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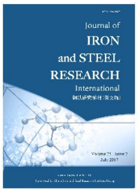

Precipitation behavior of titanium nitride on a primary inclusion particle during solidification of bearing steel

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ABSTRACT

Titanium nitride precipitation on a primary inclusion particle during solidification of bearing steel has been tracked by varying temperature in a confocal scanning violet laser microscope. Upon precipitation, an obvious growth of titanium nitride on a primary inclusion particle was observed due to the rapid solute diffusion in liquid steel. The onset of titanium nitride precipitation did not change with primary inclusion particle size, but the time of growth was greater for a smaller primary inclusion particle. Meanwhile, the particle size displayed little influence on the total precipitated amount of titanium nitride on it under the same conditions. At the later period of solidification, almost no change occurred in inclusion size, but the inclusion shape varied from circle to almost square in two-dimension, or cubic in three-dimension, to attain the equilibrium with steel.

1. Introduction

It is well-known that microcracks nucleate easily between steel and titanium nitrides, which deteriorates steel mechanical properties. Titanium nitrides are usually a component of complex, multi-phase inclusions. During solidification of steel, titanium and nitrogen will enrich at the solidification front because they have different solubilities in liquid and solid phases. When the actual concentration product exceeds the equilibrium value in liquid steel, heterogeneous nucleation and growth of titanium nitride on primary inclusion particles, such as magnesium aluminate spinel in bearing steels[1], will occur. The nucleation barrier is relatively low for spinel inclusions because there is little lattice mismatch between these two phases[2].

In studies of complex inclusions with titanium nitride at the periphery, Suito et al. focused on the number, the size, and the morphology of complex inclusions under different contents of solutes, different holding time at some temperature, and various kinds of primary oxide inclusion particles in alloy[3-5] and stainless steels[6,7]. These results give much guidance for selecting deoxidant and controlling solute content, etc. Studies in IF steel[8] have shown that the smaller the primary oxide inclusion particle, the larger the extent of titanium nitride growth degree. The present authors found a similar result in bearing steel[9] and quantified effects of cooling rate and the initial content of solute carbon in steel on the growth of complex inclusion with a combined thermodynamic and kinetic model. However, all above analyses are based on titanium nitride in solidified steel samples. The behavior of titanium nitride at steelmaking temperatures and its formation on primary inclusion particles have not been systematically studied.

In this paper, the confocal scanning violet laser microscope (CSLM) was employed to observe in real time the growth of titanium nitride, especially growth phase, time and morphology, on two different sizes of primary inclusion particles and the results were analyzed.

2. Experimental Procedure

A steel specimen with chemical composition (wt. %) of C 1.0, Si 0.57, Mn 1.03, Cr 1.46, Al 0.039, Ti 0.0048, N 0.0062, O 0.0009, and Fe balance was machined into a disc (7.8 mm in diameter and 2.5 mm

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in height), carefully polished into a plane, and set in an alumina crucible (8.0 mm in inner diameter and 3.0 mm in height). The gap between the steel specimen and crucible, as shown in Fig. 1, was small to avoid the effect of declining liquid level on the focus during the steel remelting. In the confocal microscope, laser light (408 nm in wavelength) was focused onto steel specimen surface via an objective lens. The reflected beam was focused through a beam splitter onto the photo detector, building up an image for subsequent display. By passing a device that got rid of water and oxygen, a protective argon gas atmosphere (the content of oxygen was lower than 10^{-9}) was used in this experiment to prevent the specimen surface from reoxidation. The deviation of temperature due to thermocouple placement in chamber was around 10 K, and the thermocouple readings have been used here.

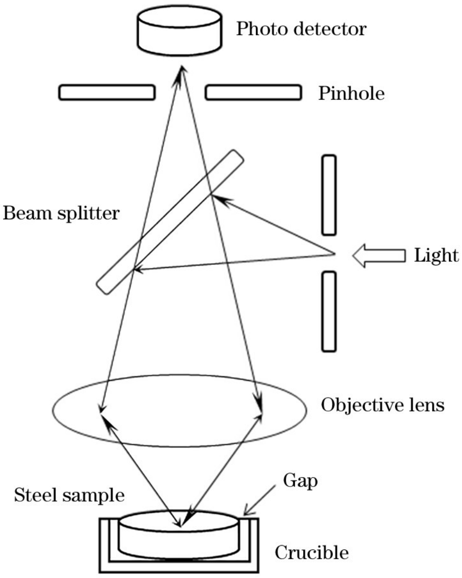

Fig. 1. Confocal nature of optics.

Real time observation of the growth characteristics of titanium nitride on a primary inclusion particle in steel is challenging. The procedure used here was based on the authors' previous work studying dissolution of titanium nitride[10], and was carried out in the following steps.

(1) The scanning electron microscope with energy dispersive spectrometry (SEM-EDS) was first employed to select and mark the location of a relatively large titanium nitride particle near the center of a polished steel specimen. A grid was applied to the sample with a scoring tool to track the position of inclusion in the SEM and the CSLM. The selected titanium nitride particle served as a primary inclusion on which precipitation and growth of new titanium nitride was observed.

(2) The exposure to high temperature was performed in two parts as shown in Fig. 2. In Part one, the steel specimen was heated to 1663 K (1390 ℃) and in this process part titanium nitride dissolved into the steel. The sample was held for some time to let titanium and nitrogen around the inclusion diffuse into the steel. After that, the temperature was decreased to the solidus temperature of this steel, 1583 K (1310 ℃), as calculated according to $T_S = 1811 - \sum \Delta t_i x_i$, where Δt_i and x_i represent respectively the temperature coefficient and mass percentage of solute i in steel[11] (using the same method the liquidus of this steel was 1733 K (1460 ℃)). A cooling rate of 0.15 K/s was employed and the growth of new titanium nitride on the remaining primary titanium nitride particle was observed and measured.

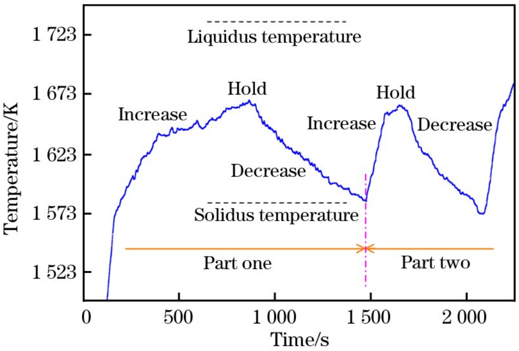

Fig. 2. Temperature change during experiment.

(3) In Part two of this experiment, the sample was reheated and the titanium nitride particle again dissolved. In this part the sample was held until the titanium nitride particle was smaller than it was in Part one of this experiment. The temperature was then decreased with the same rate as that in Part one also to 1583 K (1310 ℃) and the inclusion growth was again observed and measured. Inclusion sizes were measured from CSLM images with the software Image-Pro Plus.

In this experiment, the remaining titanium nitride particles at the end of the holding time in Part one and Part two processes were considered to act as primary inclusion particles in steel, on which new titanium nitride would precipitate upon cooling. Using titanium nitride as a primary inclusion particle means there was zero disregistry with the precipitating titanium nitride. Temperature variations were used to create different sized primary inclusion particles so that the growth of titanium nitride during cooling could be studied. At the holding temperatures, homogeneous precipitation of titanium nitride in this bearing steel was not possible based on previous studies[12,13]. In other words, all titanium and nitrogen in liquid steel were consumed to form the titanium nitride precipitating on primary inclusion particles.

Moreover, the three-dimensional morphology of