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Investigation on the formation mechanism of Ti-bearing non-metallic inclusions in Fe-Al-Ti-O-N alloy by inductive separation method

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A B S T R A C T

Formation mechanism of Ti-bearing non-metallic inclusions was studied by inductive separation method, and characterization method was selected according to the type of each inclusion. The Al + Ti oxide was heterogeneous consisting of Al_2O_3 with Ti rich oxide layer. Compositional and size distribution was characterized by automated SEM observation and EDS analysis. It is formed as the reduction of Al_2O_3 by Ti in molten alloy at 1873 K. The Al-Ti oxide was homogenous. It was liquid at 1873 K characterized by EBSD. The Al/Ti molar ratio of oxide was consistent with that in each alloy as analyzed by manual SEM/EDS analysis. Its formation mechanism is proposed to be the reaction among dissolved Ti, Al, and O in molten alloy at 1873 K. TiN precipitated during solidification. The fraction of TiN smaller than 1 μm was larger in alloy2 than that in alloy1 as analyzed by automated SEM/EDS analysis. It is resulted from larger supersaturation degree in alloy2 during solidification.

1. Introduction

Ti-containing steel is widely used in the field of automobile sheets, heavy plates, and pipeline. The strength and toughness of steel is enhanced through grain refinement by induction of intragranular acicular ferrites (IAF) during solidification by Ti-bearing oxide inclusion or by pinning effect of TiN inclusion during reheating process. Titanium is added to steel after Al dexodiation to improve the yield of Ti in steel production process. However, clogging of submerged entry nozzle (SEN) frequently occurs during production of Ti-containing steel, which is ascribed to the formation of Al and Ti complex oxide inclusion as reported by Basu et al. [1]

Thermodynamics of Fe-Ti-Al-O system has been studied intensively. Nevertheless, discrepancies in thermodynamic description of the Fe-Al-Ti-O system in molten iron still remain, especially on the argument of the existence of liquid oxide phase as discussed by Ruby-Meyer et al. [1] or Jung et al. [2,3] The detail of previous studies have been summarized in our previous paper [4]. Very recently, Kang and Lee reassessed the phase stability of this system and the new oxide stability diagram was reported [5].

Transient evolution of Ti-bearing oxide inclusion in liquid iron has been intensively studied by various researchers [6–10]. It was found that the final state of inclusion significantly depends on the Ti sources, the order of Al and Ti addition, and the Al to Ti ratio. Wang et al. [10] pointed out the necessity to consider the formation of nonstoichiometric liquid Al-Ti oxide phase and the experimental data to prove its existence. Sampling method was applied in previous researches to observe the formation and evolution of Ti-bearing oxide inclusions in liquid iron. However, Ti forms various type of oxides and thus the formation mechanism of a certain type of Ti-bearing oxide inclusion is still unclear because the information on its formation sequence and characteristics are mixed with other types of Ti-bearing oxide by using sampling method. Difference in sampling location, sampling time, and sampler type renders the analysis results between heats hardly to compare. Moreover, manual SEM-EDS analysis is unable to give statistically sufficient information on compositional and size distribution of heterogeneous inclusion. Appropriate characterization method should be employed considering each type of Ti-bearing oxide phase.

Formation of TiN inclusion in liquid steel and during solidification has been studied intensively [11–17]. However, most previous researches concerned the formation of TiN in industrial steels containing considerable amount of alloying elements such as Cr and Si, which can significantly affect the formation behavior of TiN. In addition, oxides were found to act as nuclei for the heterogeneous nucleation of TiN [15–17]. Recently, automated SEM-EDS analysis was used to characterize size distribution of inclusions from statistical perspective. However, it fails to distinguish TiN and TiN with oxide core as reported by Michelic et al. [17] In order to deliver accurate formation behavior of TiN such as size distribution while utilize the advantage of

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automated SEM-EDS analysis in statistics, oxide and TiN inclusions should be separated before characterization.

Wang et al. [18] mentioned high frequency electromagnetic field is efficient to remove oxide inclusions from molten steel. Inductive separation method employed in the present research denotes the separation of preformed oxide inclusions from molten steel by the inductive force generated by high frequency induction furnace. There has not been any study on the clarification of inclusion formation mechanism in multicomponent system such as Fe-Al-Ti alloy using this method.

The current study proposes a novel method to clarify the formation mechanism of Ti-bearing oxide and TiN inclusions in Fe-Al-Ti-O-N alloy system. It contains two parts: separation of different type of inclusion by the inductive separation method and adoption of different characterization method including optical microscopy, manual and automated SEM/EDS analysis, and EBSD analysis according to the type of each inclusion.

2. Experimental and Characterization Method

2.1. Preparation of Alloys

Composition of alloys made by an induction furnace (200 kHz, maximum 5 kW) is shown in Table 1. The experimental setup is shown in Fig. 1. Around 120 g electrolytic iron was melted in an MgO crucible (OD. 40 mm, ID. 28 mm, H. 100 mm) under Ar-3%H_2-N_2 atmosphere at 1873 K. Temperature of the melt was measured by a dual wavelength pyrometer (CHINO, IR-CZ) and controlled by tuning the furnace output power manually. The deviation of temperature during experiment was within 3 K. After keeping the melt for 30 min, Al (99.9 mass% purity) and Ti (99.9 mass% purity) were added in sequence with 2 min interval. The melt was further maintained for 3 min before quenching by immersing the crucible into water. The measured cooling rate was approximately 3.7 K/s. Compositions of produced alloys were analyzed by the inert gas fusion impulse infrared absorption spectroscopy for total oxygen content, the inert gas fusion thermal conductivity method for total nitrogen, and ICP-OES for soluble Al and Ti, respectively. Note that the chemical homogeneity of each alloy was carefully checked by analyzing samples cut from three different positions from top to bottom.

Compositions of alloys were plotted on the oxide phase stability diagram equilibrating with molten Fe-Al-Ti-O alloy at 1873 K as shown in Fig. 2 [19]. Calculation of this stability diagram is based on the assumption that Ti_2O_3, Ti_3O_5, and Al_2TiO_5 are the stable Ti-bearing oxide phases. It is seen Al_2O_3 is the stable phase for prepared alloys, while it should be noted that these compositions are also close to the Al_2TiO_5 stable area in which arguments regarding the existence of a liquid phase remain [1,3,7–10].

2.2. Inclusion Characterization Method

A piece of produced alloy including the whole part from the edge near crucible wall to the center was machined from the cylindrical alloy ingot. It was embedded in resin, and polished by SiC abrasive papers and from 6 to 0.25 μm diamond suspensions before inclusion characterization.

Table 1
Composition of alloys.

Alloy	Soluble Al mass %	Soluble Ti mass %	Total O mass %	Total N mass %
1	0.0497	0.0636	0.0020	0.0032
2	0.0123	0.0586	0.0069	0.0110
3	0.0304	0.0529	0.0125	0.0091

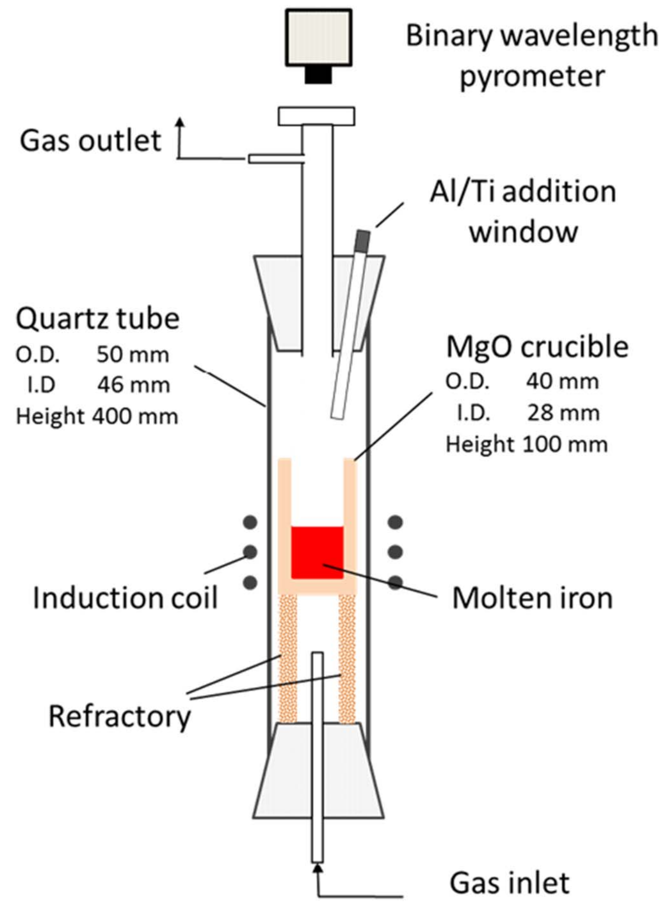

Fig. 1. Experimental setup of induction furnace for preparation of alloys.

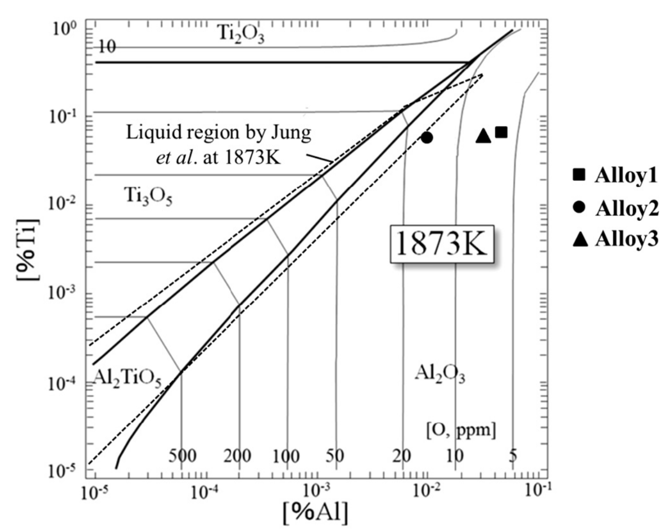

Fig. 2. Alloy compositions on the calculated oxide phase stability diagram in equilibrium with molten Fe-Al-Ti-O alloy at 1873 K [19].

In current research, different type of inclusions was initially separated by inductive force, i.e. inductive separation method. Then, different characterization method was employed according to the type of each inclusion.

An optical digital microscope (KEYENCE, VHX-1000) was employed to deliver overall information on the characteristic morphology and existing area of each inclusion with a magnification from 500 to 5000 times before SEM characterization. Composition and morphology of each type of inclusions were characterized by a field emission scanning