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Influence of Coiling Temperature on Microstructure, Precipitation Behaviors and Mechanical Properties of a Low Carbon Ti Micro-Alloyed Steel

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Abstract: The microstructural evolution, nanosized precipitation behaviors and mechanical properties of a Ti-bearing micro-alloyed steel at different coiling temperatures were studied using optical microstructure (OM), scanning electron micrograph (SEM), transmission electron microscopy (TEM), Vickers hardness and tensile tests. When the coiling temperature was 500 °C, the specimen showed mainly bainitic structure, whereas polygonal ferrite was visible as the coiling temperature increased to 650 °C and 700 °C. The Vickers hardness of tested steel reached the maximum, which can be attributed to the largest number of nanosized precipitates in ferrite at the coiling temperature of 650 °C. A coiling temperature of 650 °C was optimal for the formation of TiC because of the high diffusion rate of alloying elements and kinetics of precipitation. In the laboratory rolling experiment, when the coiling temperature was 630 °C, the steel with yield strength of 682 ± 2.1 MPa and tensile strength of 742 ± 4.9 MPa was produced. The fine-grain strengthening and precipitation strengthening were 262 MPa and 268 MPa, respectively.

Keywords: coiling temperature; microhardness; nanosized precipitation; precipitation strengthening

1. Introduction

Nanosized precipitation of titanium (Ti), niobium (Nb), molybdenum (Mo) and vanadium (V) carbonitrides has been known to be effective for strengthening high-strength low-alloy (HSLA) steels, which are widely used in industrial equipment, bridge, pipeline and automobile. High strength for micro-alloyed steel is one of the researching hotspots in recent years [1–5]. Ti, Nb, V and Mo elements with individually or combined added have been employed to refine microstructure and enhance precipitation strengthening, which could significantly improve the strength of steel. Previous studies showed that the precipitation could provide more than 300-MPa strength increment based on Orowan mechanism when the particle size was less than 10 nm [6–11].

With the development of micro-alloyed steels, cost reduction becomes a challenge in industrial production due to the high price of micro alloys. Hence, the addition of Ti has attracted increasing attention as a main micro-alloying element to produce high-strength steel because of its abundant resources and the low price. Some studies have attempted to investigate precipitation behaviors of Ti-bearing steels [10–15]. Funakawa and Shiozaki [1] developed a Ti–Mo-based hot-rolled steel with a yield strength over 700 MPa which was coiled at 620 °C by use of nanosized (Ti, Mo)C particles in ferrite. Kong [10] and Wang [11] studied the influence of hot-rolling process parameters on the precipitation behaviors of nanosized (Ti, Mo)C particles and found that uniform distributed particles with nanometer size could be obtained in ferrite based on rolling in recrystallization region.

Xu [12] reported that the mechanism between dislocation and precipitates for random precipitation and interphase precipitation of a Nb–Ti micro-alloyed steel which was formed at different coiling temperature, was different. Natarajan [15] studied the effect of microstructural evolution, precipitation behaviors and dislocation structure at two coiling temperature in a Nb–Ti high-strength steel. The results showed that there was insufficient time for the dissolved microalloying elements to precipitate during cooling process at high cooling rate, so precipitates mainly formed during coiling stages. Furthermore, in most of the reported literature, the volume fraction of precipitates was artificially determined by transmission electron micrograph (TEM) and the precipitation strengthening was calculated according to theoretical model [16,17]. Therefore, detailed investigation on precipitation behaviors at different coiling temperatures should be conducted in Ti micro-alloyed steels.

In this study, the effect of coiling temperature on the microstructure and precipitation behavior of a Ti-bearing micro-alloyed steel was investigated. The optimum precipitation temperature was confirmed by Vickers hardness and TEM. Meanwhile, strengthening mechanism was discussed at hot-rolled condition. The findings from the present study are expected to provide some technical supports for the development of high-strength hot-rolled steels.

2. Experimental Procedure

The chemical composition of the tested steel was C 0.05, Si 0.39, Mn 1.5, Ti 0.2, P 0.005, S 0.002, N 0.005 and Fe balance (mass%). The steel was prepared by vacuum melting and forged into billets with a size of 80 mm × 80 mm × 120 mm, which were then used for hot-rolling experiments. The billets were rolled into 12 mm in thickness by use of a Φ 450 mm mill of the state key laboratory of rolling and automation (Shenyang, China). The plates were soaked at 1200 °C for 24 h followed by water quenching in order to homogenize the micro-alloying elements. Specimens for thermal simulation experiments were cut from the plates along the rolling direction and then machined into a dimension of Φ 8 mm × 15 mm.

Thermal simulation experiments were conducted on a Gleeble 3800 thermal simulation machine (DSI, Poestenkill, NY, USA). As shown in Figure 1, the specimens were heated to 1200 °C with a heating rate of 10 °C/s and held for 300 s to homogenize the micro-alloyed elements and dissolve carbonitrides. The specimens were then cooled with a cooling rate of 10 °C/s to 1050 and 850 °C to be deformed by 0.3 with a strain rate of 5/s. After deformation, specimens were cooled to 700, 650, 600, 550 and 500 °C with a cooling rate of 10 °C/s and then cooled to room temperature at a cooling rate of 0.1 °C/s to simulate the coiling process.

Figure 1. Schedule of thermal simulation experiments.

Hot-rolling experiments were carried out on a Φ 450 mm mill with a variety of cooling devices. The billets for hot rolling were isothermally treated at 1200 °C for two hours in a K010 box-shaped furnace, then rolled to 4 mm through nine-pass rolling. Rolling process was carried out in two stages (roughing rolling and finish rolling). The corresponding rolling reduction was 80% and

75%, respectively. The reduction schedule was: 80–53–36–24–16–12–9–7–5–4 (mm). The billet was rough-rolled through four-pass rolling with average reduction of 33% and deformation temperature in the range 1100–1050 °C. The intermediate billet was rolled to the desired thin strips through five-pass rolling with average reduction of 24% and finish rolling temperature in the range 870–850 °C. Finally, the product was laminar cooled with a cooling rate of 20 °C/s to 630 °C and then put into the asbestos (which was used in fire protection and insulation, because of its fiber strength and heat resistance) to cool to room temperature.

Samples for optical metallography of the thermal simulation experiments were cut by wire cutting along the radial direction, while samples for hot-rolling experiments were cut along the perpendicular to the rolling direction. Microstructures of the thermal simulation experiments were observed by use of a LEICAQ550 metalloscope (LEICA Microsystems, Wetzlar, Germany) after etched with 4% nital. A QUANTA 600 SEM (FEI, Hillsboro, United States) was used to observe the microstructure of the hot-rolled specimen, which was polished and etched using 4% nital solution. Vickers hardness of different processes was detected with a load of 50 g and a load time of 10 s. Ten measurements were made on each sample.

Specimens for tensile experiments were cut from hot-rolled slabs along the rolling direction and then machined to the dimension according to the GB/T2975 standard, as shown in Figure 2. Tensile strength, yield strength and elongation were obtained on a CMT-5105 tensile testing machine (MTS, Shenzhen, China) at ambient temperature, and the tensile rate was 5 mm/min. Three tensile experiments were conducted per condition.

Figure 2. Dimension of specimens for tensile experiments.

Carbon replica and thin foil for TEM observation were prepared to analyze the microstructure of precipitates. Specimens for carbon replicas were cut from the thermal simulation samples along the radial direction. Qualitative analysis of precipitates was performed using an energy-dispersive spectrometer (EDS). The specimen of the hot-rolled steel for TEM was cut into a thickness of 0.5 mm along perpendicular to the rolling direction, mechanically thinned to 0.05 mm by abrasion SiC papers and then twin-jet electro polished to perforation using a mixture of 20% perchloric acid and 80% ethanol at −20 °C, using a potential of 35 V. The characterization of precipitates was carried out on a TecnaiG2 F20 field-emission-gun TEM (FEI, Hillsboro, OR, USA).

3. Results and Discussion

3.1. Optical Metallography

The optical microstructures of the tested steel at different coiling temperatures during thermal simulation experiment are shown in Figure 3. The microstructure was composed of granular bainite (GB) and acicular ferrite (AF) when coiled at 500, 550 and 600 °C. The microstructure was mainly polygonal ferrite (PF) when the coiling temperatures were 650 and 700 °C. Bainite and ferrite become finer with the decreasing of coiling temperature. Using a linear interception method, the average ferrite grain sizes at 650 and 700 °C were measured to be $12.2 \pm 2.2 \mu\text{m}$ and $18.4 \pm 3.7 \mu\text{m}$, respectively. It is important to note that coiling temperature was a significant factor to the microstructural evolution. As the coiling temperature decreased, diffusibility of carbon was reduced, resulting in the inhibition of

ferrite formation. Further, low coiling temperature which caused larger undercooling, contributes to ferrite grain refinement.

Figure 3. Optical microstructures of tested steel at different coiling temperatures. (a) 500 °C; (b) 550 °C; (c) 600 °C; (d) 650 °C; (e) 700 °C.

3.2. Vickers Hardness

Figure 4 shows the variation of Vickers hardness at different coiling temperatures. The Vickers hardness increased with the increasing of coiling temperature from 500 °C to 650 °C and rose from HV 250 ± 5 to HV 304 ± 11 . As the coiling temperature increased from 650 °C to 700 °C, Vickers hardness decreased. In generally, Globular bainite should have a higher hardness than polygonal ferrite. Combining with the microstructural evolution, the higher hardness cannot be obtained when the microstructure was granular bainite at low coiling temperature. The significant difference in Vickers hardness could be mainly attributed to the precipitation strengthening effect due to the addition of Ti. Precipitation behaviors will be discussed to reveal the hardness difference at different coiling temperatures in the following sections.

Figure 4. Vickers hardness of the tested steel at different coiling temperatures.

3.3. Precipitation Behavior

In the studied steel, Ti was added for grain refinement and precipitation strengthening. The characterization of precipitates at different coiling temperatures and EDS results is shown in Figure 5. When the coiling temperature was 500 °C, particles with the size larger than 100 nm were observed according to the replica technique. With the increasing of coiling temperature, besides the particles with the size larger than 100 nm, increasing particles, which did not exceed 50 nm, could be observed in ferrite. Two representative particles with the size of 200 and 30 nm were selected for energy spectrum analysis as shown in Figure 5f. The cubic precipitate in Figure 5a and the spherical precipitates in Figure 5d were confirmed by EDS analysis to be Ti(C, N) and TiC, respectively. Nitrogen preferentially precipitates in the form of large TiN particles, which are formed at high temperature. Ti and C could accumulate by attaching on TiN, thus the formation of Ti(N, C) particles occurs during deformation process. In addition to this, it is evident from Figure 5 that the tested steel contains a large number of spherical precipitates in the form of TiC. The solid solubility of TiN and TiC precipitation could be expressed in Equation (1) through (3) [4]:

$$\lg\{[Ti][N]\}_{\gamma} = 3.94 - 15190/T \quad (1)$$

$$\lg\{[Ti][C]\}_{\gamma} = 5.33 - 10475/T \quad (2)$$

$$\lg\{[Ti][C]\}_{\alpha} = 4.40 - 9575/T \quad (3)$$

where $[Ti]$, $[N]$ and $[C]$ are the solid solution amount of element Ti, N and C in austenite and ferrite, T is the solid solution temperature, γ and α represent austenite and ferrite, respectively. When the samples were soaked at temperature of 1200 °C, the solid solubility of TiN and TiC were calculated as 4.24×10^{-7} and 1.65×10^{-2} . It is indicated that N almost fully precipitated as TiN at high temperature. Based on precious studies [17], TiN particles could be neglected for the strengthening effect because of their large size. The remaining Ti of 0.183 wt% at 1200 °C mainly precipitated in the form of TiC precipitation during the subsequent deformation, cooling and coiling process, which had strong effect on strength.

Figure 5. TEM morphologies of precipitates at different coiling temperatures. (a) 500 °C; (b) 550 °C; (c) 600 °C; (d) 650 °C; (e) 700 °C; (f) EDS patterns of different precipitated particles indicated as the red label “A” in (a) and “B” in (d).

Figure 6 presents the size distribution of precipitation for carbon replica specimens at different coiling temperatures. More than eight images were used to determine the size of precipitation using Image-Pro Plus software. It is obvious that when the coiling temperature was 500 °C, the main precipitates were large ones with the size over 100 nm. With the increasing of coiling temperature, the solute Ti became reactive for nucleation, and the volume fraction of small particles increased. When coiled at 650 °C, it is obvious that the TEM morphology is dominated by precipitates with the size less than 50 nm. As the coiling temperature increased to 700 °C, the volume fraction of large precipitates increased, which can be attributed to the coarsening of TiC precipitates.

Figure 6. Frequency distribution of precipitates at different coiling temperatures. (a) 500 °C; (b) 550 °C; (c) 600 °C; (d) 650 °C; (e) 700 °C.

According to the classical nucleation theory, nucleation rate can be described in Equation (4) through (7) [4,18]:

$$J^* = N_V \beta^* Z \exp\left(-\frac{\Delta G^*}{kT}\right) \quad (4)$$

$$\beta^* = \frac{16\pi\sigma_{\text{TiC-Matrix}}^2 D_M}{a_{\text{TiC}}^4 \Delta G_V^2} \quad (5)$$

$$Z = \frac{V_a \Delta G_V^2}{8\pi (kT \sigma_{\text{TiC-Matrix}}^3)^{1/2}} \quad (6)$$

$$\Delta G^* = \frac{16\pi \sigma_{\text{TiC-Matrix}}^3}{3\Delta G_V^2} \quad (7)$$

where J^* denotes the nucleation rate of TiC, N_V is the number of potential nucleation sites per unit volume, β^* is the frequency factor, Z is the Zeldovich non-equilibrium factor, ΔG^* is the activation energy of nucleation, k is the Boltzmann coefficient, T is the temperature, $\sigma_{\text{TiC-Matrix}}$ is the interfacial energy between TiC and the matrix, D_M is the volume diffusion coefficient of component Ti in the matrix, a_{TiC} is the lattice parameter of TiC, ΔG_V is the driving force of nucleation and V_a is the atomic volume of a substitutional atom of TiC.

In the present study, the heating temperature and deformation factors were all consistent in thermal simulation experiments. It is concluded that N_V , V_a , D_M and $\sigma_{\text{TiC-Matrix}}$ were all the same. The difference in the precipitation behavior could be attributed to the kinetics of precipitation at different coiling temperatures. The nucleation and coarsening of TiC were depended on the diffusion rate of Ti and C. As the coiling temperature decreased, atomic diffusion was limited, resulting in the restriction of formation and growth of TiC. Thus, a large number of Ti and C atoms were kept in solution at low coiling temperature, while they precipitated in the form of TiC particles at high coiling temperature. When the coiling temperature increased to 700 °C, the diffusion of Ti and C was promoted, which caused a larger size of TiC particles. On the comparing of the precipitation behavior at different coiling temperatures, it is clear that the largest volume fraction of finer precipitates could be obtained at coiling temperature of 650 °C for Ti-bearing steels.

Based on previous studies, nanosized precipitates could extremely improve the strength of matrix by impeding the movement of dislocations, which can be described by the Ashby–Orowan model according to the theory of Gladman as shown in Equation (8) [4]:

$$\Delta \sigma_{ppt} = 0.3728 \frac{Gb}{1-\nu} \times \frac{f^{1/2}}{d} \ln \left(\frac{1.2d}{2b} \right) \quad (8)$$

where $\Delta \sigma_{ppt}$ denotes the strength increment attributed to precipitation strengthening (MPa), G is the shear modulus and equal to 81,600 MPa, b is the Burgers vector of magnitude 0.246 nm, ν is the Poisson ratio, f is the volume fraction of precipitates, and d represents the diameter of precipitates. According to Equations (8), precipitation strengthening is proportional to the volume fraction and inversely proportional to the size of precipitates. As shown in Figures 5 and 6, with the increasing of coiling temperature, the high volume fraction of finer TiC precipitates was obtained, which could provide larger contribution to the strength. Thus, precipitation strengthening was the highest at the coiling temperature of 650 °C, while it decreased at lower and higher coiling temperature. Since the volume fraction of precipitates was not exactly inferred from the replica technique, the precipitation strengthening could not be precisely calculated by Equation (8). In order to quantitatively analysis the effect of TiC particles on strength increment, a further rolling experiment was conducted.

3.4. Rolling Experiment

The rolling process parameters and mechanical properties are shown in Table 1. The yield strength, tensile strength and elongation were 682 ± 2.1 MPa, 742 ± 4.9 MPa and $24.2 \pm 1.7\%$, respectively. The SEM and TEM of the tested steel are presented in Figure 7. The hot-rolled steel predominately consisted of polygonal ferrite and quasi-polygonal ferrite, which is consistent with the thermal simulation experiment. The average diameter of ferrite grain was 4.4 ± 0.8 μm measured by Image-Pro Plus software. It can be concluded that the ferrite grain in hot-rolling process is significant finer than that in thermal simulation experiment, which can be mainly attributed to larger deformation and

higher cooling rate. There existed many spherical and cell-like nanosized precipitates in ferrite matrix, which were identified to be TiC in the range of 5–30 nm.

Table 1. Rolling parameters and mechanical properties of the hot-rolled steel.

Heating Temperature °C	Cooling Rate °C/s	Coiling Temperature °C	Yield Strength MPa	Tensile Strength MPa	Elongation %
1200	20–30	630	682 ± 2.1	742 ± 4.9	24.2 ± 1.7

Figure 7. (a) SEM and (b) TEM of the hot-rolled steel.

The yield strength of Ti-bearing steel can be expressed by Equation (9) [19,20]:

$$\sigma_y = \Delta\sigma_0 + \Delta\sigma_{ss} + \Delta\sigma_{GB} + \sqrt{\Delta\sigma_{dis}^2 + \Delta\sigma_{ppt}^2} \quad (9)$$

where σ_y is the yield strength, $\Delta\sigma_0$ is the crystal lattice strengthening (in the present study, the value was assumed to be 54 MPa), $\Delta\sigma_{ss}$ is the solution strengthening, $\Delta\sigma_{GB}$ is the fine grain strengthening, $\Delta\sigma_{dis}$ is the dislocation strengthening. The individual contribution of each strengthening mechanism can be evaluated by Equation (10) through (12):

$$\Delta\sigma_{GB} = 17.4d^{-1/2} \quad (10)$$

$$\Delta\sigma_{ss} = 360[C] + 32[Mn] + 83[Si] + 700[P] \quad (11)$$

$$\Delta\sigma_{dis} = \alpha M G b \rho^{1/2} \quad (12)$$

where d is the average size of ferrite grain, $[M]$ ($M = C, Mn, Si, P$) is the mass fraction of element M in solute state, ρ is the dislocation density in per unit area (in present study, the value was assume to be 10^{13} m^{-2}), α is equal to 0.38, M is the Taylor factor and equals to 2.2 for ferrite grain. The grain refinement strengthening and precipitation strengthening values were calculated as 262 MPa and 268 MPa. Although, bainite and acicular ferrite were not obtained at high coiling temperature, the tested steel exhibited high yield strength owing to the grain refinement strengthening and precipitation strengthening because of the Ti addition.

4. Conclusions

(1) The samples were composed of granular bainite and acicular ferrite at coiling temperature of 500, 550 and 600 °C, while mainly polygonal ferrite was observed at 650 and 700 °C. Coiling temperature has a significant effect on the microstructure evolution and grain size for Ti-bearing steels.

(2) Coiling temperature had an important influence on Vickers hardness attributed to TiC particles. Vickers hardness was the highest at coiling temperature of 650 °C.

(3) The nucleation rate of TiC was depended on the diffusion rate of Ti and C, which was determined by coiling temperature. A large volume fraction of fine TiC particles could be formed in ferrite at the optimal coiling temperature of 650 °C.

(4) In the hot-rolling condition, yield strength and tensile strength were 682 ± 2.1 MPa and 742 ± 4.9 MPa, respectively. The intense precipitation strengthening effect increased the yield strength by 268 MPa attributed to the large number of nanosized TiC precipitates in ferrite.

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