論 文 の 内 容 の 要 旨

論文題目　Compositional and morphological evolution of Ti-bearing inclusions in Fe-based alloy during heating
（Fe基合金の加熱中のTi系介在物の組成および形態変化）

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1. Introduction

Aluminum and titanium deoxidation process is widely adopted in the production of automobile sheets, heavy plates, and pipeline steels. Plenty of researches have been conducted on the behavior of inclusions in molten steel or during the solidification in Al, Ti deoxidized steel. However, the number density, composition or morphology of inclusions may vary during the following reheating and rolling process. Knowledge on the evolution of inclusions during heating in solid steel is still insufficient.

The evolution of Ti bearing-oxide and TiN in Fe-Al-Ti-O-N alloy during heating at 1273K and 1573K were studied respectively in the present research. As an extension, behavior of oxide + TiN inclusion in Fe-16%Cr ferritic alloy during heating at 1573K was investigated.

2. Experimental and characterization method

The composition of four as cast alloys prepared by an induction furnace is shown in Table 1. The procedure was as following: 120 g of electrolytic iron were melted in an MgO crucible under Ar-N2-3%H2 gas atmosphere at 1873 K. Aluminum and titanium were added into the melt with an interval of 2 min and it was further kept for 3 min before quenching into water. A piece of sample was cut from the as cast sample and sealed in a quartz tube (ID: 8 mm, OD: 9 mm, L: 30 mm) with evacuated atmosphere to prevent oxidation during heating. The samples were heated in an electric resistance furnace for 0.5, 1, 3, 6, and 12 h at 1273K and 1573 K. After prescribed time, the sample was taken out, and then quenched in water within 10 seconds. The evolution of oxide in alloy1 and 2 and TiN in all alloys during heating was studied by SEM-EDS with minimum setting size as 0.5μm.

Table 1 Compositions of alloy (mass %)

Alloy	Soluble Al Mass%	Soluble Ti Mass%	Total O Mass%	Total N Mass%	Cooling	Purpose
1	0.0497	0.0636	0.0020	0.0032	Water	Super
2	0.0123	0.0586	0.0069	0.0110	quenching	Saturation
3	0.0139	0.0540	0.0009	0.0023	Air	IF steel
4	0.0316	0.1370	0.0027	0.0035	Air	high Ti

In the outer layer near the crucible wall, oxides were mainly found, while in the inner layer titanium nitrides were mainly found in both as cast and heated alloys. The difference was attributed to the inductive force during the preparation of as cast alloys. Three types of oxide inclusions were found in the outer layer in both as cast and heated alloys: Al_2O_3, Al+Ti oxide denoted as dual phase and Al-Ti oxide denoted as single phase. The different distribution characteristics between Al-Ti oxide and Al+Ti oxide in both as cast and heating samples enables analysis of the same type of oxides after heating. The Al+Ti oxide maintained heterogeneous phase during heating, the change of composition was characterized by Auto SEM (Zeiss)-EDS (Oxford Inca). It is noted the composition is obtained by mapping analysis of the each whole oxide. The homogeneous Al-Ti oxides in cast alloys were found to change to heterogeneous ones during heating and the change of composition and morphology was characterized through Manual SEM (Hitachi S4200)-EDS (Horiba).

3. Evolution of Ti-bearing oxide during heating

Al+Ti oxide with higher Ti concentration tends to have relatively smaller size both in as cast alloys and heated alloys. Most single phase Al-Ti oxide changed to heterogeneous ones consisting of Al rich part and Ti rich part presenting compositional deviation while the morphology varied from spherical to irregular during heating. The change of composition, morphology and size distribution of Al-Ti oxide was studied. The mechanism is considered as the phase transformation of oxide in solid steel during heating.

4. Evolution of TiN during heating

Evolution of TiN was studied in terms of composition, morphology, and Ostwald ripening. The molar ratio of Ti to N of TiN maintained during heating and independent on the compositions of alloys. The range is from 0.7 to 0.8. The morphology of TiN kept rectangular during heating. TiN grew during heating. An estimation model using method of moment is employed to calculate the Ostwald ripening rate. The order of magnitude of Ostwald ripening rate is from 10^{-6} to 10^{-7}. The validity of estimation model is confirmed through agreement of the theoretical Ostwald ripening rate and experimental values in the same alloy heated at 1273 K and 1573 K.

5. Evolution of oxide + TiN during heating

Evolution of Al_2O_3+TiN, (Mg, Ti)Al_2O_4 and (Mg, Ti)Al_2O_4+TiN during heating at 1573K was investigated in Fe-16%Cr ferritic stainless alloy. All of these three types of inclusions were found to be stable during heating. The addition of Mg refines the size of inclusions, and this effect maintained during heating. It indicates the addition of Mg can refine oxide + TiN in as cast and this effect maintained during heating.

6. Conclusions and suggestions for future work

The evolution of Ti-bearing oxide and TiN in Fe-Al-Ti-O-N alloy during heating at 1273K and 1573K were studied. The dual phase Al+Ti oxide was found to be stable regarding composition and size distribution characteristics during heating. Most homogenous Al-Ti oxide changed to heterogeneous ones consisting of Al rich part and Ti rich part presenting compositional deviation while the morphology varied from spherical to irregular during heating. The mechanism is considered as the phase transformation of oxide in solid steel during heating.

The coarsening of TiN occurs during heating. The morphology of TiN kept rectangular during heating. TiN grew during heating. An estimation model using method of moment is employed to calculate the Ostwald ripening rate. The order of magnitude of Ostwald ripening rate is from 10^{-6} to 10^{-7} (um^3/s). The validity of estimation model is confirmed by agreement of the theoretical Ostwald ripening rate and experimental values in alloy heated at 1273 K and 1573 K.

Furthermore, evolution of oxide + TiN in Fe-16%Cr ferritic alloy during heating at 1573 K is studied. The characteristics of oxide + TiN in Fe-16%Cr alloy were investigated. Inclusions are expressed as Al_2TiO_4+TiN, $Al_2(Mg, Ti)O_4$ and $Al_2(Mg, Ti)O_4$+TiN in as cast alloy Cr1, Cr2 and Cr3 respectively. All of these three types of inclusions were found to be stable during heating. The addition of Mg can refine oxide + TiN in as cast and this effect maintained during heating.

The variation of structure of Ti-based inclusions during heating should be clarified in future research.