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# Isochronal phase transformation of Nb–V–Ti microalloyed ultra-high strength steel upon cooling

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### ABSTRACT

The transformation behavior of Nb–V–Ti microalloyed ultra-high strength steel during continuous cooling was investigated by Gleeble-1500 thermal mechanical simulator. Thermal dilation curves were measured at different cooling rates, based on which continuous cooling transformation (CCT) curve was established. The microstructure at slow cooling rate, primarily consists of polygonal ferrite, pearlite and lower bainite, while at intermediate cooling rate besides dominated lower bainite, small amount of polygonal ferrite can also be observed. At higher cooling rate, martensite lath is obtained. The nano-sized precipitates at slow cooling rate include  $M_{23}C_6$ ,  $M_2C$ ,  $M_3C$ , rich-Nb MC and rich-Ti MC and at intermediate cooling rate and higher cooling rate, the type of precipitates were similar,  $M_3C$  and two types of MC. At cooling rates arging from 0.2 to 1 °C/s, Vickers hardness of the steel decreases significantly. While at intermediate and higher cooling rate, the Vickers hardness increases gradually with increasing cooling rate. In addition, the polygonal ferrite transformation start temperatures (Ar<sub>3</sub>) decreases with increasing cooling rate, which is related to the carbide precipitation.

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### 1. Introduction

In recent years, microalloyed ultra-high strength steels have been widely used in nuclear industry due to its ultra-high strength and good hardness [1]. However, the improvement of yield strength usually accompanies the deterioration of fracture toughness, which will initiate stress induced cracking. Therefore, it is important to develop the material that exhibits high strength and favored toughness and formability [2–5].

Under this background, it is essential to appropriately select microalloyed elements that can meet the requirements of ultrahigh strength and good toughness. Nb, V, Ti and Mo, ect. microalloyed elements are usually added for production of ultrahigh strength steels to obtain the desired microstructure and mechanical properties [6,7]. These elements can improve the strength of steels by mechanisms including grain refining, solid solution, dislocation and precipitation hardening [8]. Low alloy Cr-Mo ultra-high strength steels are widely used for the pressure vessel in nuclear industry due to its ultra-high strength, good impact toughness, and excellent creep resistance. The most important properties of these steels are ultra-high strength to

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http://dx.doi.org/10.1016/j.fusengdes.2017.05.025 0920-3796/© 2017 Elsevier B.V. All rights reserved. withstand internal pressure and sufficient impact toughness to assure safety from instantaneous shock [9]. The judicious selection of microstructure is also an important factor to further improve the strength and toughness behaviors of the microalloyed ultrahigh strength steels [10]. Proper control of cooling rate is one way to obtain optimized microstructures and mechanical performance [11–14].

Currently, considerable attention has been focused on controlling the microstructure in steel during continuous cooling [15,16]. However, the studies concerning multi-micro alloyed steels subjected to different cooling conditions have not been reported sufficiently, which should be paid more attention.

In this work, the transformation behavior during continuous cooling for Nb-V-Ti microalloyed ultra high strength steel is systematically investigated. The precipitation of carbide under different cooling rate conditions is carefully defined. The effects of precipitates on Vickers hardness values and ferrite transformation start temperatures are also discussed in detail.

### 2. Experimental

The chemical composition of investigated steel is given in Table 1. The steel was machined into  $8 \text{ mm} \times 10 \text{ mm}$  (diameter  $\times$  height) cylindrical samples for dilatometry test. The specimens were heated to 900 °C and held for 300 s, and then fol-

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

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С	Mn	Cr	Si	Р	S
0.025	0.39	0.97	0.29	0.005	0.001
N 0.006	Mo 0.85	Nb 0.012	V 0.089	Ti 0.026	

lowed by continuous cooling with different cooling rates of 0.2, 0.5, 1, 2, 3, 5, 10, 15, 30, 50  $^{\circ}$ C/s.

Specimens for metallographic observation were mechanically polished and etched with 4% Nital solution, and examined by optical microscopy. The continuous cooling transformation (CCT) diagrams were obtained according to the transformation temperature determined from dilatometric curves and the metallographic observation. For TEM observation, specimens were sliced to  $300\,\mu\text{m}$  thickness and mechanically polished to  $50\,\mu\text{m}$ , and then electrically thinned by a twin-jet polisher in a 7 vol.% perchloric acid and 93 vol.% ethanol solution. The morphology, size, distribution and chemical composition of second phase particles were determined through examining carbon extraction replicas in a IEM-2000F transmission electron microscopy. The carbon coating of 250 Å was deposited in a vacuum system. The film was scored with a sharp blade to divide it into several smaller squares. Then, chemical etching in 7% Nital was conducted to remove the carbon film with the extracted carbides. Finally, the small films were mounted on copper grids for TEM analysis. The hardness was determined with MH-6 Vickers hardness tester using a 100 g load. An image analyzer (Image Pro Plus 6.0) was used to quantitatively analyze the percentage of each phase and the sizes of the precipitates.

#### 3. Results and discussion

#### 3.1. Microstructural evolution

Representative optical micrographs of the specimens after cooling are shown in Figs. 1 and 2. At slower cooling rates  $(0.2-1 \circ C/s)$ , the microstructure are composed of polygonal ferrite, pearlite and lower bainite. As the cooling rate is increased from 0.2 to 1 °C/s, the amount of polygonal ferrite is reduced, and that of pearlite is increased gradually. As shown in Fig. 1, the contents of polygonal ferrite at different cooling rates (0.2, 0.5, 1 °C/s) are determined as 28.66%, 20.97% and 14.27% respectively. Under these three slower cooling rates, the relative amounts of lower bainite are close. With the increase in cooling rate, the grain sizes of polygonal ferrite decrease gradually. This is due to the increase of the cooling rate, the degree of super cooling is increased, as well as the driving force of phase transformation. On the other hand, the size of critical nucleation and the critical nucleation energy reduces, contributing to the increase in nucleation rate. The enhanced nucleation rate restricts grain growth due to impingement of intergranular and intragranular, leading to polygonal ferrite grain refinement [17]. In addition, at intermediate cooling rates  $(2-10 \circ C/s)$ , the microstructure is mainly dominated by lower bainite, but small amount of polygonal ferrite can be also observed. At higher cooling rates ( $\geq 10^{\circ}$ C/s), predominantly, lath martensite is obtained as illustrated in Fig. 2.

### 3.2. The CCT curves

A typical dilatometric curve of investigated steel at the cooling rate of 15 °C/s is shown in Fig. 3. The curve indicates the progress of heating and cooling cycles. As illustrated in Fig. 3, the start and finish temperature of martenite phase transformation can be detected clearly. The measured  $M_s$  and  $M_f$  temperature of experimental steel at the cooling rate of 15 °C/s are 420.3 and 332.3 °C, respectively.



Fig. 1. Optical micrographs of experimental steel after continuous cooling at (a)  $0.2 \circ C/s$ ; (b)  $0.5 \circ C/s$ ; (c)  $1 \circ C/s$ .

The critical transformation temperature under different cooling rates can be seen from CCT curve as shown in Fig. 4.

Fig. 4 shows the continuous cooling transformation (CCT) diagram of Nb–V–Ti microalloyed ultra-high strength steel. Phase transformation takes place with changes of volume, and these changes can be recorded by studying the length changes of samples during the heating and cooling process. Depending on the microstructures and cooling rate, the phase transformation temperatures can be determined. As shown in Fig. 4, the CCT diagram is characterized by multi-transformation curves. The complex

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