Research Article

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Effect of adding yttrium on precipitation behaviors of inclusions in E690 ultra high strength offshore platform steel

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Abstract: The mechanism of inclusions precipitation of the E690 offshore platform steel, with and without addition of yttrium, was studied using the thermodynamic calculation method. The results show that in the E690 steel in the absence of yttrium, the MnS inclusions were precipitated in the liquid phase at the solidification front. By adding the yttrium, MnS inclusions were replaced by spindle and spherical yttrium-containing oxide and sulfide complex inclusions, and the precipitation sequence of yttrium-containing inclusions in the liquid steel was Y2O3, Y2O2S, and YS. However, Y2S3 inclusions cannot be precipitated in the liquid steel under the experimental conditions. It was also found that Al2O3 inclusions can be formed in the liquid steel with and without addition of yttrium. The thermodynamic calculation results are in accordance with the experimental results.

Keywords: yttrium, E690 offshore platform steel, yttrium-containing inclusion, thermodynamic calculation

1 Introduction

The scientific exploration and exploitation of the ocean resources is now proceeding at a greatly accelerated rate [1]. Consequently, the offshore platform steel must possess high mechanical properties, excellent corrosion resistance, and high heat input welding performance. The generation of nonmetallic inclusions, inevitably occurring after the contact and reaction between the active elements in steel and the refractory materials and slags during the steelmaking and casting processes, reduces the properties of steel, such as strength, toughness, and corrosion resistance [2–4]. Therefore, it is important to control the inclusions as much as possible to avoid the deterioration of steel.

Several investigations have been carried out to control the inclusions in steel, and the main control methods include the elimination and the modification of inclusions. The elimination of inclusions in steel is the use of appropriate technology in the steelmaking process to remove inclusions from the liquid steel, and the main technologies include gas stirring [5–7], electromagnetic purification [8,9], slag washing [10], and filter technique [11]. The methods of inclusions modification include calcium (Ca) treatment [12–15], magnesium (Mg) treatment [16–18], calcium–magnesium treatment [19,20], and so on. Zhang et al. [16,17] reported that Mg has very strong thermodynamic affinities with oxygen and sulfur, and magnesia inclusions present a poor agglomeration character than alumina inclusions. However, Mg treatment can easily cause liquid steel spatter. Ca treatment is commonly used to modify high melting point Al2O3 inclusions into fully or partially liquid calcium aluminate to diminish the clogging of the submerged entry nozzle during the continuous casting process [21]. However, either insufficient or excessive addition of calcium alloys is detrimental because incomplete or superfluous calcium can lead to the formation of solid CaO, CaS, calcium alumina, and other unfavorable inclusions deteriorating the quality of the steel [22,23].

Rare earth (RE) elements have very strong chemical activity because of their unique electronic structures, with a variable valence state of the 4f channel [24]. RE elements have a strong affinity toward O and S and react with these elements to form spindle and spherical RE2O3, RE2S3, and RE2O3S2 inclusions, thus avoiding the anisotropy of
mechanical properties in the final rolled products [25–27]. In recent years, extensive research has been carried out on the influence of the RE elements lanthanum and cerium on the microstructures and mechanical properties of various test steels [28–30]. Yttrium, another reactive element, not only exhibits some similarities with lanthanum and cerium but also displays its own favorable characteristics [31]. Unfortunately, to the best of the authors’ knowledge, few studies have considered yttrium-modified steel, especially in terms of the inclusions modification of steels with addition of different amounts of yttrium.

In this article, the inclusion changes in E690 steel with addition of different yttrium contents were compared. In addition, the mechanism of inclusions precipitation was clarified by thermodynamic calculation.

2 Experiments

The test steels were prepared by vacuum induction melting furnace (VAIR). The pure iron with a chemical composition (wt%) of 0.002C, 0.005Si, 0.02Mn, 0.01Cr, 0.01Ni, and balance Fe was used as the raw material, in which Si, Mn, Cr, Ni, Nb, V, Ti, graphite, and yttrium ferroalloy were subsequently added. The final chemical compositions of test steels are presented in Table 1. Here, the yttrium ferroalloy is composed of 65 wt% Y and 35 wt% Fe in this study. Ingots were forged and cut into 90 mm × 90 mm × 120 mm slabs. After homogenization at 1,200°C for 30 min, 7-pass hot rolling was carried out to obtain a final thickness of 16 mm by using a rolling mill (model 350 Reversing, Shanxi Rolling Equipment Factory, China). Here, the finish rolling temperature at 810°C, which is measured by an infrared thermometer (model IR-HOH, Beijing Duhelichuang Technology Co., Ltd, China). Finally, the thick plates were air cooled to 700°C and water cooled to 430°C at a rate of 25°C s⁻¹ and then air cooled to room temperature.

To clarify the precipitation behaviors of inclusions in the test steels, 10 × 10 × 10 mm³ cubic samples were cut from the steel plates and then ground and polished using 3.5 and 0.5 µm diamond compounds. The types of inclusions and their morphology were extensively analyzed by the scanning electron microscopy (model ProX) and energy-dispersive spectrometry (model ProX). In addition, the mechanism of inclusions precipitation was also studied using thermodynamic calculation.

3 Results and discussion

3.1 Effect of yttrium on the morphology and type of inclusions

Figure 1 shows the morphology and the type of inclusions formed in the test steels with and without addition of yttrium. In the absence of the yttrium, MnS is the dominating inclusion, as shown in Figure 1(a). In addition, Al₂O₃ inclusions can be precipitated in the steel without the addition of yttrium, and MnS inclusions adhere to the surface of the Al₂O₃ inclusions. During the rolling process, the MnS inclusions extend along the rolling direction, resulting in the Al₂O₃ inclusions embedded in the MnS inclusions. By adding the yttrium, the strip-like sulfide inclusions were replaced by yttrium-containing complex inclusions with the size lower than 10 µm, as shown in Figure 1(b–d). Compared with Mn and Fe, yttrium has a stronger affinity toward O and S and is more prone to react with these elements to form yttrium-containing oxide and sulfide. In addition, Al₂O₃ inclusions were found in the test steels with addition of yttrium. This is because of the formation of Al₂O₃ inclusions in steel before the addition of yttrium.

3.2 Thermodynamic calculation of inclusions precipitation in test steels

The formation of inclusions in steel is a complex process, and it is difficult to determine the precipitation condition using 3.5 and 0.5 µm diamond compounds. The types of inclusions and their morphology were extensively analyzed by the scanning electron microscopy (model ProX) and energy-dispersive spectrometry (model ProX). In addition, the mechanism of inclusions precipitation was also studied using thermodynamic calculation.

Table 1: Chemical compositions of test steels with and without addition of yttrium (wt%)

| No. | C    | Si  | Mn  | P   | S    | O   | Al  | Nb  | V   | Ti  | Ni  | Cr  | Cu  | Mo  | Y   |
|-----|------|-----|-----|-----|------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 1   | 0.070| 0.25| 1.0 | 0.0080 | 0.0020 | 0.0028 | 0.029 | 0.030 | 0.030 | 0.012 | 0.60 | 0.70 | 0.30 | 0.35 | 0   |
| 2   | 0.074| 0.28| 1.1 | 0.0080 | 0.0020 | 0.0019 | 0.022 | 0.029 | 0.024 | 0.022 | 0.63 | 0.77 | 0.45 | 0.39 | 0.015 |
| 3   | 0.070| 0.25| 1.0 | 0.0077 | 0.0014 | 0.0015 | 0.031 | 0.027 | 0.021 | 0.019 | 0.78 | 0.60 | 0.41 | 0.32 | 0.036 |
| 4   | 0.077| 0.3 | 1.1 | 0.0070 | 0.0016 | 0.0017 | 0.037 | 0.032 | 0.028 | 0.019 | 0.62 | 0.67 | 0.46 | 0.39 | 0.055 |
Figure 1: Morphology and type of inclusions formed in test steels. (a) No. 1 steel; (b) No. 2 steel; (c) No. 3 steel; and (d) No. 4 steel.
and the process of inclusions by means of experimental methods. Therefore, this article uses the thermodynamic calculation to study the precipitation regularity of inclusions so as to better control the precipitation of inclusions in steel.

The liquidus temperature \( T_L \) and the solidus temperature \( T_S \) of steel can be calculated by equations (1) and (2), respectively [32]. Table 2 presents the liquidus temperature and solidus temperature of test steels with and without addition of yttrium.

\[
T_L = 1809 - 83 \text{wt}\% (C) - 7.8 \text{wt}\% (Si) - 5 \text{wt}\% (Mn) - 32 \text{wt}\% (P) - 31.5 \text{wt}\% (S) - 1.5 \text{wt}\% (Cr) - 2 \text{wt}\% (Mo) - 2 \text{wt}\% (V) - 3.6 \text{wt}\% (Al) - 18 \text{wt}\% (Ti) \tag{1}
\]
\[
T_S = 1809 - 344 \text{wt}\% (C) - 12.3 \text{wt}\% (Si) - 6.8 \text{wt}\% (Mn) - 124.5 \text{wt}\% (P) - 183.5 \text{wt}\% (S) - 1.4 \text{wt}\% (Cr) - 4.1 \text{wt}\% (Al) - 4.3 \text{wt}\% (Ni) \tag{2}
\]

### 3.2.1 Thermodynamic calculation of inclusions precipitation in liquid steel

In No. 1 test steel, namely, the steel without addition of yttrium, MnS, and Al\(_2\)O\(_3\) are the dominating inclusions. By adding the yttrium, because the RE yttrium has a strong affinity towards S and O, and therefore, Y\(_2\)O\(_3\), Y\(_2\)O\(_3\)S, Y\(_2\)S\(_3\), and YS may form in the molten steel. The standard Gibbs free energy change for the formation of these various inclusions in the liquid steel is reported in equations (3)–(8) [33].

\[
[Mn] + [S] = \text{MnS}(s) \quad \Delta G^\theta = -15,21,000 + 536.0T \quad \text{(J mol}^{-1}\text{)} \tag{3}
\]
\[
2[Al] + 3[O] = \text{Al}_2\text{O}_3(s) \quad \Delta G^\theta = -15,21,000 + 536.0T \quad \text{(J mol}^{-1}\text{)} \tag{4}
\]
\[
2[Y] + 3[O] = \text{Y}_2\text{O}_3(s) \quad \Delta G^\theta = -15,21,000 + 536.0T \quad \text{(J mol}^{-1}\text{)} \tag{5}
\]

In the liquid steel, the reaction equation of metal element E reacts with nonmetal element F to generate \( E_F \), can be expressed through equation (9).

\[
x E + y F = E_xF_y \tag{9}
\]

When equation (9) reaches equilibrium, the Gibbs free energy \( \Delta G \) is equal to zero. Therefore, the relation between the standard Gibbs free energy \( \Delta G^\theta \) and the equilibrium constant \( K \) can be expressed as equation (10).

\[
\ln K = - \frac{\Delta G^\theta}{RT} = -C_2 - \frac{C_1}{T}, \tag{10}
\]

where \( R \) is the gas constant, in J mol\(^{-1}\)K\(^{-1}\); \( C_1 \) and \( C_2 \) are constants; and \( T \) is the temperature (K).

According to equation (9), the equilibrium constant can also be described by equation (11).

\[
K = \frac{a_{E_xF_y}^{i}}{a_{E}^{i} \cdot a_{F}^{i}} = \frac{1}{f_{E}^{i} \cdot w(E)^{x} \cdot f_{F}^{i} \cdot w(F)^{y}}, \tag{11}
\]

where \( a_i \) is the activity of component \( i \); \( f_i \) is the activity coefficient of element \( i \); and \( w(i) \) is the mass fraction of component \( i \) (wt%).

Substituting equation (11) into equation (10) can result in equation (12).

\[
x \ln f_{E} + y \ln f_{F} + \ln(w(E)^{x} \cdot w(F)^{y}) = C_2 + \frac{C_1}{T} \tag{12}
\]

Therefore, the equilibrium solubility product of \( E,F_y \) in the liquid steel can be expressed as equation (13).

\[
\ln(w(E)^{x} \cdot w(F)^{y}) = C_2 + \frac{C_1}{T} - x \ln f_{E} - y \ln f_{F} \tag{13}
\]

In the E690 liquid steel, the activity coefficient is related to the temperature and interaction coefficient of various elements in the liquid steel. However, E690 offshore platform steel belongs to high-strength low alloy steels, the mass fraction of each solute element tends to 0, and the mass fraction of the solvent iron approaches 1. The second-order interaction coefficients have little effect on the activity coefficient of solute; therefore, the activity coefficients of the components E and F are calculated directly by using the Wagner model.
characterized by the first-order interaction coefficients \( e_{ij} \). The activity coefficient of each solute element in the liquid steel is calculated by equation (14).

\[
\ln f_i = 2.303 \sum_{j=1}^{n} e_{ij} w[j],
\]

where \( e_{ij} \) is the first-order interaction parameter between \( i \) and \( j \) and \( w[j] \) is the mass percentage of element \( j \).

Table 3 presents the first-order interaction coefficient \( e_{ij} \) of various solute elements in the liquid steel. Substituting the data in Table 3 into equation (14), the activity coefficients of O, S, Y, Al, and Mn in the liquid steel are calculated and presented in Table 4.

Substituting the activity coefficients of O, S, Y, Al, and Mn into equation (13), the logarithm of the equilibrium solubility product of MnS, Al2O3, Y2O3, Y2O2S, Y2S3, and YS can be obtained. Figures 2–7 show the logarithm of the equilibrium solubility product and the actual solubility product as a function of temperature for MnS, Al2O3, Y2O3, Y2O2S, Y2S3, and YS.

During the cooling process of the liquid steel, the solubility of inclusions in steel is gradually reduced. When the actual solubility product of the inclusion generating elements is larger than the equilibrium solubility product, the inclusion can be formed. As shown in Figures 2 and 3, for test steels No.1 to No.4, MnS inclusions cannot be generated in the liquid steel, while Al2O3 inclusions can be precipitated. Therefore, for test steels with addition of yttrium, as yttrium has a strong affinity toward O and S and react with these elements, Y2O3, Y2O2S, Y2S3, and YS may form in the liquid steel. Wang and Du [34] investigated the thermodynamics of deoxidation and desulfurization of yttrium in the liquid steel. Their research results show that when yttrium is added to the liquid steel, it reacts with oxygen first; when the precipitated Y2O3 inclusions are not removed from the liquid steel, S in the liquid steel reacts with the precipitated Y2O3 inclusions to generate Y2O2S inclusions; at very late times, when the oxygen content in the liquid steel is very low, yttrium in the liquid steel will react with sulfur to generate Y2S or YS inclusions. Wang and Du [34] also reported that under the reaction equilibrium conditions, the product of the reaction between yttrium and S is mainly YS. As shown in Figures 4–7, Y2O3, Y2O2S, and YS inclusions can be precipitated and Y2S3 inclusions cannot be formed in the liquid steel with addition of different amounts of yttrium. When the yttrium content in the liquid steel is 0.015 wt%, there are no YS inclusions in the liquid steel. This is because all the yttrium in the liquid steel reacts with O to generate Y2O3 inclusions, and the partial Y2O3 inclusions react with S to form Y2O2S inclusions. However, when the yttrium content in the liquid steel increases up to

| Table 3: First-order interaction coefficients \( e_{ij} \) of various elements in liquid steel |
|---|
| \( e_{ij} \) | C | Si | Mn | P | S | O | Cr | Ni | Mo | Cu | Al | Y |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| O | -0.45 | -0.131 | -0.021 | 0.07 | -0.133 | -0.2 | -0.04 | 0.006 | 0.0035 | -0.013 | -3.9 | -16.3 |
| S | 0.112 | 0.063 | -0.026 | 0.029 | -0.028 | -0.27 | -0.011 | 0 | 0.0027 | -0.0084 | 0.035 | -0.55 |
| Y | -0.22 | — | — | — | -7.34 | -90.7 | — | — | — | — | — | — |
| Al | 0.091 | 0.056 | 0.012 | 0.05 | 0.03 | -6.6 | 0.025 | 0.008 | — | 0.008 | 0.045 | — |
| Mn | -0.07 | 0.39 | 0 | -0.0035 | -0.048 | -0.083 | 0.003 | -0.007 | 0.0045 | — | 0.07 | — |

| Table 4: The activity coefficients of O, S, Y, Al, and Mn in liquid steel |
|---|---|---|---|---|---|
| No. | O | S | Y | Al | Mn |
|---|---|---|---|---|---|
| 1 | 0.5674 | 0.9702 | — | 1.0725 | 1.2421 |
| 2 | 0.3315 | 0.9458 | 0.6260 | 1.0997 | 1.2750 |
| 3 | 0.1452 | 0.9274 | 0.6887 | 1.0935 | 1.2383 |
| 4 | 0.0646 | 0.9059 | 0.6558 | 1.0994 | 1.2999 |

**Figure 2:** Logarithm of the solubility product as a function of temperature for MnS in the liquid steel.
0.036 wt%, the residual yttrium after reacting with O in the liquid steel will react with S to precipitate YS inclusions. The calculated results are in accordance with the experimental results.

### 3.2.2 Thermodynamic calculation of MnS precipitation in solid–liquid dual phase zone

Because there is no MnS inclusions precipitation in the liquid steel, it is necessary to calculate the thermodynamics of MnS inclusions precipitation at the solidification front. When the liquid steel begins to crystallize into δ-ferrite, a solid–liquid dual phase zone is formed. In the solidification process of the liquid steel, the forming elements of MnS inclusions in steel tend to be enriched in the liquid phase at the solidification front, leading to an increase in the actual solubility product [32]. Provided that the forming elements of MnS inclusions in the solid phase are nondiffused, the liquid phase is uniformly mixed, and the solid–liquid interface is in a local equilibrium state. Under these conditions, the contents of S and Mn can be used in the Scheil model, namely, equations (15) and (16).

\[
w(S)_{L} = \frac{w(S)}{[P(K_S - 1) + 1]} \quad (15) \\
w(Mn)_{L} = w(Mn)\cdot (1 - P_{L})^{(K_{Mn}-1)}, \quad (16)
\]

where \(w(S)_{L}\) and \(w(Mn)_{L}\) indicate the mass fractions of S and Mn in the liquid phase at the solidification front,
respectively; \( w(S) \) and \( w(Mn) \) are the mass fractions of S and Mn in the liquid phase when the liquid steel begins to solidify, respectively, in wt\%; \( K_S \) and \( K_{Mn} \) are the equilibrium distribution coefficients of S and Mn, respectively. Zhang et al. [35] reported that the equilibrium distribution coefficients of S and Mn are 0.035 and 0.785, respectively; \( P \) is the solid phase fraction, and its relationship with temperature can be expressed in equation (17).

\[
P = \frac{(T_m - T_S)(T_L - T_f)}{(T_L - T_f)(T_m - T_f)},
\]

where \( T_m \) is the melting point of pure iron, and its value is 1,809 K; \( T_f \) is the temperature at the solidification front (K); and \( T_S \) and \( T_L \) are the solidus temperature and liquidus temperature (K), respectively.

According to equations (15) and (16), the actual solubility product in the liquid phase at the solidification front can be calculated. Zhang et al. [35] and Ueshima et al. [36] reported that the equilibrium solubility product for MnS inclusions in the liquid phase at the solidification front can be calculated by equation (18).

\[
\ln [w(Mn) \cdot w(S)] = -19873.92/T + 10.93 - \ln f_m - \ln f_S
\]

Figure 8 shows the logarithm of the equilibrium solubility product and the actual solubility product as a function of temperature for MnS inclusions in the liquid phase at the solidification front.

The contents of S and Mn in the solid phase at the solidification front can be used in the Scheil model and expressed through equations (19) and (20) [37].

\[
w(S)_S = \frac{K_S \cdot w(S)}{[P(K_S - 1) + 1]}
\]

\[
w(Mn)_S = K_{Mn} \cdot w(Mn) \cdot (1 - P)^{K_{Mn} - 1},
\]

where \( w(S)_S \) and \( w(Mn)_S \) indicate the mass fractions of S and Mn in the solid phase at the solidification front, respectively.

According to equations (19) and (20), the actual solubility product in the solid phase at the solidification front can be calculated. In addition, Zhang et al. [35] and Ueshima et al. [36] reported that the equilibrium solubility product for MnS inclusions in the solid phase at the solidification front can be calculated by equation (21).

\[
\ln [w(Mn) \cdot w(S)] = -21274.24/T + 6.97
\]

Figure 9 shows the logarithm of the equilibrium solubility product and the actual solubility product as a function of temperature for MnS inclusions in the solid phase at the solidification front.
function of the temperature for MnS inclusions in the solid phase at the solidification front.

As shown in Figures 8 and 9, when the test steels with addition of yttrium, no separate MnS inclusions are formed in the solid–liquid dual phase zone. This is because yttrium has a strong affinity toward O and S and reacts with O and S to form Y₂O₃, Y₂O₂S, and YS inclusions. For test steels without addition of yttrium, MnS inclusions can be precipitated in the liquid phase at the solidification front. While the equilibrium solubility product was greater than the actual solubility product, and no MnS inclusions can be precipitated in the solid phase at the solidification front. The calculated results are in accordance with those reported by Zhang et al. [35], who indicated that MnS inclusions precipitated at the end of solidification, rather than in the liquid steel.

### 3.2.3 Thermodynamic calculation of MnS precipitation in solid steel

It can be seen from the iron–carbon phase diagram that when the liquid steel solidifies completely, the liquid phase and ferrite transform into austenite almost at the same time. Because the solubility of MnS inclusions in the austenite phase is very small, the solid phase can be considered to be in accordance with Henry’s laws (1 wt% standard state). The equilibrium solubility product can be calculated through equation (21). Figure 10 shows the logarithm of the equilibrium solubility product and the actual solubility product as a function of temperature for MnS inclusions in solid steel.

As shown in Figure 10, it was found that the actual solubility product is always larger than the equilibrium solubility product. This means that MnS inclusions can be precipitated in solid steel. However, MnS inclusions precipitation is a chemical reaction controlled by S and Mn diffusion in the solid steel because the diffusion rate of S and Mn in the solid steel is very slow. Therefore, the precipitation of MnS inclusions in solid steel is mainly controlled by dynamic conditions.

### 4 Conclusions

1. The MnS inclusions were precipitated in the liquid phase at the solidification front in E690 steel in the absence of yttrium and were replaced by yttrium-containing oxide and sulfide complex inclusions on addition of yttrium.
2. By adding the yttrium, Y₂O₃, Y₂O₂S, and YS inclusions were precipitated in the liquid steel, and the precipitation sequence was Y₂O₃, Y₂O₂S, and YS. While Y₂S₃ inclusions cannot be formed in the liquid steel.
3. In E690 steels with and without addition of yttrium, Al₂O₃ inclusions can be formed in the liquid steel.

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