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Nature of large (Ti, Nb)(C, N) particles precipitated during the solidification of Ti, Nb HSLA steel

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Abstract: To investigate the microsegregation phenomena and complex (Ti, Nb)(C, N) precipitation behavior during continuous casting, a unidirectional solidification unit was employed to simulate the solidification process. The samples of Ti, Nb-addition steels after unidirectional solidification were examined using field emission scanning electron microscope (FE-SEM) and electron probe X-ray microanalyzer (EPMA). In such specimens, dendrite structure and mushy zone can be detected along the solidification direction. It shows that the addition of titanium, niobium to high-strength low-alloyed (HSLA) steel results in undesirable (Ti, Nb)(C, N) precipitation because of microsegregation. The effect of cooling rate on (Ti, Nb)(C, N) precipitation was investigated. The composition of large precipitates was determined using FE-SEM with EDS. Large (Ti, Nb)(C, N) precipitates could be divided into three kinds according to the composition and morphology. With the cooling rate increasing, Ti-rich (Ti, Nb)(C, N) precipitates are transformed to Nb-rich (Ti, Nb)(C, N) precipitates.

Key words: carbonitride; microsegregation; dendritic region; unidirectional solidification; cooling rate

1. Introduction

Microalloying elements in steels, such as Ti, Nb, and V, can facilitate grain refinement, and contribute to dispersion hardening through the precipitation in austenite, and through the precipitation in ferrite during or after $\gamma \rightarrow \alpha$ transformation. Especially, titanium is usually added to high strength low alloy (HSLA) steels because TiN precipitates with a suitable size can suppress austenite grain coarsening in following high temperature processes such as welding or reheating, thereby improving the toughness of final steel products. Multi-microalloying can, thus, lead to the formation of complex compounds, which can influence the mechanical properties of the HSLA steels [1-10].

However it was shown that very large precipitates can be found in the titanium-containing HSLA steels produced by continuous casting process [11-13] and it is unlikely that these large precipitates could play any useful role in refining grain size. Instead they affect the distribution and formation of smaller precipitates and thus the efficiency of titanium and niobium as grain size refiners. In previous researches, the nano size (Ti, Nb)(C, N) precipitates formed during the thermomechanical process were paid much attention. But large (Ti, Nb)(C, N) precipitates were also observed. So the aim of this study is to identify the precipitation of large (Ti, Nb)(C, N) particles in the continuously cast slab of Ti, Nb-addition HSLA steel.

2. Experimental process

2.1. Unidirectional solidification unit

To simulate the inclusion formation during solidification, unidirectional solidification unit was employed. Fig. 1 is the schematic of the unidirectional solidification unit. There are three main parts: the induction furnace, gear set, and water tank. The induction furnace is equipped with a 20 kW-15 kHz generator. The copper end cap is placed at the top part of the induction furnace. Mg chips are placed at the low part of the induction furnace. During operation, He gas is used as the protector. Hot zone is formed inside the coil. The gear set consists of gears and motor which can control the moving direction and the speed of specimens. The water tank is used to quench the specimens after trial.

2.2. Sample preparation

The composition of Ti, Nb-steels used is given in Table 1. These specimens were taken from industrial thermomechanical control process (TMCP) steels. The niobium and titanium contents of B steel are about double those of A steel.

The samples of ϕ4 mm×125 mm were cut from the

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billets using the wire-cut electro discharge machining (WEDM) method. During one experiment, two samples with the same composition were sealed in a ϕ6.2 mm×240 mm (inner radius: 4.2 mm) alumina tube as shown in Fig. 2. The samples were melted and cooled at speeds of 20, 60, and 120°C/mm respectively in He atmosphere during the descent. When a part of specimen was still molten, the specimen was quenched in cold water immediately. The mushy zone including dendrites and melt were presented for investigation.

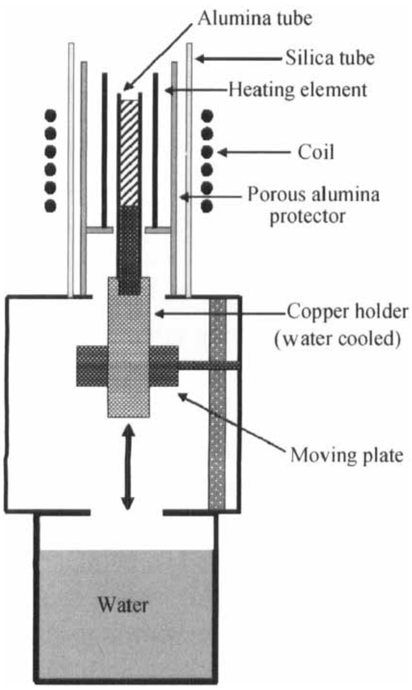

Fig. 1. Schematic diagram of the unidirectional solidification unit.

2.3. Thermal condition

B-type thermocouple was placed at the center to measure the thermal condition along vertical direction in the induction furnace. The temperature at different positions from bottom to top of the coil was measured. During the measurement, different moving speeds of the thermocouple were employed to investigate the effect of specimen movement. The results show that the temperature gradient is also constant, about 10°C/min at different moving speeds as shown in Fig. 3. So different cooling rates can be achieved with different moving speeds following the calculation equation as $R_C(^\circ\mathrm{C/min}) = S_M(\mathrm{mm/min}) \times T_g(^\circ\mathrm{C/mm})$, where R_c is the cooling rate, S_m the moving speed, and T_g the temperature gradient. Table 2 shows the measured results of thermal condition.

2.4. Experimental process

When the temperature in the induction furnace reached 1600°C, the sample on the copper holder was moved into the hot zone. To ensure the specimen melting, the sample was held in hot zone for 15 min. Then the sample was moved down from the hot zone at the scheming speed (2, 6, and 12 mm/min). After moving down for 153 mm, the sample was quenched by dropping into water.

The microstructure and (Ti, Nb)(C, N) particles in the mushy zone of the sample were analyzed by following steps with different methods.

2.5. Analysis

For microscopic observation, the samples were cut, ground, and polished. For resolution observation of inclusions, SEM examinations were carried out using a scanning electron microscope (Jeol™ JSM-6330F) with a field emission gun (FE-SEM), together with an Inca™ analytical system. The composition of the selected inclusions had been measured by energy dispersive X-ray spectra (EDS) analysis with full atomic number absorption fluorescence (ZAF) corrections, using a scanning electron microscope. More than 30 inclusions were examined in each specimen at an acceleration voltage of 15 kV. During measurement, area mapping around each inclusion was employed to determine the average composition of an inclusion.

Table 1. Chemical composition of prepared steel samples wt%

Sample	C	Mn	Si	S-Al	Nb	Ti	P	S	N	O	Cu, Ni, V
A	0.078	1.48	0.314	0.021	0.011	0.005	0.01	0.002	0.003	—	—
B	0.0866	1.278	0.251	0.031	0.018	0.011	0.124	0.0024	0.004	0.0009	—

Fig. 2. Steel samples with/without alumina tube before unidirectional solidification experiment.

To investigate the element distribution in the mushy zone and the region among inclusions, electron probe X-ray microanalysis (EPMA) was carried out by JEOL 8100-JXA EPMA in wavelength dispersive X-ray spectrometer (WDS) mode. During the analysis on element distribution at the mushy zone, an acceleration voltage of 20 kV was employed. Although, the inclusions in interdendritic zone were analyzed with an acceleration voltage of 15 kV.