Effect of the Simulated and Pilot-scaled Thermomechanical Processes on the Microstructure, Precipitates and Mechanical Properties of V–N Alloyed Steel

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(Received on April 4, 2018; accepted on May 31, 2018)

The effect of the two-stage continuous cooling process with different finish cooling temperatures (FCTs) after 950°C high temperature finish deformation on microstructural evolution, precipitation and mechanical properties of V–N alloyed steel has been investigated under the simulated and pilot-scaled thermomechanical condition. Microstructural observation of dilatometry specimens indicates that FCT should be above 600°C to obtain microstructure composed of polygonal ferrite (PF), pearlite (P), and less than 5 vol. pct bainite (B) in steel. Based on the simulation result, three pilot-scaled cooling paths with FCTs of 950, 750 and 600°C are designed and all microstructure consists of PF + P. The decreased FCT contributes to a refinement in the effective ferrite grain size and interlaminar spacing of pearlite, coupled with a decrease in PF content. Conversely, the amount of high misorientation angle boundary (above 15°) is increased. Moreover, the lower FCT promotes the precipitation of refined nano-scaled V(C,N) particles including inter-phase and random precipitates. Furthermore, an optimal combination of strength, ductility and toughness has been acquired at 600°C, of which the yield strength, tensile strength, total elongation, uniform elongation, and impact energy at room temperature are 769 MPa, 935 MPa, 23 pct, 11 pct and 30 J, respectively. The negligible variation of mechanical properties after artificial ageing exhibits that the addition of 0.024 mass% N in vanadium alloyed steel is available.

KEY WORDS: finish cooling temperature; simulated and pilot-scaled thermomechanical processes; microstructure; precipitates; mechanical properties.

1. Introduction

The high strength low alloy steel (HSLA) with enhanced mechanical properties is commonly applied as structural steel in architectural building,1) automobile,2) energy transportation,3) etc. To satisfy the increasingly complicated structure, such as large-span, light-weight and long-distance, and to reduce the heavy environmental pressure and huge waste of energy resources, structural steel currently tends to be high strengthened, accompanying with good ductility and toughness.4–7) For purpose of improving mechanical properties, the microalloying technology combined with thermomechanical controlled process (TMCP) is frequently applied to absolutely realize the synergistic mechanism of precipitation strengthening, grain refinement, dislocation strengthening and so on.8–12)

The microalloy element of Nb, V, Ti, Mo, etc., is commonly added into the low carbon steel individually or in combination to form carbide and/or carbonitride precipitates which significantly influence the final microstructure and mechanical properties.13–18) Among them, V combined with N has been recognized as an effective method of improving precipitation hardening and grain refinement strengthening because adding nitrogen in V alloyed steel greatly enhances the formation of vanadium precipitates, coupled with their large solubility. On the one hand, VN and V(C,N) particles could act as the heterogeneous nucleation sites for intragranular ferrite (IGF), which obviously refines the grain size and thus increases the grain refinement strengthening to yield strength, as well as improves the toughness.19,20) On the other hand, the nano-scaled VC and V(C,N) particles precipitated in matrix, on dislocations and at grain boundaries could play an essential role in precipitation strengthening.21–25) It was reported that the contribution of precipitation hardening to yield strength reached 300 MPa in V–Ti microalloyed steel with appropriate TMCP.26,27) Besides, the microstructural characteristics including grain size, micro-constituents and grain boundary are also directly related to TMCP parameters, and consequently determine mechanical properties.28–30) The required microstructure for our developed V–N alloyed high strength structural steel is composed of PF, P and/or a small amount of B. Therefore, to completely utilize the V–N alloying effect on strengthening and toughening the steel, it is necessary to design an optimal TMCP well matching V–N alloying to control...
The process of high temperature rolling, accelerated cooling to FCT and slow cooling to room temperature is applied to produce high strength structural steel. The high temperature rolling not only relieves the mill load but also improves the production efficiency and weldability. The accelerated cooling procedure after hot deformation could dramatically refine the austenite grain, resulting in the fine substructure such as ferrite grain, pearlite lamellar and bainitic lath. Furthermore, it leads the carbonitride forming elements supersaturated in the steel and the fine particles adequately precipitated during the followed slow cooling process, which as a result promotes carbonitrides precipitation and avoids coarsening. Undoubtedly, a reasonable FCT is the crucial parameter to obtain excellent mechanical properties.

Few literatures paid attention to the abovementioned thermomechanical process in V alloyed steel containing high content of 0.024 mass% N, which effectively decreases the quantity of vanadium and the production cost. Meanwhile, the ageing effect caused by free nitrogen atoms has been evaluated by comparing the mechanical properties before and after artificial ageing to confirm whether this nitrogen content is excessive or not. The aim of the present work is to develop a high strength structural steel in good combination with ductility and toughness by investigating the relationship between microstructure, precipitates, mechanical properties and FCTs ranging from 950°C to 550°C under the simulated and pilot-scale condition. From the study of dilatometry specimens, the controlled FCT range could be narrowed, laying the basis of designing the pilot-scaled thermomechanical process which is more approaching to the actual production.

2. Materials and Experimental Procedure

2.1. Materials and Thermomechanical Controlled Processes

The experimental steel was melted by vacuum induction furnace and cast into a 39 kg ingot. The chemical composition (mass fraction) of the experimental steel is 0.23 C, 0.74 Si, 1.54 Mn, 0.007 P, 0.004 S, 0.22 V, 0.024 N (V/N ratio = 9) and the balance Fe.

A two-stage continuous cooling process after hot deformation was conducted using a Gleeble-1500 thermomechanical simulator and schematically shown in Fig. 1. The dilatometry specimens with a diameter of 8 mm and length of 15 mm were firstly cooled to different FCTs ranging from 950°C to 550°C, at cooling rate of 15°C/s, and then slowly cooled to room temperature (RT) at an average rate of 0.5°C/s. The process with FCT of 950°C is actually equals to directly cooling to RT at 0.5°C/s, respectively. The cooling path A is equals to the two-stage continuous cooling procedure with a FCT of 950°C.

Considering that ageing effect may be happened in the steel with high content nitrogen, the samples under pilot-scaled condition were treated with artificial ageing (300°C × 1 h) by using a muffle furnace and then air cooled to RT.

2.2. Thermodynamic Calculation

The thermodynamic calculation on equilibrium phase evolution and chemical composition of V(C,N) as a function of temperature was calculated using the Thermo-Calc software based on TCFE8 database.

2.3. Microstructural Analysis

Microstructural study was conducted by use of optical microscope (OM, 9XB-PC), scanning electron microscope (SEM, FEI MLA250), conventional and high resolution transmission electron microscope (HRTEM, FEI Tecnai G² F30) equipped with energy dispersive spectrometry (EDS, Ametek EDAX). Besides, electron back-scattered diffraction (EBSD) was applied to study the distribution of effective ferrite grains and characterization of grain boundary. The EBSD maps were scanned via field emission scanning electron microscope (FE-SEM, Zeiss Auriga) in conjunction with HKL-Channel 5 software (Oxford Instruments). The scanning step length was set as 0.5 μm and the scanned area was about 232 μm×174 μm. The effective grain size was obtained from EBSD orientation map, and the minimum boundary misorientation is 15°. The detailed preparation for metallographic specimens was described in our previous work. Notably, as the ferromagnetic nature of the experimental steel made HRTEM analysis particularly difficult, the specimens needed degaussing.

The relative amount of micro-constituents and the average size of ferrite grain and precipitated particles were statistically measured by image software Image-Pro Plus™ 6.0 (Media Cybernetics, Inc., Rockville, MD).

2.4. Mechanical Properties

Vickers hardness was measured at ten randomly selected regions under a load of 200 g using a 430SH type Vickers tester. The average values reflected the variation of hardness for the experimental steel with different FCTs.

Tensile specimens with 5 mm gauge diameter and 30 mm gauge length, machined from the plates parallel to rolling...
direction, and the Charpy impact specimens with a dimension of 10 mm × 10 mm × 55 mm, machined from the plates with a v-notch parallel to rolling direction. Tensile and impact tests were conducted at RT using a universal testing machine (CMT4150) and a pendulum impact testing machine (ZBC2452-B). The fracture surfaces of the specimens were observed by FE-SEM (JSM-6701F).

3. Results

3.1. Microstructure Characterization and Vickers Hardness under a Simulated Thermomechanical Condition

The optical and scanning electron micrographs of the experimental steel subjected to a two-stage continuous cooling process with different FCTs ranging from 950°C to 550°C, are illustrated in Figs. 2 and 3. From Figs. 2(a)–2(c) and Fig. 3(a), the microstructure consisted of PF and P is observed over a wide FCT interval of 950°C–625°C. At FCT of 600°C shown in Figs. 2(f), 2(g) and 3(b), the transformed microstructure is dominated by PF + P, coupled with a small amount of locally distributed B. It is evident that the critical temperature for the formation of B is about 600°C. With the further decrease of FCT, the volume fraction of bainite sharply increases and meanwhile the amount of F and P decreases. As shown in Figs. 2(h) and 3(c) corresponding to the FCT of 550°C, the micro-constituents are bainite, pearlite and fine ferrite grains formed along the prior austenite grain boundaries. In addition to the microstructure evolution, white spherical-shaped particle with a diameter of 0.64 μm is observed to be distributed in ferrite grains, as shown in Fig. 3(d). Based on the EDS spectrum presented in Fig. 3(e), the precipitated particle is nitrogen-enriched vanadium carbonitride.

Fig. 2. Representative OM micrographs of the experimental steel at different FCTs: (a) 950°C; (b) 750°C; (c) 700°C; (d) 650°C; (e) 625°C; (f) 600°C (Field 1); (g) 600°C (Field 2); (h) 550°C.

Fig. 3. Representative SEM micrographs of the experimental steel at different FCTs and the chemical composition of the precipitated particle (Point 1): (a) 750°C; (b) 600°C; (c) 550°C; (d) magnification of the zone marked in the red frame; (e) EDS spectrum. (Online version in color.)
The statistically quantitative data including relative amount of micro-constituent and average size of ferrite grain is summarized in Fig. 4, by randomly measuring ten fields with an area of 200 μm × 250 μm. From Fig. 4(a), the volume fraction of PF with white-etching contrast in optical micrographs decreases from 62.6 ± 3.52 vol. pct to 31.9 ± 3.00 vol. pct, as the FCT decreases from 950°C to 550°C. The ferrite grain size is refined from 9.5 ± 0.74 μm to 3.3 ± 0.21 μm, as shown in Fig. 4(b). The decreasing rate of the volume fraction and average grain size of PF at low FCTs tends to be faster than that at the higher FCTs. In terms of pearlite (dark-etching contrast), the volume fraction is firstly increased from 37.4 ± 3.52 vol. pct to 58.1 ± 1.94 vol. pct and then decreased to 34.3 ± 3.50 vol. pct. The maximum value is obtained at 600°C. In parallel, the bainite which is grey-etching contrast appeared at 600°C with the volume fraction of 4.0 ± 1.22 vol. pct (less than 5 vol. pct). And it is sharply increased to 33.8 ± 1.83 vol. pct, when the FCT reaches 550°C. It should be noted that standard deviation values are calculated to represent error (similarly hereinafter).

In addition, Fig. 4(b) also presents the Vickers hardness versus FCTs ranging from 950°C to 550°C. The average micro-hardness values, indicative of the strength for the experimental steel, are corresponding to the transformed microstructure obtained at different FCTs. As the FCT decreases from 950°C to 625°C, the increased volume fraction of P and the refinement of PF result in progressively increasing the Vickers hardness from 235 ± 15 HV0.2 to 288 ± 22 HV0.2. When FCT is decreased to 600°C and 550°C, the hardness is largely increased to 294 ± 24 HV0.2 and 318 ± 26 HV0.2, attributed to the increase in grain boundary effect and the formation of bainite.

Based on OM and SEM microstructural observation, the FCT after a 950°C finish deformation should be controlled in the range of 600°C–950°C to obtain microstructure composed of PF, P and/or small amount of B in V–N alloyed steel.

3.2. Mechanical Properties under a Pilot-scaled Thermomechanical Condition

According to the result under a simulated condition, three different cooling paths with FCTs of 950°C, 750°C and 600°C after high temperature deformation are designed as the pilot-scaled TMCP. On the one hand, it could obtain the data of mechanical properties, because the size of diametery specimens were not enough for tensile and impact dimensions. On the other hand, it is more approaching to the industrial practice, which could provide necessary data support on V–N alloying and appropriate themomechanical parameters.

Tensile and impact properties for three different cooling processes with and without artificial ageing including yield strength (ReL), tensile strength (Rm), total elongation (A), uniform elongation (Agt) and the Charpy impact energy (Av) are listed in Table 1. The data given in Table 1 is average value of at least three standard tensile and impact specimens.

As the FCT decreases from 950°C to 600°C, the yield and tensile strength increase from 717 MPa to 769 MPa and from 886 MPa to 935 MPa, respectively. Moreover, the uniform elongation and total elongation are the same or similar and in the range of 22 pct to 24 pct. For the impact energy, they are basically the same with the value of 29 J and 30 J. Therefore, the increase in strength for the experimental steel is not accompanied by loss of ductility and toughness. Furthermore, the optimal mechanical properties are obtained from cooling path C and its yield strength is much higher than 700 MPa.

In order to exhibit the ageing effect caused by free nitrogen atoms, the values of tensile and impact tests before and after artificial ageing have been compared. It is obvious that the yield strength and tensile strength are slightly increased in the range of 0.4%–2.1% and 0.6%–0.8%. The ductility and impact toughness are slightly decreased. More importantly, the ageing effect is negligible for cooling path C and it is probably that the nitrogen is almost existed in the form of V(C,N) precipitates. The addition of nitrogen in V alloyed steel is not excessive and the alloy design for the experimental is available.

Figure 5 illustrates the typical engineering stress-strain curves for the experimental steel with three different cooling paths. It is obvious that the steel exhibits pronounced discontinuous yielding behavior which resulted from the

| Table 1. | Results of tensile and impact tests with and without artificial ageing. |
|----------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Cooling paths | ReL (MPa) | Rm (MPa) | A (pct) | Agt (pct) | Av (J) |
| Path A | 717 ± 5 | 886 ± 4 | 24 ± 1.2 | 11 ± 0.7 | 29 ± 1 |
| Path B | 735 ± 3 | 915 ± 9 | 22 ± 1.6 | 11 ± 0.5 | 30 ± 3 |
| Path C | 769 ± 8 | 935 ± 6 | 23 ± 0.4 | 11 ± 0.1 | 30 ± 2 |
| Path A + Ageing | 732 ± 7 | 893 ± 10 | 22 ± 2.5 | 10 ± 0.3 | 24 ± 1 |
| Path B + Ageing | 744 ± 6 | 922 ± 8 | 22 ± 1.0 | 10 ± 0.2 | 26 ± 2 |
| Path C + Ageing | 772 ± 14 | 941 ± 10 | 22 ± 0.3 | 10 ± 0.1 | 27 ± 1 |

Fig. 4. (a) the relative amount of micro-constituents versus FCT; (b) the average value of ferrite grain size and Vickers hardness versus FCT (The error bars represent standard deviation). (Online version in color.)
interactions between dislocations and carbonitride precipitates as well as the interstitial solute atoms, principally C.

The typical SEM micrographs of the tensile and impact fracture surface for cooling path C at low and high magnifications are taken for example, as illustrated in Fig. 6, and there is no observable change existed in the tensile and impact samples subjected to the different cooling processes. From Fig. 6(a), the overall fracture morphology is the well-known cup-and-cone in shape and could be divided into three zones A, B and C. In the center of specimens (Fig. 6(b)), the morphology of zone A is characterized by deep dimples of different sizes, indicative of typical ductile fracture. For the morphology of zone B (Fig. 6(c)), the number of small size dimples is increased and the dimples become flat, compared to that of zone A. The morphology of zone C (Fig. 6(d)) is presented by flat and elongated dimples and some voids due to the inclusions or large precipitated particles dropping from matrix. As for the impact fracture morphology, it could be divided into three zones including ductile region, brittle region and shear lips apart from v-notch, as shown in Fig. 6(e). In terms of the SEM micrographs at high magnification in Fig. 6(f), the ductile region is mainly composed of deep dimples of large and small sizes. As shown in Fig. 6(g), the brittle region consists of small quasi-cleavage facets, large cleavage facets, elongated dimples and tear ridges. In Fig. 6(h), the shear lips region is composed of the relatively large and elongated dimples.

4. Discussion

4.1. Microstructure Characterization under a Pilot-scaled Thermomechanical Condition

The typical OM and TEM micrographs of the experimental steel subjected to controlled rolling and three different cooling processes are shown in Figs. 7 and 8. The microstructures consist of PF and P. From the observation of substructure, the different orientation ferrite grains contained dislocations are presented, as well as the paralleled lamellar pearlite. For cooling paths A, B and C, the polygonal ferrite accounts for 65.5 ± 2.51 vol. pct, 62.5 ± 1.40 vol. pct, and 60.8 ± 1.77 vol. pct, respectively. Compared to the experimental steel subjected to cooling path A, the ferrite grain coupled with laminar pearlite is significantly refined and a higher volume fraction of pearlite forms for cooling paths B and C. During the hot rolling process, a large amount of defects generated by the heavy deformation such as deformation bands, subgrains and dislocations are nucleation sites for ferrite, which promotes the formation of PF. The \( \gamma \rightarrow \alpha \) transformation leads to the diffusion of carbon atoms, which makes the neighboring supercooled austenite carbon enriched and transformed into pearlite. With the accelerated cooling, it takes less time to pass through the (\( \gamma + \alpha \)) region, resulting in less volume
fraction of PF and increased amount of P. The grain size of PF is determined by its nucleation rate and growth rate. The lower FCT restrains the grain boundary migration and growth of ferrite grains so that the decrease of ferrite grain size becomes inevitable. Moreover, the refined interlaminar spacing of pearlite is also directly related to the increase of supercooling degree caused by decreasing FCT.

Figure 9 shows the crystallographic characteristics of microstructure analyzed by EBSD for three cooling paths. As shown in Figs. 9(a)–9(f), the effective grain size considering misorientation criteria of 15° decreases from 6.5 μm to 6.0 μm and 4.4 μm for cooling paths A, B, and C.
respectively. Meanwhile, the microstructural homogeneity for cooling path C is better than that for the other two cooling paths because the obtained size distribution range is narrower, as well as larger frequency of fine ferrite grains. In Figs. 9(g)–9(i), the black lines represent the low misorientation angle boundaries of 2°–15°, while the red lines represent the high misorientation angle boundaries above 15°. Combined with the data in Figs. 9(j)–9(l), the misorientation spectrum corresponding to the process of cooling path A is characterized by a higher fraction of low misorientation angles less than 5°, whereas a higher fraction of high misorientation angle grain boundaries are obtained for cooling paths B and C. The number fraction of high misorientation angle boundaries for different cooling paths follows the sequence: path A < path B < path C, which is 43.6 pct, 54.4 pct, and 58.0 pct, respectively.

4.2. Precipitation Behavior under a Pilot-scaled Thermomechanical Condition

The equilibrium phase diagram for the experimental steel is presented in Fig. 10, which summarizes the nature and stability of V(C,N). From Fig. 10(a), the V(C,N) precipitation began at 1230°C for precipitation in equilibrium. Compared to that of the steel bearing 0.019 mass% N, the temperature is increased from 1210°C to 1230°C.34) Increasing nitrogen content could promote the formation of vanadium carbonitrides when the V/N ratio is largely higher than 3.64. Besides, the start and end temperatures of austenite to ferrite transformation are 814°C (Ae3) and 700°C (Ae1), respectively. From Fig. 10(b), the precipitates are nitrogen-enriched carbonitrides at austenitic region, while precipitates forming at ferrite single region and ferrite + austenite two-phase region are carbon-enriched. It is obvious that the particle (Point 1) shown in Fig. 3(d) was precipitated at the temperature of austenite region which could be the nucleation site of ferrite grains, due to the lower lattice mismatch between the precipitated particle and ferrite grain, and consequently it enhances grain refinement strengthening to yield strength and raises impact toughness.

The bright field and dark field TEM images of the nano-scaled particle and the corresponding EDS analysis are shown in Fig. 11. The selected area electron diffraction (SAED) pattern is inserted in Fig. 11(b). The small precipitate with a diameter of 77.3 nm were identified as C-rich V(C,N), based on EDS and SAED analysis. In combination with N-rich V(C,N) with a diameter of 0.64 μm shown in Fig. 3(d), it is obvious that the experimental result of V(C,N) chemical composition is consistent with the thermodynamic calculation in Fig. 10(b).

TEM studies of thin film of the precipitated particles are presented in Fig. 12. From Figs. 12(a) and 12(b), the bright field and dark field TEM images exhibit the presence of numerous particles precipitated in ferrite matrix, greatly contribute to precipitation strengthening. In Fig. 12(c), the mutual interaction between precipitates and dislocations, coupled with dislocation tangle or stacking could be observed in ferrite grain, which greatly increases the dislocation strengthening to yield strength. In addition, precipitates pinning on grain boundary are indicated by black arrows in Fig. 12(d), which could inhibit the growth of ferrite grain and increase the yield strength.

Figure 13 exhibits the representative TEM micrographs showing interphase precipitation (IP), random precipitation (RP) and HRTEM images of nano-scaled particles obtained.
in the experimental steel subjected to three different cooling paths. The parallel arrays of IP with different orientations and sheet spacings are shown in Figs. 13(a)–13(c). The row-like IP morphology is difficult to exhibit in TEM observation because $\alpha$ and $\beta$ directions of thin foils need to be tilted to satisfy the specific condition that the direction of incident electron beam is parallel to the zone axis of sheet plane on which carbonitrides precipitated. The statistical results reveal that the sheet spacing varies in the range of 27.4–30.7 nm for cooling path A. For cooling paths B and C, the sheet spacing is refined to 22.8–25.1 nm and 12.3–15.5 nm, respectively. As the FCT decreases, the $\gamma\rightarrow\alpha$ transformation temperature is decreased and the driving force increases simultaneously, which accelerates the mobility of $\gamma\alpha$ interface, and eventually refines the sheet spacing.

From Figs. 13(d)–13(f), for cooling paths A and B, the low volume faction of V(C,N) particles are optionally precipitated in the ferrite matrix. The precipitates obtained from cooling path C are finer, more numerous and more uniformly distributed. To quantitatively compare the nano-scaled precipitates, size distribution, average particle size and number density have been statistically investigated, as presented in Fig. 14. In Fig. 14(a), the size of precipitates in the experimental steel with cooling path A is mainly in the range of 2.0–24.0 nm. Precipitates of size 4.0–6.0 nm account the highest density, which is slightly higher than that of 6.0–8.0 nm. For cooling path B, the size distribution is similar to that of cooling path A as a whole, whereas the number fraction of particle with a size of 2.0–4.0 nm is obviously higher and the distribution range becomes narrower. In terms of cooling path C, the size of precipitates is mainly in the range less than 20 nm. Precipitates of size 2.0–4.0 nm account the highest density. The number fraction of small size precipitates follows the sequence: path

![Fig. 12. The typical TEM micrographs of nanoscaled particles: (a) bright field image of precipitates within a ferrite matrix; (b) dark field image of precipitates within a ferrite matrix; (c) precipitates on dislocations; (d) precipitates at the grain boundary.](image1)

![Fig. 13. Bright field TEM micrographs of nano-scaled V(C,N) precipitates in the experimental steel with different cooling paths: (a)–(c) interphase precipitation; (d)–(f) random precipitation; (g)–(h) HRTEM images; (a), (d) and (g) cooling path A; (b), (e) and (h) cooling path B; (c), (f) and (i) cooling path C.](image2)
C > path B > path A. From Fig. 14(b), the average particle size for cooling paths A, B and C was 6.3 ± 1.55 nm, 5.9 ± 1.53 nm and 5.1 ± 1.32 nm, respectively. As plotted in Fig. 14(c), the number density of particle with a size below 10 nm follows the sequence: path C > path B > path A. The number density of above 10 nm precipitates follows the opposite sequence but the distinction is imperceptive. It could be concluded that decrease in FCT from 950°C to 750°C not only increases the precipitate number density but decreases the average size to a small extent. Further decreasing FCT to 600°C largely refines the precipitate size and enhances the number density. It is evident that combining high temperature rolling with accelerated cooling to 600°C and slowly cooled to RT has a promotion effect on the formation and refinement of nano-scaled precipitates. The size and number density of randomly precipitated particles are directly related to the nucleation driving force and the kinetic diffusion rate of V atoms. Moreover, the dislocation defects are preferred heterogeneous nucleation sites for precipitation, because they provide diffusion pipelines for atoms, as shown in Fig. 12(c). During hot rolling and continuous cooling process, the high density of dislocations is generated by the heavy deformation and \( \gamma \rightarrow \alpha \) transformation stress due to the lattice structure change from FCC to BCC. Therefore, the accelerated cooling process enhances the formation of dislocations and the nucleation sites for V(C,N) precipitation is increased in the steel with lower FCT. Combined with the increased supercooling, V(C,N) precipitation is promoted with the enhanced nucleation driving force. In addition, the diffusion rate of V atoms is decreased, which tends to suppress the growth of the precipitates and finally refines the V(C,N) size. Furthermore, V(C,N) particles could not completely precipitate or even nucleate in the water cooling process and V atoms are reserved in solution, which prevents the coarsening of V(C,N) particles and promotes the nucleation of fine nano-scaled particles during the followed air cooling process at lower temperature.

HRTEM is applied to characterize the morphology and atomic arrangement of the fine V(C,N) particles in the experimental steel with different cooling paths, as shown in Figs. 13(g) through 13(i). The aspect ratios are 1.02, 1.05, and 1.02, corresponding to cooling paths A, B, and C, respectively. These results provide evidence that the particles precipitated within the ferrite matrix are approximately spherical in shape. It should be noted that the moiré fringes could be clearly observed in Fig. 13(h), due to their diffraction effect between the precipitated particle and ferrite matrix.

### 4.3. Relationship between Microstructure, Precipitation Behavior and Mechanical Properties

For the experimental steel composed of PF + P, the high yield strength is attributed to the following multiple strengthening mechanisms: (1) grain boundary strengthening caused by nucleation of ferrite and pinning effect of particles precipitated at grain boundary; (2) precipitation strengthening from the nano-scaled vanadium carbonitrides particles; (3) dislocation strengthening resulted from heavy deformation, accelerated cooling procedure and interaction between dislocations and precipitates; (4) transformation strengthening of hard phases such as cementite island in lamellar pearlite; (5) solid solution strengthening from the atoms of C, Si and Mn. The yield strength of the experimental steel subjected to cooling paths A and B is 717 MPa and 735 MPa, respectively, which is just above 700 MPa. By contrast, it is increased to 769 MPa for cooling path C, which is rather much higher than 700 MPa. According to strengthening mechanism, the reason is that the effective ferrite grain and precipitated particles are refined with the decrease of FCT, as well as the increased number density of precipitates and volume fraction of pearlite. Therefore,
the accelerated cooling procedure with the lower FCT of 600°C brings about more contribution of grain refinement strengthening, precipitation strengthening and transformation strengthening to the yield strength. Moreover, in comparison with the microstructure and mechanical properties of the steel containing 0.019 mass% N, the increased strength caused by higher N content was primarily attributed to grain refinement strengthening and precipitation strengthening.\textsuperscript{34)

The toughness can be represented by the total impact energy which is comprised of crack initiation energy and crack propagation energy. For the PF energy which is comprised of crack initiation energy and refinement strengthening and precipitation strengthening.

caused by higher N content was primarily attributed to grain

The EDS result on chemical composition of precipitates is in [2018 ISIJ 1892]

Acknowledgements

The authors gratefully acknowledge the financial support from the scientific innovation fund of Inner Mongolia University of Science and Technology (Grant No. 2016 QDL-B16) and the scientific research project of higher education in Inner Mongolia autonomous region (Grant No. NJZY18144).

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5. Conclusion

(1) From microstructural observation of dilatometry specimens, the FCT should be controlled in the range not lower than 600°C to obtain microstructure composed of PF, P and/or a small amount of B (less than 5 vol. pct) in V–N alloyed steel.

(2) Under a pilot-scaled condition, all microstructure for different cooling paths consists of PF + P. With the decrease of FCT, the volume fraction of PF is decreased. The effects of strain concentration. Therefore, the volume fraction of pearlite and its interlamellar spacing are decisive to crack initiation energy. Conversely, the high misorientation angle boundary could change crack growth direction or even arrest cracks, which yields crack propagation and results in high impact energy.\textsuperscript{35)

Besides, the occurrence of dimples and tear ridges, as shown in Fig. 6(g), could contribute to the total impact energy. Compared to cooling path A, the lower FCT significantly refines the interlamellar spacing and increases the amount of high misorientation boundary and the volume fraction of pearlite. Since the total impact energy for different cooling paths is similar, it is reasonably indicated that the steel with cooling path A has higher crack initiation energy and lower crack propagation energy than those of the steel with cooling paths B and C. The deterioration effect of pearlite on toughness is weakened by its smaller interlamellar spacing and remedied by the high misorientation angle boundary, dimples and tear ridges.

(3) The lower FCT largely increases the yield and tensile strength without the loss of elongation and impact energy. The optimal mechanical properties are obtained from the steel at FCT of 600°C. The corresponding ageing effect is negligible and it exhibits that the addition of 0.024 mass% N in V alloyed steel is not excessive.

(4) The beneficial effect of this work is so evident that mechanical properties are improved by decreasing FCT, which is more cost and energy saving in comparison with the common strengthening measures taken in actual production, such as adding microalloy element, increasing deformation or lowering rolling temperature.