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Laboratory Study on Oxide Inclusions in High-Strength Low-Alloyed Steel Refined by Slag with Basicity 2–5

Abstract: Non-metallic inclusions in high-strength low-alloyed steel refined by slag with basicity (B) 2–5 and Al_2O_3 content 20%–30% (in mass percent) were investigated by slag–metal equilibrium experiments in laboratory and thermodynamic calculations. Most inclusions in equilibrium were quasi-spherical $\text{CaO-MgO-Al}_2\text{O}_3\text{-SiO}_2$ system and the sizes were less than 5 μm . The average content ratio, $\text{CaO}/\text{Al}_2\text{O}_3$ and CaO/SiO_2 of the inclusions, increased with slag basicity increasing when Al_2O_3 content in slag was around 25%, $\text{MgO}/\text{Al}_2\text{O}_3$ and $\text{CaO}/\text{Al}_2\text{O}_3$ increased with Al_2O_3 content in slag decreasing when slag basicity was around 3.3. The $\text{MgO}/\text{Al}_2\text{O}_3$ of the inclusions was influenced mainly by Al_2O_3 content in slag and $\text{CaO}/\text{Al}_2\text{O}_3$ was influenced mainly by slag basicity. To make the inclusions out of relative lower melting region ($<1,773\text{ K}$), the values of $\text{CaO}/\text{Al}_2\text{O}_3$, $\text{MgO}/\text{Al}_2\text{O}_3$ and $(\text{CaO} + \text{MgO})/\text{Al}_2\text{O}_3$ of the inclusions should be enhanced by increasing basicity and decreasing Al_2O_3 content in slag properly.

Keywords: HSLA steel, oxide inclusions, refining slag, basicity, Al_2O_3 content

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Introduction

High-strength low-alloyed (HSLA) steel plates are widely used in shipbuilding and offshore platform and are strictly required for strength, low-temperature toughness and ductility properties. HSLA steels are usually Al-killed and refined by slag of high basicity ($B = \% \text{CaO} / \% \text{SiO}_2$, in mass percent) and containing high Al_2O_3 content [1–4]. As a result, inclusions of $\text{MgO-Al}_2\text{O}_3\text{-CaO}$, $\text{CaO-Al}_2\text{O}_3$ and $\text{CaO-CaS-Al}_2\text{O}_3$ system are usually formed including calcium aluminates with low melting temperature, such as $12\text{CaO} \cdot 7\text{Al}_2\text{O}_3$ and $\text{CaO-Al}_2\text{O}_3$ [5, 6]. Inclusions with low melting point can be elongated to stringers during subsequent rolling, which are the main reason that deteriorate the properties of ductility, anti-hydrogen-induced cracking and anti-stress corrosion cracking of the steel plates [7, 8]. The detrimental effect of inclusions becomes more apparent with increasing strength level of HSLA steel.

Wang et al. and Son et al. studied the transformation of inclusions in steel during aluminum deoxidation process as well as the refining process with high basicity and Al_2O_3 -bearing slags [5–7, 9, 10]. During these processes, MgO and CaO in slag and ladle refractory can be reduced by the dissolved aluminum in the molten steel, resulting in the pick-up of [Mg] and [Ca] in the steel to several mass ppm, so that deoxidation products of Al_2O_3 gradually change to inclusions of $\text{MgO-Al}_2\text{O}_3$, $\text{MgO-Al}_2\text{O}_3\text{-CaO}$ or $\text{CaO-Al}_2\text{O}_3$.

Researches on the effect of refining slag on inclusions in equilibrium have been reported. Slags refined with <2 basicity and $<20\%$ Al_2O_3 were reported by Wang et al. and Jiang et al. [11–13]. Chen and Jiang et al. investigated the effect of high basicity and Al_2O_3 content ($B = 8$, $\text{Al}_2\text{O}_3 \geq 35\%$), and studied inclusions in 0.6%C-1.5%Si-0.8%Mn steel refined by slags with basicity 3.5 and containing 25% Al_2O_3 as well as those in 0.4%C-0.25%Si-0.65%Mn-1.1%Cr-0.2Mo steel under slag with basicity 5 and containing 25% Al_2O_3 [14–16]. They reported that inclusions in equilibrium with these slags were mainly $\text{MnO-SiO}_2\text{-Al}_2\text{O}_3\text{-CaO}$ and $\text{CaO-MgO-Al}_2\text{O}_3\text{-SiO}_2$ with low melting point; inclusions in the steel

refined with 3.5 or 5 basicity and 25% Al_2O_3 slag contained more MgO and had relatively higher melting temperature.

Very few researches were reported that studied inclusions in HSLA steel refined by 2–5 basicity slag, and whether calcium aluminate inclusions with low melting point can be effectively lowered by the refining slag with relative lower basicity and Al_2O_3 content was rarely reported. For this reason, the current paper investigates inclusions in HSLA steel refined by the slag with 2–5 basicity and 20%–30% Al_2O_3 using slag–metal equilibrium experiments.

Experiment

A vertical Si-Mo-heated high-temperature furnace was used for the equilibrium experiments between the metal and slag. Two hundred grams steel (0.045 mass% C, 0.19 mass% Si, 1.8 mass% Mn) and 40 g slag were melted together in MgO crucibles at 1,873 K under argon-protective atmosphere. During the experiments, 99.999% purity Ar gas was introduced into the reaction tube at a constant flow rate of 1.5 L/min. The slag was made by mixing reagent-grade CaO, Al_2O_3 , SiO_2 and MgO oxide powder in proportion together. The slag composition before experiments is shown in Table 1. Ninety minutes was needed to reach slag–metal equilibrium under 1,873 K, so that all experiments were performed for 90 minutes under 1,873 K without stirring to float inclusions out and establish equilibrium, and then the crucible was taken out from the furnace and quenched rapidly in water.

Table 1: Original chemical composition of slags, mass%.

Slag No.	B (CaO/SiO ₂)	Al ₂ O ₃	MgO
1	2	25	11
2	3.5	25	7
3	5	25	6
4	3.5	20	6
5	3.5	30	8

The contents of silicon, manganese and phosphorus in steel sample were analyzed by inductively coupled plasma atomic emission spectroscopy (ICP-AES), and carbon and sulfur by infrared absorption method. The contents of CaO, MgO, Al_2O_3 and MnO in slag sample were analyzed by ICP-AES, FeO by titration method and SiO_2 by gravimetric method. Thirty-five points on the cross-

sectioned plane of each steel sample were randomly chosen to detect inclusions by scanning electron microscope and energy-dispersive x-ray spectrometer (EDS) to get statistical information of inclusions including morphology, size and chemical composition.

The effect of slag basicity and Al_2O_3 content in the slag on inclusions was investigated. As shown in Table 1, the basicity levels of 2, 3.5 and 5 with 25% Al_2O_3 were chosen to study the effect of basicity. The Al_2O_3 levels of 20%, 25% and 30% with 3.5 slag basicity were chosen to study the effect of Al_2O_3 content. MgO was added to the slag at a saturated level that was predicted by thermodynamic software Thermo-Calc.

Before equilibrium experiments, pre-equilibrium experiments were performed to decide the time to reach slag–steel equilibrium for the slag with 3.5 basicity, and containing 25% Al_2O_3 and saturated MgO under 1,873 K. In previous investigations [9, 14–16], 90–120 minutes was always considered as the holding time for the equilibrium experiments between steel and top slag. In the present work, three holding times of 90, 90 and 120 minutes were chosen to decide the time for equilibrium and study the repeatability of equilibrium experiments under the current conditions.

Results

Pre-equilibrium experiments

The chemical composition of steel and slag samples of the three heats, with holding times of 90, 90 and 120 minutes, respectively, after equilibrium is shown in Tables 2 and 3, showing similar composition and low sulfur content for the three heats.

Table 2: Chemical composition of steel samples, mass%.

Heat No.	C	Si	Mn	P	S
1	0.044	0.18	1.77	0.0070	0.0004
2	0.041	0.18	1.78	0.0080	0.0003
3	0.040	0.17	1.78	0.0070	0.0003

Table 3: Chemical composition of slag samples, mass%.

Heat No.	CaO	MgO	SiO ₂	Al ₂ O ₃	MnO	FeO	S	%CaO/%SiO ₂
1	49.63	8.74	15.26	24.8	0.19	<0.1	0.015	3.25
2	50.21	8.66	15.39	25	0.18	<0.1	0.016	3.26
3	50.11	9.04	15.31	25.03	0.18	<0.1	0.017	3.27

Figure 1: Composition distribution of inclusions in steel samples of pre-equilibrium experiments. (a) 90 min holding; (b) 90 min holding; (c) 120 min holding.

The EDS detection indicated that inclusions in steel for the three heats mainly contained CaO, Al₂O₃, MgO and approximately 11% SiO₂ as shown in Figure 1. It can be seen that the composition distribution of inclusions of the three heats had little difference.

Since the composition of steel, slag and inclusions for the three heats was approximately identical, the equilibrium can be reached after 90 minutes under the present experimental conditions, and the results showed good repeatability. Thus, the reaction time for the following experiments was set to 90 minutes.

Equilibrium experiments

The chemical composition of steel samples after equilibrium under the five slag conditions is shown in Table 4 and the chemical composition of the slag samples after equilibrium is listed in Table 5. Low contents of MnO and FeO suggest that the reduced atmosphere in the reaction tube was controlled well. For the slag conditions No. 1, 2 and 3 (Table 1), the basicity was 1.93, 3.26 and 4.54, respectively, after equilibrium, and Al₂O₃ content was similarly 24%–26%. For the slag conditions No. 4, 2 and 5 (Table 1), the Al₂O₃ content in the slag was about 21%, 25% and 30%, respectively, after equilibrium, and slag basicity after equilibrium was fixed at approximately 3.3.

The typical morphology and corresponding EDS of inclusions in the steel samples equilibrated with slag No. 1–5 are shown in Figure 2(a)–(e), respectively, indicating spherical shape and <5 μm size. The boundaries of inclusions under slag No. 3 and 4 conditions are less smooth than those under slag No. 1, 2 and 5 conditions.

Table 4: Chemical composition of steel samples, mass%.

Composition	C	Si	Mn	P	S
Concentration	0.041–0.048	0.17–0.19	1.76–1.82	0.0065–0.0082	0.0003–0.0010

Table 5: Chemical composition of slag samples, mass%.

Slag No.	<i>B</i> (CaO/SiO ₂)	Al ₂ O ₃	MgO	MnO	FeO
1	1.93	24.48	13.65	0.34	<0.1
2	3.26	25	8.66	0.18	<0.1
3	4.54	26.12	9.56	0.16	<0.1
4	3.27	21.22	9.76	0.2	<0.1
5	3.31	30.04	10.19	0.17	<0.1

Most inclusions in steel samples after equilibrium were CaO-MgO-Al₂O₃-SiO₂ system, and SiO₂ content in inclusions was relative stable for each slag condition but varied with slag basicity, as shown in Figure 3, where the liquid regions of different temperature calculated using thermodynamic software Factsage are also plotted. For slag No. 1, 2 and 3 which had different basicity but identical Al₂O₃ content, the average SiO₂ content in inclusions in equilibrium was 22%, 11% and 9%, respectively. The dots in Figure 3 represent the composition of separate inclusions and the stars are the average values. The curved regions stand for the liquid zones of different temperature in Celsius degree.

With the decrease of SiO₂ content in CaO-MgO-Al₂O₃-SiO₂ system, the liquid zone (<1,773 K) moved toward the direction of (CaO + MgO)/Al₂O₃ and decreases. The SiO₂ content in inclusions decreased with the rise of slag basicity and (CaO + MgO) content increased. The inclusions were mainly liquid (<1,773 K) when the slag basicity was 1.93 or 3.26, while some inclusions entered higher temperature region (≥1,773 K) when the slag basicity was 4.54.

For slags No. 4, 2 and 5 which had different Al₂O₃ content but identical basicity of 3.3, the average SiO₂

Figure 2: Typical morphology and EDS of inclusions detected in steel samples under slag No. 1–5. (a) Slag No. 1; (b) slag No. 2; (c) slag No. 3; (d) slag No. 4; (e) slag No. 5.

content in inclusions in equilibrium was approximately 11% as shown in Figure 4.

The inclusions under slag No. 4 condition were significantly different from those under slag No. 2 and 5. Most inclusions were liquid ($<1,773$ K) when Al_2O_3 in slag was about 25% or 30%, while many inclusions were solid ($\geq 1,773$ K) when Al_2O_3 was about 21%.

Discussion

Effect of slag basicity on inclusions

The ratios of components' average content in inclusions, $\text{CaO}/\text{Al}_2\text{O}_3$, $\text{MgO}/\text{Al}_2\text{O}_3$ and CaO/SiO_2 , under different

Figure 3: Composition distribution of inclusions under different basicity slag conditions. (a) Slag No. 1 (B : 1.93); (b) slag No. 2 (B : 3.26); (c) slag No. 3 (B : 4.54).

Figure 4: Composition distribution of inclusions under different Al_2O_3 slags. (a) Slag No. 4 (Al_2O_3 : 21.22%); (b) slag No. 2 (Al_2O_3 : 25%); (c) slag No. 5 (Al_2O_3 : 30.04%).

basicity slag conditions are shown in Figure 5. With the rise of slag basicity but identical Al_2O_3 content, the CaO/SiO_2 of inclusions increased sharply with values of 1.39, 3.89 and 5.77, $\text{CaO}/\text{Al}_2\text{O}_3$ increased with values of 0.93, 1.08 and 1.42 and $\text{MgO}/\text{Al}_2\text{O}_3$ showed no trend with values of 0.24, 0.21 and 0.27, respectively. Among the three slag conditions, the inclusions equilibrated with slag No. 3 with basicity 4.54 had the highest value of $\text{CaO}/\text{Al}_2\text{O}_3$ and $\text{MgO}/\text{Al}_2\text{O}_3$.

Wang et al. [9] also investigated the effect of slag basicity on inclusions by slag-metal reactions in laboratory; the steel used in experiments was high-strength alloying steel (0.35C-0.24Si-0.62Mn-1.13Cr-0.23Mo). The average CaO content in inclusions increased and Al_2O_3 content decreased, and the ratio $\text{CaO}/\text{Al}_2\text{O}_3$ increased with increasing $(\text{CaO} + \text{MgO})/\text{SiO}_2$ in slag, while the ratio $\text{MgO}/\text{Al}_2\text{O}_3$ in inclusions showed no trend, as shown in Figure 6.

Figure 5: The relationship between average content ratio of inclusions and slag basicity.

Figure 6: Influences of [Al] and slag basicity on composition of the inclusions [9].

Figure 7: The relationship between average content ratio of inclusions and Al₂O₃ content in slag.

Effect of Al₂O₃ content in slag on inclusions

The ratios CaO/Al₂O₃, MgO/Al₂O₃ and CaO/SiO₂ of inclusions under the slag conditions containing different Al₂O₃ content are shown in Figure 7. With decreasing Al₂O₃ content in slag but identical slag basicity of 3.3, the MgO/Al₂O₃ of inclusions increased greatly with values of 0.15, 0.21 and 0.57, CaO/Al₂O₃ increased a little with values of 1.05, 1.08 and 1.13 and CaO/SiO₂ showed no trend with values of 3.55, 3.89 and 3.21, respectively. Among the three slag conditions, the inclusions equilibrated with slag No. 4 containing about 21% Al₂O₃ had the highest value of CaO/Al₂O₃ and MgO/Al₂O₃.

The effect of Al₂O₃ content in slag on inclusions obtained in the present work agrees with the research of Wang et al. [9]. The inclusions in high-strength alloying steels in equilibrium with slag A (6.5 basicity and 41% Al₂O₃) and slag B (5 basicity and 23% Al₂O₃) were investigated by slag-metal reactions in laboratory. The Al₂O₃ content in inclusions equilibrated with slag A was more than that with slag B, MgO content was less and the ratio MgO/Al₂O₃ in inclusions was lower, as shown in Figure 8.

The relationship between $\log(X_{\text{MgO}}/X_{\text{Al}_2\text{O}_3})$ of inclusions and $\log(a_{\text{MgO}}/a_{\text{Al}_2\text{O}_3})$ of slags at 1,873 K is shown in Figure 9, where X_i is the mole fraction of i in inclusions and a_i is the activity of i in slags calculated using thermodynamic software Thermo-Calc. The calculated conditions were as follows: temperature (T), 1,873 K; pressure (P), 101,325 Pa; B, 3.3; MgO, saturated; Al₂O₃, 21%, 25% and 30%. The $\log(X_{\text{MgO}}/X_{\text{Al}_2\text{O}_3})$ of inclusions exhibited a linear relation to $\log(a_{\text{MgO}}/a_{\text{Al}_2\text{O}_3})$ of slags with the slope close to unity. These phenomena could reveal that the slag/metal/inclusion system investigated in the present work was in thermodynamic equilibrium. The results were identical to that of Jiang et al. [15] and Park et al. [17], as shown in Figure 10.

Figure 8: Effect of slag on composition distribution of the inclusions [9]. (a) Slag A; (b) slag B.

Figure 9: Composition of inclusions, $\log (X_{\text{MgO}}/X_{\text{Al}_2\text{O}_3})$ as a function of $\log (a_{\text{MgO}}/a_{\text{Al}_2\text{O}_3})$ of slag at 1,873 K.

Figure 10: Composition of inclusions, $\log (X_{\text{MgO}}/X_{\text{Al}_2\text{O}_3})$ as a function of $\log (a_{\text{MgO}}/a_{\text{Al}_2\text{O}_3})$ at 1,823 K [17].

The method to increase the melting temperature of inclusions

The liquid zone ($<1,773$ K) moved toward the direction of $(\text{CaO} + \text{MgO})/\text{Al}_2\text{O}_3$ decreasing with SiO_2 content in $\text{CaO-MgO-Al}_2\text{O}_3\text{-SiO}_2$ system decreasing, as shown in Figure 3. For the present work, the SiO_2 content in inclusions was mainly influenced by slag basicity. For example, the basicity of slag No. 1–5 after equilibrium was 1.93, 3.26, 4.54, 3.27 and 3.31, and the average SiO_2 content in inclusions was approximately 22%, 11%, 9%, 11% and 11%, respectively. The $\text{CaO}/\text{Al}_2\text{O}_3$ in inclusions was more than 1 when slag basicity was ≥ 3.26 as shown in Figure 5. For HSLA steel, the slag basicity should not be low to make high efficient desulfurization. So to make the

inclusions out of the liquid region ($<1,773$ K), the inclusions should move toward the direction of $(\text{CaO} + \text{MgO})/\text{Al}_2\text{O}_3$ increasing, that is to say, the ratios $\text{CaO}/\text{Al}_2\text{O}_3$, $\text{MgO}/\text{Al}_2\text{O}_3$ and $(\text{CaO} + \text{MgO})/\text{Al}_2\text{O}_3$ of inclusions should be increased.

The ratios $\text{CaO}/\text{Al}_2\text{O}_3$, $\text{MgO}/\text{Al}_2\text{O}_3$ and $(\text{CaO} + \text{MgO})/\text{Al}_2\text{O}_3$ of inclusions under the five slag conditions are shown in Figure 11. The inclusions in equilibrium with slag No. 4 had the highest $(\text{CaO} + \text{MgO})/\text{Al}_2\text{O}_3$ and $\text{MgO}/\text{Al}_2\text{O}_3$ and the second highest $\text{CaO}/\text{Al}_2\text{O}_3$, then lots of inclusions were solid ($\geq 1,773$ K). The inclusions in equilibrium with slag No. 3 had the second highest $(\text{CaO} + \text{MgO})/\text{Al}_2\text{O}_3$ and $\text{MgO}/\text{Al}_2\text{O}_3$ and the highest $\text{CaO}/\text{Al}_2\text{O}_3$, then part of inclusions were solid ($\geq 1,773$ K). The inclusions in equilibrium with slag No. 12 and 5 had relatively lower $(\text{CaO} + \text{MgO})/\text{Al}_2\text{O}_3$, $\text{MgO}/\text{Al}_2\text{O}_3$ and $\text{CaO}/\text{Al}_2\text{O}_3$, then most inclusions were liquid ($<1,773$ K).

Figure 11: The average content ratio of inclusions under the five slag conditions.

As shown in Figures 5, 7 and 11, the $\text{CaO}/\text{Al}_2\text{O}_3$ of inclusions increased with the rise of slag basicity and the $\text{MgO}/\text{Al}_2\text{O}_3$ increased with Al_2O_3 in slag decreasing, and the $\text{CaO}/\text{Al}_2\text{O}_3$ was mainly influenced by slag basicity and the $\text{MgO}/\text{Al}_2\text{O}_3$ was influenced mainly by Al_2O_3 content in slag. So the values of $\text{CaO}/\text{Al}_2\text{O}_3$, $\text{MgO}/\text{Al}_2\text{O}_3$ and $(\text{CaO} + \text{MgO})/\text{Al}_2\text{O}_3$ of inclusions should be enhanced by increasing basicity and decreasing Al_2O_3 content in slag properly to make the inclusions solid ($\geq 1,773$ K).

Conclusions

Laboratory-scale experiments as well as thermodynamic calculations have been done to investigate the effect of

refining slags (B : 2–5, Al_2O_3 : 20%–30%) on oxide inclusions in HSLA steel. The following conclusions were obtained:

- 1) Oxide inclusions in equilibrium with the slags were mainly quasi-spherical $\text{CaO-MgO-Al}_2\text{O}_3\text{-SiO}_2$ system with size less than 5 μm .
- 2) The average content ratios, $\text{CaO}/\text{Al}_2\text{O}_3$ and CaO/SiO_2 of inclusions, increased with the rise of slag basicity when Al_2O_3 content in slag was around 25%. The $\text{MgO}/\text{Al}_2\text{O}_3$ and $\text{CaO}/\text{Al}_2\text{O}_3$ of inclusions increased with Al_2O_3 content in slag decreasing when basicity was around 3.3. The $\text{MgO}/\text{Al}_2\text{O}_3$ of inclusions was influenced mainly by Al_2O_3 content in slag and the $\text{CaO}/\text{Al}_2\text{O}_3$ was influenced mainly by slag basicity.
- 3) The inclusions under slag No. 3 and 4 conditions had relatively higher values of $(\text{CaO} + \text{MgO})/\text{Al}_2\text{O}_3$, $\text{MgO}/\text{Al}_2\text{O}_3$, and $\text{CaO}/\text{Al}_2\text{O}_3$. As a result, lots of inclusions were solid ($\geq 1,773\text{ K}$). While the inclusions under slag No. 1, 2 and 5 conditions had relatively lower values of $(\text{CaO} + \text{MgO})/\text{Al}_2\text{O}_3$, $\text{MgO}/\text{Al}_2\text{O}_3$ and $\text{CaO}/\text{Al}_2\text{O}_3$, most inclusions were liquid ($< 1,773\text{ K}$).
- 4) To make the inclusions out of relative lower melting region ($< 1,773\text{ K}$), the ratios, $\text{CaO}/\text{Al}_2\text{O}_3$, $\text{MgO}/\text{Al}_2\text{O}_3$ and $(\text{CaO} + \text{MgO})/\text{Al}_2\text{O}_3$ of inclusions, should be enhanced by increasing basicity and decreasing Al_2O_3 content in slag properly.

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