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Communication

Dissolving of Nb and Ti carbonitride precipitates in microalloyed steels

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Abstract: The dissolving behaviour of Nb and Ti carbonitride precipitates in microalloyed steels during isothermal holding at 1300℃ was investigated by Transmission electron microscopy (TEM) and energy dispersion x-ray spectrum (EDX). It was found that all precipitates in Nb-Ti microalloyed steel are (Nb, Ti)(C, N). With holding time increasing, the atomic ratio of Nb/Ti in precipitates decrease gradually. These precipitates still existe even after holding for 48 h at 1300℃ while Nb(C, N) precipitates dissolve away in Nb microalloyed steel only after 4 h at the same temperature. These results show that formation and thermostability of precipitates are considerably influenced by interaction between Nb and Ti.

Keywords: dissolving; (Nb, Ti)(C, N); TEM; EDX

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1 Introduction

Ti and Nb are frequently added to steel with distinct objects. TiN precipitates, which formed during solidification, can powerfully inherit the motion of austenite grain boundaries [1] while NbC, which usually precipitated after deformation of austenite, can pin dislocations so that recovery and recrystallization of defomed austenite can be intensively retarded [2-4]. While steels are heated to austenitize, it is generally believed that TiN precipitates can keep stable while NbC precipitates dissolve away rapidly [5]. However, when Nb and Ti are co-existed in same microalloyed steel, the effect of their interaction on formation and thermostability of precipitates, especially during solidification and austenization, were frequently neglected. That is what this paper aims to throw light on.

2 Materials and experimental procedures

The materials utilized in this investigation are microalloyed steels melt in a 25 kg vacuum induction finance and cast in vacuum. The chemical compositions of the tested steels are exhibited in **table 1**.

Table 1 The chemical compositions of the tested steels (mass fraction) %

Tested steel	C	Si	Mn	S	P	Nb	Ti
Nb microalloyed steel	0.048	0.28	1.75	0.0085	0.0071	0.044	—
Nb-Ti microalloyed steel	0.038	0.26	1.66	0.0086	0.0066	0.041	0.010

The ingots were forged into ϕ14 mm circular rods, then the rods were airproofed into vacuumed quartz tubes and isothermally held at 1300℃ for different time before quenched into water. Carbon extraction replicas of the samples etched in 2% Nital were prepared. The replicas were examined with an H8100 transmission electron microscope operated at 200 kV.

3 Result and discussion

3.1 The feature of the precipitates in original samples

A great number of precipitates, which appear as nearly rectangular, in Nb-Ti microalloyed steel were detected under TEM. By their size, they can be easily classified into two types: the first type is that the size (diagonal length) of precipitates is larger than 50 nm with half of them in size beyond 100 nm; the second type is that the size of precipitates is under 20 nm and most of them are in size less than 10 nm. By EDX, it was found that both of them are the complex precipitates (Nb,Ti)(C,N), the ratio of Nb/Ti of first type of precipitates is higher than 1, which is lower than that of second type of ones. The obvious distinction in size between the two types of precipitates implys their dif-

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ferent origin. In terms of references [6], the first type of precipitates are formed during solidification while the second type is strain induced precipitates during forging. **Figure 1** shows the two types of the precipitates.

There are two types of distinct precipitates in Nb microalloyed steel, which are shown in **figure 2**.

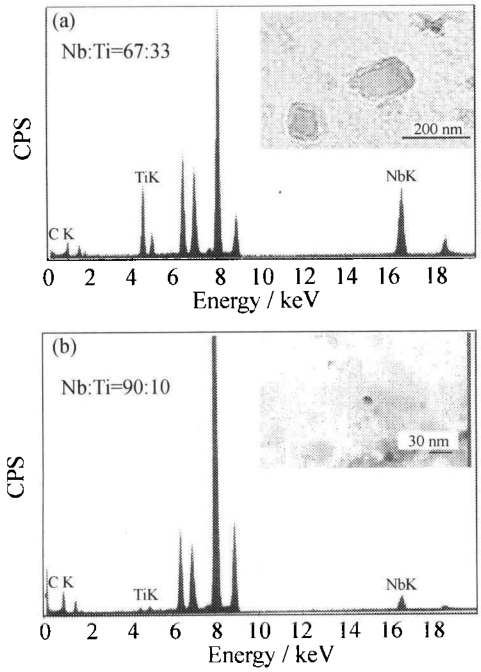

Figure 1 TEM images and corresponding EDX spectra in Nb-Ti microaloyed steel, (a) the first type of precipitates; (b) the second type of precipitates.

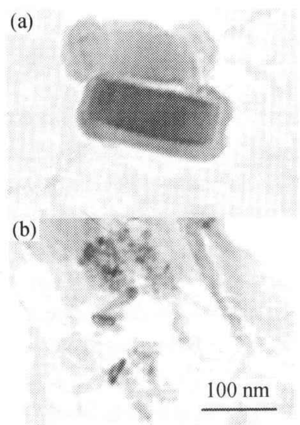

Figure 2 The two types of precipitates in Nb microalloyed steel; (a) the first type of precipitates; (b) the second type of precipitates.

3.2 Dissolving of the precipitates during holding at 1300℃

After reheating the Nb-Ti microalloyed steel at 1300℃ for 20 min, the distribution density of the second-type precipitates decreases obviously, while the ratio of Nb/Ti for the first type precipitates exhibites a little drop, as displayed in **figure 3**. After reheating the Nb-Ti microalloyed steel at 1300℃ for 2h, no second type of pricipitates could be detected while obviously perceived drop in the size and the ratio of Nb/Ti for the first-type precipitates occurre as shown in **figure 4**. With further holding, the drop continues slowly. However, after reheating for 48 h, there are still a few precipitates in the Nb-Ti microalloyed steel and Nb could be detected in these precipitates, which is shown in **figure 5**.

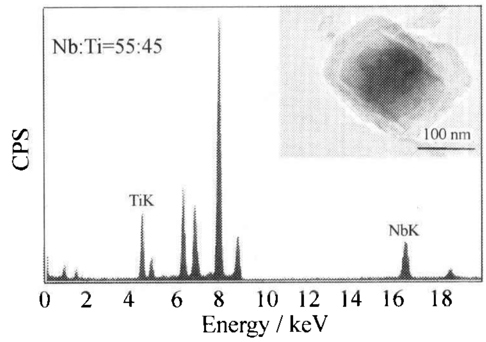

Figure 3 TEM image and corresponding EDX spectrum of the first type of precipitates after reheating the Nb-Ti microalloyed steel at 1300℃ for 20 min.

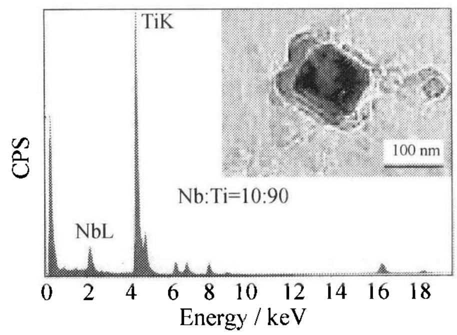

Figure 4 TEM image and corresponding EDX spectrum of the first type of precipitates after reheating the Nb-Ti microalloyed steel at 1300℃ for 2 h.

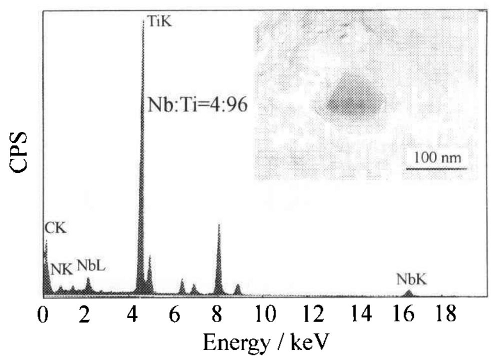

Figure 5 TEM image and EDX spectrum of precipitates after reheating the Nb-Ti microalloyed steel at 1300℃ for 48 h.

The second type precipitates vanish completely and the size of the first type precipitates decrease to below 100 nm in the Nb microalloyed steel held at 1300℃ for 20 min, which can be seen in **figure 6**. When the Nb microalloyed steel is reheated for 2 h, the density of Nb(C,N) drops dramatically and it is difficult to find out the precipitates larger than 70 nm.When reheated for 4 h, no precipitates could be detected in Nb microalloyed steel.

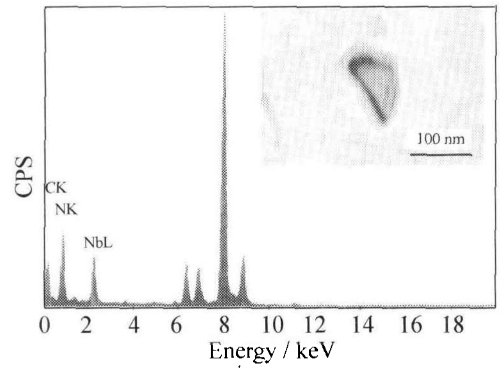

Figure 6 The first-type precipitates in Nb microalloyed steel reheated at 1300℃ for 20 min.

3.3 Discussion

By observing on the dissolving process of precipitates, their precipitation behavior can be derived. According to the reference [7], TiN particles form during solidification of steels and if the further precipitation after solidification happen, it will be NbC forming around TiN. However, in present investigation, the electron diffraction of larger precipitate in Nb-Ti microalloyed steel (shown in **figure 7**) indicates that it is mono-phase particle containing Nb and Ti. On the other hand, If NbC precipitates around TiN , due to its poor thermo-stability compared to TiN, it would dissolve away quickly at 1300℃ and only a TiN core could be remained. However, the experimental results mentioned above do not support the assumption.

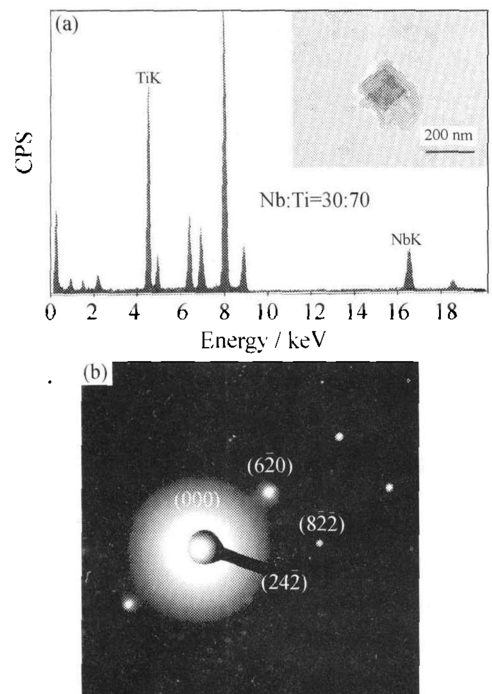

Figure 7 TEM image, corresponding EDX spectra (a) and electron diffraction pattern (b) of the first-type precipitates in Nb-Ti microalloyed steels.

In terms of present investigation, it is reasonable to assume that (Ti,Nb)(C,N), rather than TiN, precipitates during solidification of Nb-Ti microalloyed steel. Due to stronger bond between Ti and N atoms, the precipitates are Ti-rich in initial stage, but they gradually turn to Nb-rich during their growth because of the higher concentration of Nb in the steel.

The other phenomenon deserves to pay attention is that, though the Nb(C,N) particles in Nb microalloyed steels have dissolved away before holding for 4 h at 1300℃, precipitates in Nb-Ti microalloyed steel remain and still contain some Nb atoms even after holding for 48 h at the same temperature. The strong interaction between Nb and Ti, which no sufficient emphasis was put on by previous studies, may be explained for above mentioned phenomena.

4 Conclusions

The following phenomena were observed in this investigation due to strong interaction between Nb and Ti.

(1) In Nb-Ti microalloyed steel, precipitates formed during solidification are (Ti,Nb)(C,N) rather than TiN.

(2) With addition of Ti to Nb-containing steel, Nb will exhibit a far stronger tendency to exist as precipitates at elevated temperature.

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