 3 \times 10 \text{ mm}^2$) was put between SiN_2 anvils in a vacuum chamber, heated to 900 °C at 5 °C/s and held at this temperature for 600 s followed by cooling at different cooling rates between 0.05 and 20 °C/s. The Vicker hardness was measured under a load of 100 g.

2.3 Heat treatment

A ceramic fiber muffle furnace was used for heat treatments and Pt/Pt-Rh thermocouples were used for temperature monitoring. The fluctuation of temperature in the furnace is within 2 °C while the temperature difference along the sample ($10 \times 10 \times 55 \text{ mm}^3$) is within 2 °C. The samples were austenitized at 900 °C for 40 min followed by oil-quenching and then were tempered at 180, 200, 230, 250, 320, 400, 480, 550, 600 or 650 °C for 120 min followed by air-cooling.

2.4 Microstructural observation and phase equilibrium simulation

The microstructure was evaluated by optical microscopy (OM), scanning electron microscope (SEM) and transmission electron microscope (TEM). For OM and SEM analysis, 2 vol. % nital was used as the etching agent, which can distinguish ferrite, bainite, and martensite. TEM characterization was carried out

using Hitachi H8100 TEM operated at 200 kV. ϕ 3 mm discs were prepared by twin-jet electro-polishing in a solution of 100 ml HClO_4 + 900 ml CH_3COOH .

Phase equilibrium was simulated using Thermo-Calc (TCFE6, iron base database) under the principle of minimum Gibbs energy [27, 28]. A systematic database with a set of reasonable and self-consistent thermodynamic parameters has been constructed in the Thermo-Calc based on general thermodynamic models.

2.5. Mechanical Properties

Tensile specimens were machined into a ϕ 5 mm cylinder with a gauge length of 25 mm. Tensile tests were performed using a MTS810 tensile machine. A constant cross-head speed of 0.5 mm/min in the elastic region was used, followed by a constant speed of 10 mm/min in the plastic region. Impact specimens were machined into $10 \times 10 \times 55 \text{ mm}^3$ blocks with a V-shape notch. Impact tests were conducted using a JB-30B pendulum machine based on ASTM Standard E-23.

3 Results

3.1 Continuous cooling transformation diagram

CCT diagram for studied steel is illustrated in Fig. 1. The cooling rates were 0.05, 0.1, 0.2, 0.3, 0.4, 0.5, 1, 2, 4, 6, 8, 15 and 20 $^\circ\text{C/s}$ from right to left. Only bainite and martensite transformation were observed. It is because high content of alloy elements (such as 2.83 % Mn, 1.2 %Ni, 1.19%Si and 0.031 % Cr) significantly enhance hardenability, which greatly prevents the formation of pearlite and ferrite and also depresses bainite formation. As can be seen, cooling at a rate faster than 0.5 $^\circ\text{C/s}$, which is called as the critical cooling rate, will produce a fully martensite structure. For example, Fig. 2 shows typical optical microstructures after cooling at rates of 8 $^\circ\text{C/s}$ (Fig. 2 (a)) and 2 $^\circ\text{C/s}$ (Fig. 2 (b)), which consist of martensitic packets with parallel alignment of martensitic laths. Cooling at 2 $^\circ\text{C/s}$ produced larger packet size than those after cooling at 8 $^\circ\text{C/s}$.

Figure 1. CCT diagram for the studied steel.

Figure 2. Optical micrographs after cooling at (a) 8 °C/s and (b) 2 °C/s from 900 °C.

3.3 Microstructures after tempering

Fig. 3 shows selected SEM morphologies after tempering between 230 and 650 °C. Martensitic laths within the same prior austenite grain grouped themselves into packets, as clearly shown in Figs. 3 (a) and (c). With an increase in tempering temperature from 230 to 320 °C, the parallel alignment of the laths turned “blurred” (c.f. Figs. 3 (a) and (b)). However, a sharp microstructure was observed again when the tempering temperature increased to 550 °C, probably due to carbides precipitation between martensitic laths (which was demonstrated by TEM in Fig. 6). With an increase in tempering temperature up to 650 °C, the microstructure turned “blurred” again, which may be ascribed to strong recovery in martensitic laths and parts of recrystallization (which were observed by TEM in Fig. 7).

Figure 3. Microstructures after tempering at different temperatures: (a) 230°C; (b) 320°C; (c) 550°C ; (d) 650°C.

With respect to detailed microstructure, corresponding TEM characterization was carried out after tempering at 230, 320, 550 and 650 °C. When the tempering temperature was 230 °C, large dislocation density was observed in martensitic laths (Fig. 4(a)) and very fine transition carbides precipitated in martensitic laths (Fig. 4(b)). The carbides, which were identified as ϵ -carbide by selected diffraction pattern (SAD) illustrated in Fig. 4(b), were short rod-like shape approximately with a length of 100 nm and a width of 5 nm. The ϵ -carbides presented perpendicular to the martensitic laths almost along the same direction. Noticeably, residual austenite was detected between martensitic laths (Fig. 4(b)).

Figure 4. TEM micrographs after tempering at 230 °C: (a) bright image showing high dislocation density within martensitic laths; (b) residual austenite between martensitic laths and short rod-like shape carbides within martensitic laths. *M* is martensite, *RA* is residual austenite, ϵ is epsilon carbide.

When the tempering temperature increased to 320°C, parallel martensitic laths were still observed as shown in Fig. 5. However, the dislocation density significantly decreased (c.f. Fig. 4(a) and Fig. 5). Instead of ϵ -carbide, granular cementite (Fe_3C) (distinguished by SAD in Fig. 5(b)) precipitated within martensitic laths, which is far more stable than ϵ -carbide. Its diameter ranged from 5 to 10 nm. Furthermore, residual austenite between martensitic laths transformed to a mixture of cementite and ferrite, which is illustrated by the arrow in Fig.5 (b).

Figure 5.TEM micrographs after tempering at 320°C:(a) recovered martensitic laths; (b) local magnification of circled part in (a) showing cementite and decomposition of residual austenite.

With an increase in tempering temperature to 550 °C, Fig. 6 shows that the boundaries between martensitic laths became blurred and even disappeared due to the significant recovery. Besides, some sub-grains were observed, as shown in Fig. 6(b). Many cementite precipitated along boundaries of martensitic lath. Especially, continuous carbides were observed in Fig. 6(b), probably resulting from the decomposition of residual austenite. The size of cementite ranged from 20 to 50 nm, which was much larger than those after tempering at 320 °C.

Figure 6.TEM micrographs after tempering at 550°C: (a) blurred martensitic laths and cementite; (b) continuous distribution of carbides along lath boundary.

When the tempering temperature increased up to 650 °C, extreme large polygonal carbides were

observed as shown by red circles in Fig. 7(a). Particularly, in comparison with 550 °C, many polygonal grains with sizes of 100 - 200 nm were observed in Fig. 7(b), which indicated the occurrence of recrystallization.

Figure 7. TEM micrographs after tempering at 650 °C : (a) polygonal carbides indicated by circles; (b) recrystallized grains.

3.4 Mechanical properties

Fig. 8 shows the evolution of mechanical properties with tempering temperatures. As illustrated in Fig. 8 (a), when the steel was tempered at low-temperatures (between 180 and 250 °C), the yield strength, tensile strength and hardness changed slightly. It is worth pointing out that, with an increase in tempering temperature from 180 to 250 °C, the tensile strength firstly increased and then decreased. Its peak value reached 1600 MPa after tempering at 200 °C, which was only 50 MPa higher than that after tempering at 230 °C. When the tempering temperature was increased to mid-temperatures (between 320 and 480 °C), both tensile strength and yield strength rapidly declined. However, the tensile strength still kept at a high level of ~ 1100 MPa. When tempering temperature continuously increased from 550 to 650 °C, the strength decreased slower compared with that after tempering at mid-temperatures. Noticeably, after tempering at 650 °C, the tensile strength was only 850 MPa, which was significantly lower than that after tempering at 200 °C (1600 MPa). On the other hand, Fig. 8(b) indicates that the reduction fluctuated around 58 % while total elongation tended to increase slightly with an increase in tempering temperature. The total elongation reached 28.4 % when the tempering temperature was 650 °C, which was 13% higher than that of low-temperature tempering.

Figure 8. Effect of tempering temperature on (a) yield strength (R_p), tensile strength (R_m) and hardness (HRC), and (b) total elongation (A), reduction (Z) and impact toughness (A_{kv}).

In addition, Fig. 8 (b) shows impact toughness dramatically varying with tempering temperature. During tempering at low-temperatures (between 180 and 250 °C), the impact toughness changed slightly. Its peak value was 91.5 J after tempering at 230 °C, which was 10.2 J higher than that after tempering at 250 °C. With an increase in tempering temperatures up to 320 °C, the impact toughness declined sharply, which was only 15 J. When tempering temperature increased from 320 to 550 °C, the impact toughness continuously decreased and reached a minimum value of 5 J. However, when the tempering temperature increased up to 650 °C, the impact toughness rapidly increased up to 75 J.

Fig. 9 shows the variation in fractography with an increase in tempering temperature. After tempering at 230 °C, deep and equiaxed dimples were observed, implying ductile fracture (Fig. 9 (a)). After tempering at 320 °C, intergranular crack (indicated by red arrow) and transgranular cleavage (indicated by white arrow) were dominantly observed with few shallow dimples (Fig. 9(b)). It means a transition from ductile fracture to brittle fracture. With an increase in tempering temperature to 550°C, intergranular fracture was observed (Fig. 9(c)), indicating brittle fracture. After tempering at 650 °C, dimples were observed again (Fig. 9(d)).

In a word, the 3Mn-Si-Ni steel has excellent combination of strength and toughness after tempering at 230 °C. Its tensile strength reached 1550 MPa, total elongation was 14.6 % and V-type notch impact energy was 91.5 J. In addition, tempering brittleness was observed after tempering at mid-temperatures (between 320 and 550 °C). Although tempering at a high temperate of 650 °C resulted in a good impact energy (75 J), the strength was significantly reduced (850 MPa).

Figure 9. Fractography after tempering at (a)230 °C,(b)320 °C,(c) 550 °C and (d) 650 °C.

4 Discussion

4.1 Microstructure evolution with tempering temperature

After oil quenching from 900 °C (before tempering), the microstructure consisted of martensite with typical hierarchal structure (Fig. 2). After tempering from 180 to 650 °C, carbide precipitation, recovery and decomposition of RA were observed (Figs. 4 - 7). Fig. 10 shows the evolution of carbides with temperature calculated using Thermo-Calc. M_7C_3 , cementite and MX (M represents Fe/Mn/Ni and X represents N/C) would completely dissolve at about 520, 650 and 1280 °C, respectively. It means MX would still exist after quenching from austenitization temperature of 900°C. M_7C_3 is present when the temperature was higher than 180 °C, which corresponds to the observation of ϵ -carbide after tempering at 230 °C (Fig. 4(b)). Cementite starts to numerously precipitate from 500 °C. Correspondingly, some small cementite was observed firstly after tempering at 320 °C (Fig. 5(b)) and then a lot of coarse cementite was observed after tempering at 550 °C (Fig. 6(b)). The precipitation of cementite made SEM microstructure sharp again (Fig. 3(c)). In a word, the thermo-calculation generally coincides with TEM observation. Similar evolution of carbide precipitation from transition carbide to cementite was observed in Refs. [3, 9]. In addition, with an increase in tempering temperature from 230 to 320 °C, the size of carbides increased

from ~ 4 to ~ 9 nm.

Figure 10. The relation between equilibrium carbides and temperature for 3Mn-Si-Ni steel. M and X stand for Fe/Mn/Ni and N/C, respectively.

With an increase in tempering temperature, recovery became stronger and stronger. This recovery phenomenon not only involved in the reduction of dislocation densities but also in the elimination of low angle martensitic lath boundaries ^[29, 30]. For example, when tempering temperature increased from 230 to 320 °C, the dislocation densities in martensitic laths decreased (c.f. Figs. 4(a) and 5(a)). In addition, the dislocation lines tended to rearrange as cell structures instead of tangles when tempering temperature increased to 320 °C (Fig. 5(b)). Some boundaries between martensitic laths became ambiguous at 320 °C (Fig. 5(b) and eventually disappeared at 550 °C (Fig. 6(b)). As a result, the microstructure characterized using SEM became blurry (Fig. 3(b)). In addition, few sub-grains were observed after tempering at 550 °C (Fig. 6(b)). With increasing tempering temperature up to 650 °C, many sub-grains were detected (Fig. 7(b)), indicating partial recrystallization. It is the reason why the SEM microstructure was blurred again (Fig. 3(d)). This recrystallization after tempering at a relative high temperature was also reported in Refs. [31, 32].

It is noted that the partial decomposition of residual austenite was firstly detected after tempering at 320 °C (Fig. 5(b)). With an increase in tempering temperature up to 550 °C (Fig. 6(b)), most of residual austenite decomposed into cementite and ferrite. Balliett *et al.* also reported the same phenomenon ^[33, 34].

4.2 The dependence of impact toughness on microstructure evolution

After tempering at 230 °C, the high strength of 1550 MPa and excellent toughness of 91.5 J were achieved, which were attributed to high dislocation density in martensitic laths and the absence of cementite. Non-uniform distribution of dislocations could facilitate their movements ^[35, 36]. Furthermore, local stress concentration could be relaxed by dislocation sliding, resulting in the delay of crack nucleation and the relaxation of stress on the crack tip ^[37, 38]. In addition, fine martensitic laths with fine ϵ -carbide precipitations (Fig. 4(b)) were beneficial to strengthening and toughening ^[39 - 41].

TME was observed with an increase in tempering temperature from 320 to 600 °C (Fig. 8(b)), as the same phenomenon reported in many martensitic steels [10, 12, 13]. This temperature, ranged for TME, corresponded to cementite formation and decomposition of residual austenite as discussed in *section 4.1*. Fig.11 shows impact toughness changing with the size of carbides. The size after tempering at 320 °C was in the same magnitude of that after tempering at 230 °C, but the impact toughness was sharply reduced by 67 J. Therefore, the size of carbides shouldn't be responsible for this sharp reduction. The main reason was ascribed to cementite formation instead of ϵ -carbide (c.f. Figs. 4(b) and 5(b)). In addition, the aggregated carbides, as a result of the decomposition of residual austenite (Fig. 5(b)), distributed along the boundaries of martensitic laths, contributing to brittle fracture as shown in Fig. 9(b) [1, 42]. The size of carbides after tempering at 550 °C increased approximately by 27 nm in comparison with that after tempering at 320 °C. Many of these carbides distributed along martensitic lath boundaries (Fig. 6 (b)), resulting in a further reduction of impact toughness.

However, with an increase in tempering temperature up to 600 and 650 °C, impact toughness rapidly increased, even though the size of carbides significantly increased. It was ascribed to strong recovery and partial recrystallization (detected at 550 and 650 °C as shown in Figs. 6(b) and 7(b), respectively), leading to a significant decrease in dislocation densities. Therefore, tempering after 650 °C had low tensile strength of 850 MPa, although high impact energy of 75 J was obtained.

Figure 11. The evolution of impact toughness with the size of carbides after tempering at different temperatures.

5 Conclusions

A designed low carbon 3Mn-Si-Ni steel was austenitized at 900 °C for 40 min and then tempered at different temperatures from 180 to 650 °C for 120 min. Microstructures and mechanical properties were characterized using SEM, TEM, tensile test and V-type notch impact test. The conclusions can be made as follows:

- (1) After tempering at 230 °C, the excellent combination of tensile strength (1550 MPa) and toughness (91.5 J) was achieved. It was ascribed to high dislocation density and short rod-like ϵ -carbides.
- (2) Tempering martensitic embrittlement was observed after tempering between 320 and 550 °C (impact toughness of ~ 10 J), resulted from cementite precipitation instead of ϵ -carbides and the decomposition of

residual austenite into cementite and ferrite.

(3) Although the good impact energy of ~ 75 J was obtained after tempering at high temperature of 650 °C, the tensile strength was significantly reduced to only 850 MPa, which was resulted from strong recovery and partial recrystallization.

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