Effect of austenitizing temperature on the microstructure and mechanical properties of Nb–Ti microalloyed steel

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Abstract

The effect of austenitizing temperature on the microstructure and mechanical properties of Nb–Ti microalloyed steel was investigated. Steels were subjected to different austenitizing treatments (temperatures ranging from 850 °C to 1250 °C for 5-120 min) and rolled after being austenitized at different temperatures (i.e. 1020 °C, 1070 °C and 1150 °C). The results showed that austenite grain coarsening temperature was around 1000 °C. The mean grain size of the rolled steels initially increased and then decreased, but the ferrite content decreased with increasing austenitizing temperature. The precipitates in the prior austenite and rolled steel were both complex Nb–Ti carbonitrides. As the austenitizing temperature increased from 1020 °C to 1150 °C, most precipitates were dissolved and re-precipitated as dispersive particles with mean size decreasing from 30 nm to 10 nm. Meanwhile, the 80 nm to 100 nm rectangular Ti-rich carbonitrides were not dissolved and varied during the subsequent cooling process.The Yield strength and ultimate tensile strength increased but the elongation and reduction in area (in percent) decreased with the elevated austenitizing temperature.

Keywords: Austenitizing Temperature, Precipitation, Nb–Ti Microalloyed Steel, Coarsening Temperature, Mechanical Properties
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1. Introduction

The addition of microalloying element including Nb, V and Ti play significant roles in improving the properties of steels [1], [2], [3]. Nb and Ti have a strong tendency to precipitate in the form of carbides or nitrides in austenite which can lower the grain growth rate by pinning the grain boundaries [4], [5]. The obtained fine grain microstructure can influence the mechanical properties of steels [6], [7].

Much research has been undertaken to investigate the reheating temperature, deformation temperature, amount of reduction, cooling rate and strain rate in the microstructural evolution (including precipitation and austenite recrystallization) and mechanical properties of Nb–Ti microalloyed steel[3], [4], [8], [9], [10]. Austenitizing temperature has significant influence on microstructure evolution and mechanical properties. When steels are austenized at different temperatures, the carbides and nitrides are dissolved, causing redistribution of carbon and consequently influencing phase transformation in the subsequent deformation process (e.g.the grain growth and transformation from austenite to pearlite, ferrite, bainite, and so on in the cooling process as well as precipitation of secondary carbides. All these microstructure evolution affect the mechanical properties of the steel products.

Several studies have been performed on the effect of austenitizing temperature on the structure and mechanical properties of steels containing Nb or Ti [11], [12], [13], [14], [15], especially on grain refinement [16], [17]. However, early efforts mainly focused on the effect of microalloying with Nb or Ti, grain growth behavior of coarse-grained

austenite or strips manufactured through the compact strip production (CSP) process, which has a low deformation ratio. The effect of austenitizing temperature on microstructure evolution including precipitation and phase transformation before and after rolling in steels microalloyed with both Nb and Ti has not been carefully studied. Thus, this study investigates the effects of austenitizing temperature on the microstructure evolution and mechanical properties of Nb–Ti microalloyed steel.

In the present work, steel samples were prepared by vacuum induction melting and subjected to different austenitizing temperatures. The coarsening temperature range was determined based on prior austenite grain size changes by metallographic analysis. The steel samples were rolled to investigate the influence of austenitizing temperature on the microstructure and properties of Nb–Ti microalloyed steel.

2. 2. Experimental procedures

The steel employed in this study was melted in a 200 kg vacuum induction furnace. The chemical composition is shown in Table 1. Samples of 10×10×12 mm were cut from the steel for austenitizing treatment. These samples were austenitized at 850 °C, 900 °C, 950 °C, 1000 °C, 1050 °C, 1100 °C, 1150 °C, 1200 °C and 1250 °C for 5, 10, 15, 30, 60 and 120 min in a muffle furnace. The temperature accuracy was controlled within ±2 °C. The samples were then cooled in cold water.

Samples for metallographic observations were prepared through conventional grinding and polishing and etched with nitric acid alcohol solution. Three rolling schedules with different austenitizing temperatures were created after metallographic observation, as illustrated in Figure 1. The vacuum induction melted specimens were forged into 80 mm × 80 mm × 400 mm ingots, reheated to 1020 °C, 1070 °C and 1150 °C, maintained for 30 min and then hot rolled

with a reduction of 50 % at the temperature between 980 °C and 1000 °C. The specimens were then rolled at a temperature between 920 °C and 880 °C in six rolling phases

to the obtain final thickness of 12.5 mm. The plates were air-cooled to room temperature; they are hereafter referred to as 1#, 2# and 3#.

Table 1. Chemical composition of the experimental steel (wt %)

C	Si	Mn	P	S	Ni	Cr	Cu	Mo	Nb+Ti	N
0.08	0.20	1.06	0.017	<0.01	0.28	0.50	0.29	0.18	0.045	0.004

To study the precipitates, extraction replicas were prepared in a four-step procedure by etching the polished surface in 3 % nital, coating the surface with a thin film of C, stripping the film in 5 % nital and cleaning in both distilled water and methanol. The precipitates were analysed through transmission electron microscopy (TEM). Chemical analysis of the precipitates was conducted through nanobeam energy dispersive spectrum (EDS) analysis. A tensile impact test was conducted with a universal materials tester at room temperature.
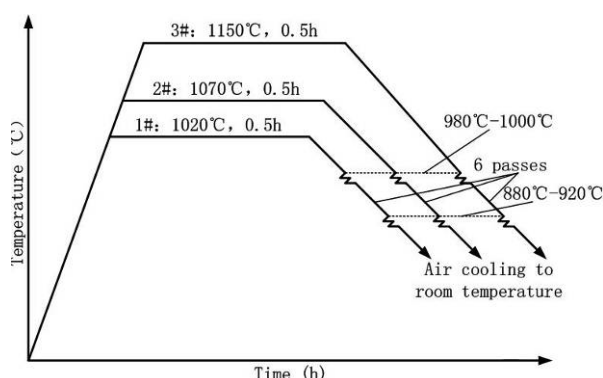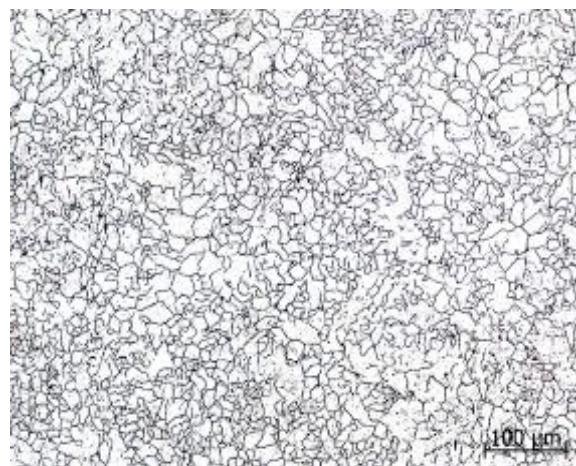

Fig.1. Schematic of the rolling process

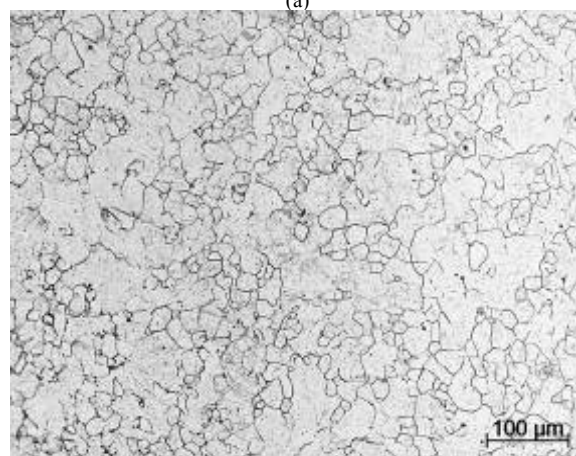## 3. Results

3.1 Austenite Grain Size Analysis
3.1.1 Effect of Austenitizing Temperature on Prior Austenite Grain Size

Figure 2 shows the metallographic microstructures of prior austenite subjected to different austenitizing temperatures. The austenite grain sizes increased with increased austenitizing temperature. When the temperature increased to 1000 °C, the grain size began to coarsen. Meanwhile, the grain size was not uniform. The mean grain size was approximately 30 μm; however, the maximum grain size was almost 100 μm, and the minimum grain size was only 10 μm. When temperature increased to 1100 °C, the prior austenite coarsened with a mean grain size of 150 μm.
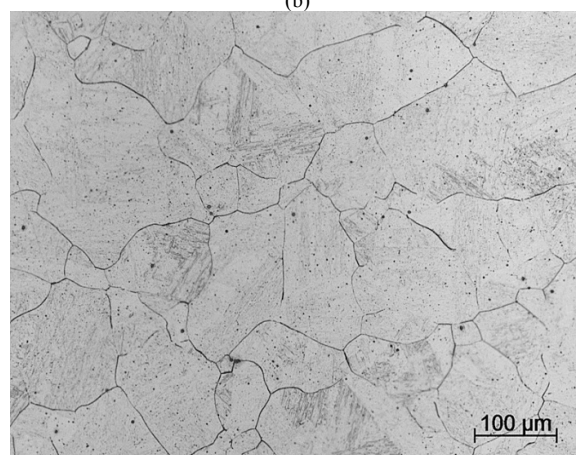
Figure 3 shows the austenite grain sizes of the experimental steels subjected to different austenitizing temperatures for different amounts of time. The mean prior austenite grain sizes were 25μm to 30 μm when temperature increased from 850°C to 1000 °C within 5 min to 120 min. The grain sizes coarsened from 30 μm to 180 μm atthe temperature range of 1000°C to 1150 °C. The grain sizes increased slightly when the temperature exceeded 1150 °C for different time of 5 min to 120 min. This result illustrates that the coarsening temperature was approximately 1000 °C.

Fig. 2. Metallographic microstructures of steel samples subjected to different austenitizing temperatures for 30 min: (a) 850 °C, (b) 950 °C, (c) 1000 °C, (d) 1100 °C
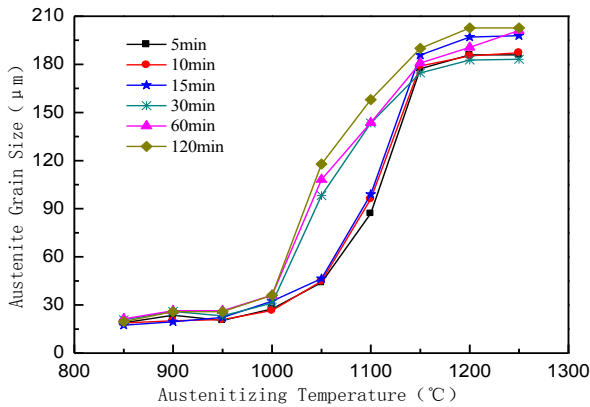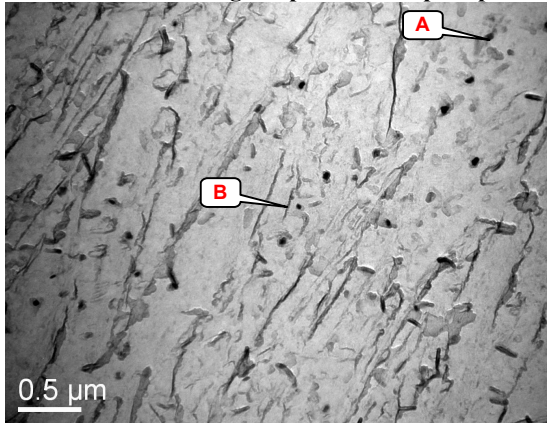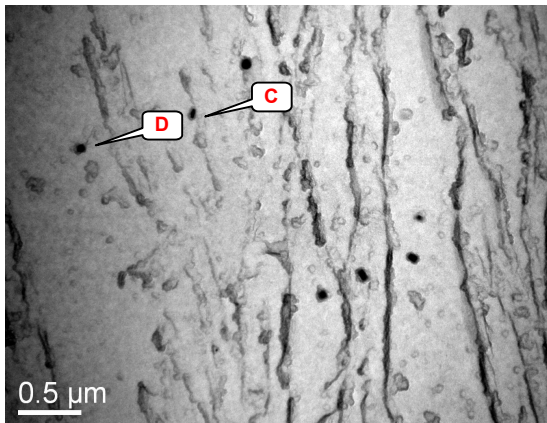
Fig. 3. Austenite grain size of the experimental steel at different austenitizing temperatures
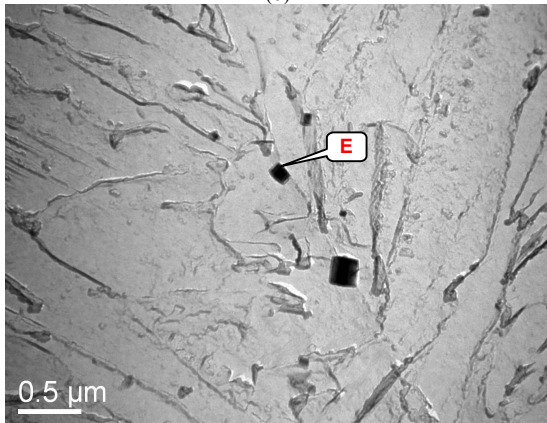
3.2 Effect of austenitizing temperature on precipitates

(a)

(b)

(c)

Fig. 4. TEM images of steel precipitates after holding for 30 min under different temperatures: (a) 950°C, (b) 1050°C, and (c) 1150 °C

Table 2. EDS analysis results of the precipitates (°C)

	A	B	C	D	E
Ti (wt%)	6.00	17.00	16.30	84.96	83.91
Fe(wt%)	80.63	8.73	15.78	14.10	5.24
Nb(wt%)	13.37	74.27	67.92	0.94	10.85

Figures 4a to 4c and Table 2 show the morphologies and chemical compositions of the precipitates under different austenitizing temperatures. The precipitates in the specimens increased with the elevated temperatures. However the volume fractions decreased. The mean size of the precipitates increased from approximately 30 nm to 100 nm when the austenitizing temperature increased from 950 °C to 1150 °C.

The precipitates can be divided into two categories. Those with regular shapes (e.g. rectangular) were Ti–rich carbonitrides. Those with irregular or spherical shapes were characterized by the main composition of Nb and minimal Ti. At 950 °C, the two types of precipitates exhibited a dispersive distribution, with a size between 20 nm to 50 nm. As temperature increased to 1050 °C, the sizes increased further because the driving force increases. Meanwhile, the volume fraction of precipitates decreased because of the dissolution of carbonitrides. At 1150 °C, the remaining precipitates which with regular shapes were Ti-rich carbonitrides.

3.3 Effect of austenitizing temperature on the microstructure of rolled steel

Figure 5 shows the ferrite grain sizes of the three steel samples. The grain size of 1# steel (12.89 µm) was the smallest and that of 2# steel was the largest (16.38 µm). For 1# steel, the austenitizing temperature was low before rolling; hence, the austenite grain lacked sufficient driving force of growth, the ferrite grain formed in the subsequent cooling stage was small. For 2# steel, the prior austenite grain began to grow up before rolling (Figure 3). The partial dissolved carbonitrides resulted in a reduction in the volume fraction of the carbonitrides in the subsequent rolling process. Thus, the effect of inhibition was insufficient. For 3# steel, the grain size decreased. It was austenitized at a high temperature of 1150 °C when many carbonitrides were dissolved. The re-precipitation of the dissolved carbonitrides during the subsequent cooling stage hindered the growth and re-crystallization of the ferrite grain. Thus, the grain size of 3 # steel was smaller than that of 2# steel. The grain size changes of the rolled steel samples are illustrated in detail in Section 4.2.

Figure 6 shows the contents of ferrites in the rolled steels. The fractions of ferrites in the three rolled steel samples initially decreased from 83.6 % to 82.1 % and then further declined to 79.1 % as the austenitizing temperature increased from 1020 °C to 1150 °C. As illustrated in Figure 4, the fine carbonitrides were dispersed in the prior austenites, which provided nucleation sites for the formation of ferrite. As the austenitizing temperature increased the volume fraction of precipitates decreased, resulting in a decrease in ferrite contents.
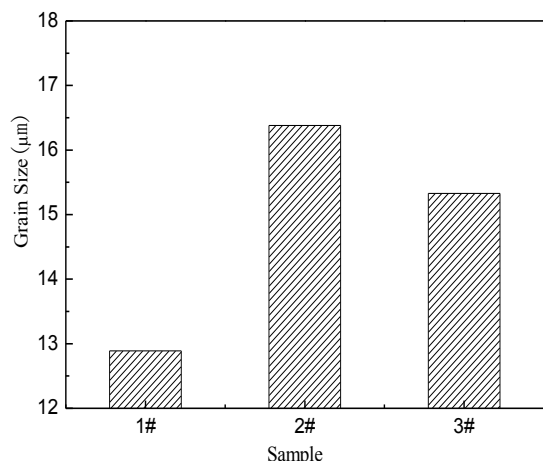
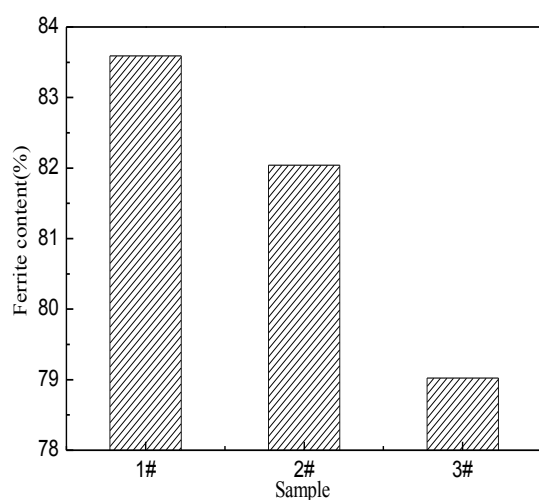**Fig. 5.** Grain sizes of the three rolled steel plates

Fig. 6. Contents of ferrite in the three rolled steel plates

3.4 Precipitates in rolled steel

Figure 7 and Table 3 shows the morphologies and chemical compositions of the secondary carbonitrides in the rolled steel samples. The 20 nm to 30 nm precipitates in 1# steel were dispersed in the steel matrix. By contrast, the sizes of most precipitates in 2# steel were smaller (approximately 20 nm). The elevated austenitizing temperature resulted in the abnormal growth of carbonitrides. Similarly, most precipitates in 3# steel were smaller, with a size of approximately 10 nm. The large rectangular precipitates with a size of 80 nm to 100 nm in 2# and 3# steel were primary Ti-rich carbonitrides that were not dissolved during the austenitizing treatment and varied slightly in the subsequent cooling stage. EDS revealed that the fine dispersive precipitates are complexs of Ni and Ti and the large precipitates mainly composed of Ti. These fine carbonitrides with 10 nm size in 3# steel were dispersed in the steel matrix and hindered the grain growth of ferrite. This is the reason the mean grain size of 3# rolled steel was smaller than that of 2# steel.
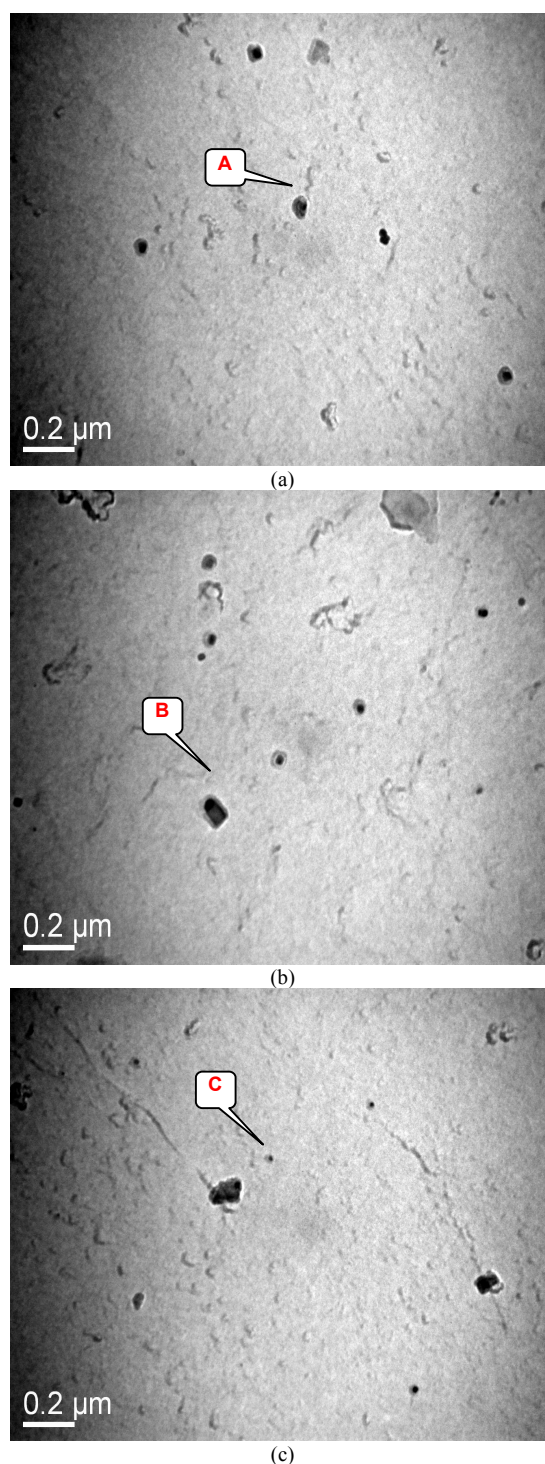

Fig. 7. TEM images of the precipitates of the three experimental steel plates：（a）1#,（b）2#, and（c）3#

Table 3. EDS analysis results of the precipitates (°C)

	A	B	C
Ti (wt%)	27.00	81.25	16.37
Fe(wt%)	18.26	14.10	14.97
Nb(wt%)	54.74	4.65	68.66

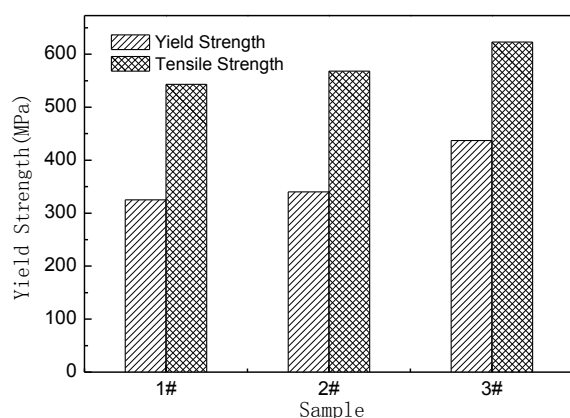## 3.5 Effect of austenitizing temperature on mechanical properties

Fig.8. Strength of steel samples

Figure 8 shows the yield strength (YS) and ultimate tensile strength (UTS) of the three steel samples; YS and UTS were 325 MPa to 437 MPa and 543 MPa to 623 MPa, respectively. YS and UTS increased with increased austenitizing temperature. Figure 9 shows the elongation and reduction (%) of the area of the rolled steel samples; elongation and reduction were 24.76 % to 30.68 % and 55.16 % to 60.83 % respectively. Both the reduction and the elongation percentages decreased as the austenitizing temperature increased. This result indicates that strengths increased but toughness decreased as austenitizing temperature increased.
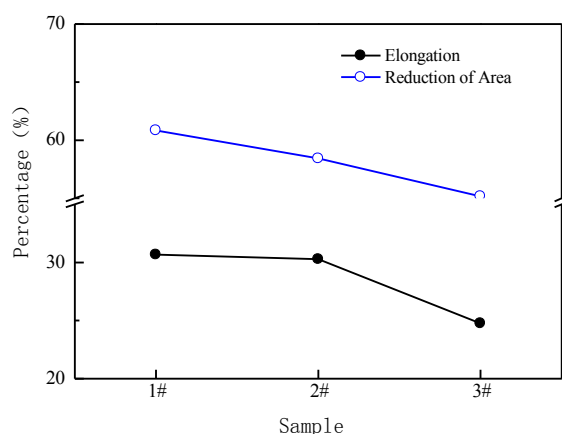

Fig. 9. Elongation and reduction of the area of the steel samples

4. Discussion

4.1 Thermodynamic precipitation of the carbides and nitrides of Nb and Ti

By assuming that the effects of other elements in steel and the distribution of elements between carbide and nitride on the solubility of NbC, NbN, TiC, Nb(C, N) and TiN can be ignored, the solution temperatures in the steels were calculated according to the solubility product proposed by the following formulas [18].

$$\lg\{[Nb]\cdot[C]\}_\gamma = 2.96 - 7510/T \qquad (1)$$

$$\lg\{[Nb]\cdot[N]\}_\gamma = 3.70 - 10800/T \qquad (2)$$

$$\lg\{[Nb]\cdot[C+12/14N]\}_\gamma = 2.26 - 6770/T \qquad (3)$$

$$\lg\{[Ti]\cdot[C]\}_\gamma = 2.75 - 7000/T \qquad (4)$$

$$\lg\{[Ti]\cdot[N]\}_\gamma = 0.32 - 8000/T \qquad (5)$$

where $[Nb]$, $[Ti]$, $[C]$ and $[N]$ denote the amounts of soluble Nb, Ti, C and N in austenite, respectively, T is the absolute temperature(K). The calculated results are provided in Table 4.

Table 4. Estimated solution temperature of precipitates in steel (°C)

Precipitates	NbC	NbN	Nb(C, N)	TiC	TiN
Solution temperature	1089	1157	1139	924	1422

Table 4 shows that TiC was completely dissolved at 924 °C; however, the dissolution temperature of TiN was as high as 1422 °C.The carbonitrides of Nb were dissolved between 1089°C and 1157 °C. Given that the content of Nb and Cwere higher than those of Ti and N, respectively, the dissolution of carbides plays a dominant role in the coarsening of prior austenite. This result explains why the prior austenite grain coarsening temperature was approximately 1000 °C (Figs. 2 to 3).

4.2 Grain growth in the austenization process

Grain coarsening behavior (Figure 2) was directly related to the nature of the precipitates present in each case. When the precipitates pinning the austenite grain boundaries began to dissolve, grain coarsening occurred. Coarsening occurred at a temperatures of approximately 1000 °C; when the dissolution of the Nb-rich carbonitrides particles was responsible for the pinning in the Nb–Ti steel (Figs. 2 to 3). When the Nb-rich carbonitrides partially dissolved at 950 °C to 1150 °C, the sizes of the precipitates initially increased slightly. A slight but obvious decrease in volume fractions ensued (Figure 4). The weakened pinning effect caused the mean prior austenite grain sizes to increase sharply at 1000 °C to 1150 °C (Figs. 2 to 3). The Nb-rich carbonitrides were almost dissolved completely at 1150 °C. However, above this temperature, a large amount of Ti was still precipitated as TiN particles which hindered grain growth and maintained it within a stable level (Figure 3).

The heterogeneity in grain size at approximately 1000 °C (Figure 2) was due to the heterogeneous micro-distribution and partial dissolution of carbonitrides in the steel. The multiple types of carbonitrides in the steel had different melting points. TiC dissolved completely at 924 °C (Table 2). The Nb–rich carbonitrides partially dissolved at 1000 °C (Table 2). Grain grew rapidly in these regions. However, the precipitated volume fraction of TiN was relatively stable, and TiN dissolved gradually with increased temperature; this condition hindered the grain growth within these regions. Thus, a heterogeneous microstructure was formed. When the austenitizing temperature increased to

1050 °C, the prior austenite grain size coarsened obviously with a mean grain size of 100 μm. The rapid growth of grains mainly resulted from the rapid dissolution of NbC and NbN, especially NbC (near dissolution temperature), with elevated temperature. When temperature exceeded 1150 °C, most of NbC and NbN were dissolved. The dissolution of TiN remained slow and resulted in the slight increment in prior austenite grain size (Figure 3).

4.3 Phase transformation in the cooling process

The microstructures of the rolled steels consisted of quasi-polygonal ferrite, granular bainite and a small amount of pearlite (Figure 5). Some of the carbonitrides were compounds. TiN with a high dissolution temperature was in the core of the compound ones and TiC, NbC and even NbN were in the outer layer [8], [19]. When a large amount of TiC, NbC and NbN was partially or completely dissolved, the nitrides core of Ti might not have been dissolved at the time. The semi-dissolved carbides or nitrides could have provided sites for pearlite nucleation. Moreover, dispersive fine carbonitrides in the prior austenites coupled with some microalloying elements (Ti, Nb) in the steel that might have transformed into oxides in the iron-making processes provided nucleation sites for the formation of ferrite in the steel during the rolling process. Thus, a microstructure of ferrite+pearite was formed.

As the austenitizing temperature increased from 1020 °C to 1150 °C, more Nb-rich carbonitrides were dissolved in the matrix. However, the steel was not homogenized fully within this time duration. In this case, the Nb-rich carbonitrides were in a state of partial dissolution. Thus, solute atom depleted and enriacheds were formed in the steel, resulting in a spinoda-like decomposition [20]. Bainite formed in this manner.

4.4 Mechanical properties

The YS and UTS of 2# steel were the result of the competition between grain refinement strengthening and precipitation strengthening, given that both the decrease in mean grain size and mean dispersive precipitate size generally leads to an increase in tensile strength. The mean grain size of 2# steel was larger than that of 1# steel (Figs.5 to 6); however, the mean precipitate size is smaller (Figure 8), and the competition between them resulted in a slight increase of YS and UTS in the2# steel relative to 1# steel. In the case of 3# steel, the joint action of grain refinement strengthening and precipitation strengthening resulted in an increase in YS and UTS because of the smaller mean grain size and mean dispersive precipitate size (Figs.5 to 6 and 8) compared with 2# steel. As the austenitizing temperature increased, the ferrite content decreased, resulting in a decrease in the area elongation and reduction percentages (Figs. 7 and 10).

5. Conclusions

The detailed micro-structural investigation and mechanical research on Nb–Ti microalloyed steel revealed the following points.

(1) The prior austenite grain size increased from approximately 25 μm to 180 μm as the austenitizing temperature increased from 850 °C to 1250 °C for 30 min. The coarsening temperature of the prior austenite was approximately 1000 °C within the time duration of 5 min to 120 min.

(2) The microstructures of the three rolled steel plates subjected to different austenitizing temperatures consisted mainly of quasi-polygonal ferrite, granular bainite and some pearlite. The grain size initially increased and then decreased, but the ferrite content decreased when the austenitizing temperature changed from 1020 °C, 1070 °C to 1150 °C.

(3) The precipitates in the prior austenite and rolled steel were both complex Nb–Ti carbonitrides, which can be mainly be divided into two types. The rectangular ones were Ti-rich carbonitrides. The others with an irregular or circular shaped one was Nb-rich carbonitrides.

(4) As the austenitizing temperature increased from 1020 °C to 1150 °C, the mean size of precipitates in prior austenite increased. However, the volume fraction decreased because of the dissolution of most precipitates. During the subsequent cooling process, most precipitates were re-precipitated as dispersive particles, with mean size decreasing from 30 nm to 10 nm. Meanwhile, the 80 nm to 100 nm rectangular Ti-rich carbonitrides were not dissolved and varied slightly.

(5) YS and UTS increased, but the area elongation and reduction percentages decreased with the elevated austenitizing temperature. The elevated austenitizing temperature was beneficial to the strength of the steel samples but negatively affected their the toughness.

References

1. Ghosh,P., Ray, R.K., Ghosh, C., and Bhattacharjee, D., "Comparative study of precipitation behavior and texture formation in continuously annealed Ti and Ti+Nb added interstitial-free high-strength steels", Scripta Materialia, 58 (11) , 2008, pp. 939-942.
2. Niakana, H., and Najafizadehb, A., "Effect of niobium and rolling parameters on the mechanical properties and microstructure of dual phase steels", *Materials Science & Engineering A*, 527 (21-22), 2010, pp. 5410-5414.
3. Ghosh, P., Ghosh, C., and Ray, R.K., "Thermodynamics of precipitation and textural development in batch-annealed interstitial-free high-strength steels", *Acta Materialia*, 58 (11), 2010, pp. 3842-3850.
4. Han, Y., Shi, J., Xu, L., Cao, W.Q., and Dong, H., "Effects of Ti addition and reheating quenching on grain refinement and mechanical properties in low carbon medium manganese martensitic steel", *Materials and Design*, 34 (8), 2012, pp. 427-434.
5. Matsuo, S., Ando, T., and Grant, N.J., "Grain refinement and stabilization in spray formed AISI 1020 steel", *Materials Science & Engineering A*, 288 (1), 2000, pp. 34-41.
6. Lazarova, R., Petrov, R.H., Gaydarova, V., Davidkov, A., Alexeev, A., Manchev, M., and Manolov, V., "Microstructure and mechanical properties of P265GH cast steel after modification with TiCN particles", *Materials and Design*, 32 (5), 2011, pp. 2734-2741.
7. Rasouli, D., Khameneh, A.S., Akbarzadeh, A., and Daneshi, G.H., "Optimization of mechanical properties of a microalloyed steel", *Materials and Design*, 30 (6), 2009, pp. 2167-2172.

8. Kostryzhev, A.G., Shahrani, A.A., Zhu, C., Ringer, S.P., and Pereloma, E.V., "Effect of deformation temperature on niobium clustering, precipitation and austenite recrystallisation in a Nb-Ti microalloyed steel", *Materials Science & Engineering A*, 581, 2013, pp. 16-25.

9. Fernández, A.I., Uranga, P., López, B., and Rodriguez-Ibabe, J.M., "Dynamic recrystallization behavior covering a wide austenite grain size range in Nb and Nb–Ti microalloyed steels", *Materials Science & Engineering A*, 361 (1), 2003, pp. 367-376.

10. Sha, Q.Y., Li, G.Y., Qiao, L.F., and Yan, P.Y., "Effect of cooling rate and coiling temperature on precipitate in ferrite of a Nb-V-Ti microalloyed strip steel", *Proceedings of Sino-Swedish Structural Materials Symposium 2007*, 14 (5), 2007, pp. 316-319.

11. Prasad, S. N., Mediratta, S.R., and Sarma, D.S., "Influence of austenitisation temperature on the structure and properties of weather resistant steels", *Materials Science & Engineering A*, 358 (1-2), 2003, pp. 288-297.

12. Babu, N.K., Suresh, M.R., Sinha, P.P., and Sarma, D.S., "Effect of austenitizing temperature and cooling rate on the structure and properties of a ultrahigh strength low alloy steel", *Journal of Materials Science*, 41 (1), 2006, pp. 2971-2980.

13. Liu, Y.J., Li, Y.M., and Huang, B.Y., "Influence of austenitizing temperature on apparent morphologies of as-quenched microstructures of steels", *Journal of Central South University of Technology*, 12 (2), 2006, pp. 122-129.

14. Zhu, Y.Z., Liang, D.M., Li, J.C., Xu, J.P., and Xue, Z.L., "Effects of thermo-mechanical treatment and microalloying with Cr, Nb and Ti on phase transformation in C-Mn steel strips produced by compact strip production process", *Materials Science & Engineering A*, 528 (29), 2011, pp. 8365-8373.

15. Sha, Q.Y., and Sun, Z.Q., "Grain growth behavior of coarse-grained austenite in a Nb-V-Ti microalloyed steel", *Materials Science & Engineering A*, 523 (1-2), 2009, pp. 77-84.

16. Han, Y., Shi, J., Xu, L., Cao, W.Q., Dong, H., "Effect of hot rolling temperature on grain size and precipitation hardening in a Ti-microalloyed low-carbon martensitic steel", *Materials Science & Engineering A*, 553(1), 2012, pp. 192-199.

17. Zrník, J., Kvackaj, T., Pongpaybul, A., Sricharoenchai, P., Vilk J., and Vrchovinsky, V., "Effect of thermomechanical processing on the microstructure and mechanical properties of Nb–Ti microalloyed steel", *Materials Science & Engineering A*, 319-321, 2001, pp. 321-325.

18. Yong, Q.L., "*Second Phases in Steels*", Metallurgical industry press, P.R.China, Beijing, 2006.

19. Craven, A. J., He, K., Garvie, L. A. J., and Baker, T. N., "Complex heterogeneous precipitation in Titanium–Niobium microalloyed Al-killed HSLA Steels-I.(Ti,Nb)(C,N) particles", *Acta Materialia*, 48 (15), 2000, pp. 3869-3878.

20. Kang, M.K., and Zhu, M., "Mechanism of bainitic nucleation and ledge growth", *Transactions of Materials and Heat Treatment*, 26 (2), 2005, pp. 1-5.