



# Influence of cooling paths on microstructural characteristics and precipitation behaviors in a low carbon V–Ti microalloyed steel

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## ABSTRACT

Based on ultra fast cooling, the microstructural characteristics, precipitation behaviors and mechanical properties of a low-carbon V–Ti microalloyed steel were investigated in details using optical microscope, electron back-scattered diffraction and transmission electron microscope. The results show that the ferrite grains can be slightly refined, the sheet spacings of interphase precipitation can be also slightly reduced and the number fraction of ferrite grains with higher precipitation hardening can be significantly enhanced by increasing cooling rate (by comparisons of air cooling and furnace cooling), and a ferritic steel precipitation-strengthened by nanometer-sized carbides was developed to produce hot rolled high strength steel with the tensile strength of  $\sim 810$  MPa, elongation of  $\sim 24\%$  and yield ratio of  $\sim 0.82$ . While for furnace cooling after ultra fast cooling, its tensile strength, elongation and yield ratio is only  $\sim 750$  MPa,  $\sim 22\%$  and  $\sim 0.84$ , respectively. The interphase precipitation in V–Ti microalloyed steel was observed, and these nanometer-sized carbides were detected as (V, Ti)C using energy dispersive X-ray spectroscopy spectra. In addition, the precipitation hardening was estimated as  $\sim 313$  MPa and  $\sim 293$  MPa for air cooling and furnace cooling after ultra fast cooling, respectively.

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## 1. Introduction

Microalloyed steels utilize chemical composition design of low carbon content and are microalloyed with niobium, vanadium and titanium, or other additions, such as molybdenum, boron, etc. The increase of strength is attributed to grain refinement, solid-solution strengthening, dislocation strengthening and precipitation hardening. Moreover, the precipitation hardening attracts more and more researchers' attentions. In 2004, Funakawa et al. developed Ti–Mo bearing high strength steels with tensile strengths of 780 MPa and excellent formability. The microstructure of these steels consists of ferritic matrix with nanometer-sized carbides, and the precipitation hardening due to these nanometer-sized carbides has been estimated as  $\sim 300$  MPa [1]. Since then, the mechanism of interphase precipitation and precipitation hardening have been further understood [2–8]. Moreover, The (Ti, Mo)C [1–5], VC [6], (Nb, Ti)C, (Nb, Ti, Mo)C [7], NbC, V(C, N), (V, Nb)C and (V, Cr)C [8] interphase precipitation has been observed. However, the interphase precipitation, the chemical composition of precipitates and precipitation hardening in V–Ti microalloyed steel have not been reported in details. Besides interphase precipitation behaviors in microalloyed steel, effects of chemical composition and thermomechanical parameters on transformation in microalloyed steels were also investigated [9–12]. For hot rolling practices, strain-

induced precipitation can hardly be avoided due to relatively longer rolling time, which significantly lowers precipitation hardening. But using ultra fast cooling can greatly suppress the amount of precipitation in austenite and increase supersaturation ratio in ferrite [13], and the precipitation hardening can be enhanced. So, at the condition of ultra fast cooling, it is of significance to understand effects of cooling paths on microstructure, precipitation behaviors and mechanical properties in V–Ti microalloyed steel.

In the current study, the effects of cooling paths on microstructure, interphase precipitation behaviors and mechanical properties were investigated using optical microscope (OM), electron back-scattered diffraction (EBSD), transmission electron microscope (TEM), tensile testing and Vickers-microhardness testing. Microstructural characteristics and precipitation behaviors for two different cooling paths were clarified. The chemical composition of nanometer-sized carbides was detected using qualitative energy dispersive X-ray spectroscopy (EDXS). The amount of precipitation hardening was also estimated.

## 2. Experimental procedure

The chemical composition of the tested steel is shown in Table 1. The alloy was prepared by vacuum melting and then cast into ingots, which were forged into 70 mm  $\times$  70 mm  $\times$  100 mm square billets.

The square billets were hot rolled using two-high 450 mm experimental hot rolling mill followed by ultra fast cooling (UFC) system. The schematic representation of thermomechanical control

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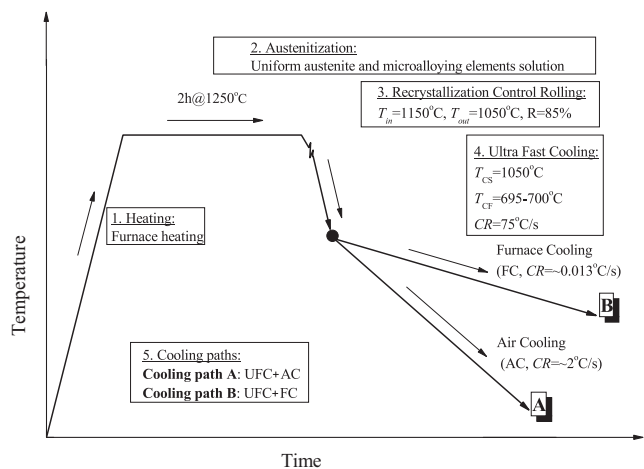
process (TMCP) is shown in Fig. 1. The billets were reheated to 1250 °C and held for 2 h to dissolve all of the carbides. After that, the recrystallization controlled rolling with the initial rolling temperature ( $T_{in}$ ) of  $\sim 1150$  °C, the finishing temperature ( $T_{out}$ ) of  $\sim 1050$  °C and the total reduction ratio ( $R$ ) of  $\sim 85\%$  was performed. And the reduction schedule is 70 mm  $\rightarrow$  52 mm  $\rightarrow$  38 mm  $\rightarrow$  28 mm  $\rightarrow$  22 mm  $\rightarrow$  17 mm  $\rightarrow$  13 mm  $\rightarrow$  10 mm. After hot rolling, the hot rolled plates were cooled to room temperature using cooling path A or cooling path B.

Specimens were cut from hot rolled plates and their surfaces along thickness direction and rolling direction were mechanically polished and then etched in 4% nital solution for the observation of OM (LEICA DMIRM). The specimens were also electropolished in a mixture of 12.5% perchloric acid and 87.5% absolute ethyl alcohol at 25 °C using voltage of 20 V for 20 s. The thin foils were prepared by mechanical abrasion at first and then twin-jet electropolished in a mixture of 9% perchloric acid and 91% absolute ethyl alcohol at  $-40$  °C using voltage of 30 V. The thin foils were examined using FEG-TEM (FEI Tecnai G<sup>2</sup> F20).

Mechanical properties in rolling direction were tested using CMT-5105 electron universal testing machine controlled by computer. Standard round tensile samples with diameter of 8 mm, original gauge length of 40 mm and parallel length of 60 mm were tested at room temperature with a cross beam speed of 3 mm/min.

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

C	Si	Mn	P	S	V	Ti	Al	N
0.06	0.31	1.31	0.005	0.003	0.06	0.11	0.06	0.0042



**Fig. 1.** Schematic representation of thermomechanical control process.

The yield strength, tensile strength and elongation were all determined. In order to investigate precipitation hardening, The Vickers-microhardness within ferrite grains was tested using Vickers-durometer with a load of 25 g. The testing standard followed the guidelines of ISO 6507-1: 2005(E) [14].

### 3. Results and discussions

#### 3.1. Microstructural characteristics and precipitation behaviors with two different cooling paths

##### 3.1.1. Optical metallography

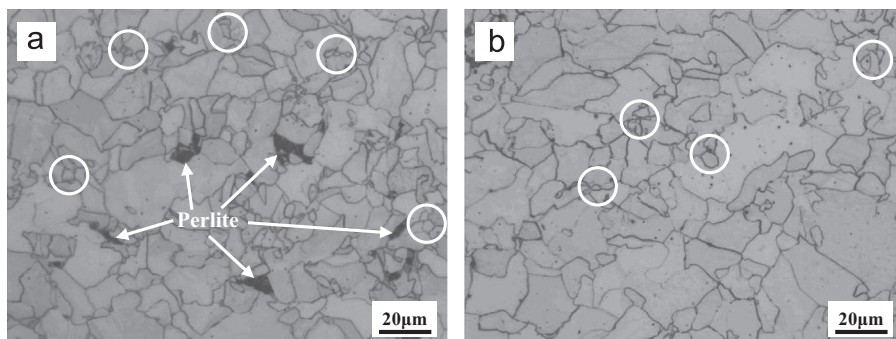
The optical metallographs of tested steel with two different cooling paths are shown in Fig. 2. For cooling path A, the microstructure composed of ferrite and perlite can be observed. The white phase is allotriomorphic ferrite, and the dark phase is perlite, marked as white arrows in Fig. 2(a). Some fine ferrite grains in local zones can be observed, marked as white circles in Fig. 2(a). While for the cooling path B, only ferrite phase can be observed. Some fine ferrite grains in local zones can be also observed in Fig. 2(b), also marked as white circles in Fig. 2(b). However, the number of these fine grains for cooling path A is pronouncedly higher than that for cooling path B.

##### 3.1.2. Electron back-scattered diffraction orientation maps

The results of electron back-scattered diffraction (EBSD) analysis are presented in Fig. 3, showing the low-angle grain boundaries with misorientation ranging from 2° to 15° in white lines and high-angle grain boundaries with misorientation higher than 15° in black lines. The ferrite grain size distributions of tested steel are depicted in Fig. 4. It can be clearly seen that all ferrite grains show irregular shape and a great number of small ferrite grains can be observed, marked as black arrows in Fig. 3(a) and (b), but the number of fine ferrite grains ( $d = 3\text{--}5$  μm) for cooling path A is obviously higher than that for cooling path B, as shown in Fig. 4. This result is in better agreement with that of the observation of optical metallographs, indicating that using air cooling after ultra fast cooling can remain larger number of fine ferrite grains, however, the microstructural homogeneity for cooling path A is worse than that for cooling path B. In addition, the average ferrite grain size for cooling paths A and B was estimated as  $\sim 9.5$  μm and  $\sim 10.9$  μm, respectively, showing that the ferrite grains can be slightly refined by increasing cooling rate (by comparisons of air cooling and furnace cooling after ultra fast cooling).

##### 3.1.3. Transmission electron microscopy

It is well known that the parallel row-like characteristic of interphase precipitation can only be observed when the electron beam direction is paralleled to plans of row precipitates [3]. So we tilted thin foils to make the zone axis of plans of row precipitates



**Fig. 2.** Optical microstructure of tested steel with (a) cooling path A and (b) cooling path B.

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