Evolution of Inclusions in Steelmaking Process of Rare Earth Steels Containing Arsenic with Alumina Crucibles

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Abstract: In order to determine strategies for removing arsenic from rare earth arsenic-containing steels, the evolution of inclusions in the whole steelmaking process with alumina crucibles was investigated. It has been proven that adding lanthanum has a significant effect on both the existing state and content of arsenic in steel. The content of arsenic steeply decreased after adding 0.148% lanthanum by generating La–S–As inclusions. The addition of 0.054% lanthanum did not dramatically affect the content of arsenic. Both 0.148% and 0.054% additions of lanthanum modified the existing Si–Mn–Al–O inclusions, making them first change to La-containing inclusions, and then change back to Si–Mn–Al–O inclusions. During this process, the compositions of inclusions changed from (SiO2–MnO)-rich to Al2O3-rich ones, owing to the reactions between lanthanum and alumina crucibles. The addition of 0.148% lanthanum resulted in a relatively severe reaction with the alumina crucible. This led to the decomposition of a part of the existing La–S–As inclusions and a slight increase in the arsenic content. Therefore, it is noted that choosing an appropriate holding time after adding rare earth elements to molten steel has a significant effect on the arsenic removal and saving the consumption of rare earth elements.

Keywords: rare earth; inclusion; arsenic; refractory; evolution

1. Introduction

The trace elements arsenic, tin, lead, antimony, and bismuth have been considered the five most harmful trace elements to steel products, especially for the high-temperature mechanical properties [1–5]. They come mainly from steel scrap and iron ores containing these elements, and cannot be removed from steel in the mainstream oxidation steelmaking process. This problem is serious in China because of the lack of high-quality iron ore resources; on the contrary, China has a large number of associated iron minerals instead, especially the arsenic iron ores.

There are two possible ways to solve the problems caused by trace elements. The first way is to remove them during the mineral separation, sintering, ironmaking, and steelmaking processes; the second way is to modify their existing state in steel products [6–10]. It should be noted that these processes often have limited removal capabilities; therefore, the second method has excellent development potential. Few elements can react with these trace elements, owing to their weak reactivity, except for rare earth elements (REs). It is the unique shell’s electronic structure that makes REs have strong affinities with trace elements. REs have been used to modify the existing state of
trace elements in steel. Lanthanum and cerium are the most widely-used REs for the application in the steelmaking process because of their abundant reserves and relatively low prices [11–14].

Adding REs to improve the existing state of trace elements involves the following two aspects: on the one hand, REs react with trace elements to generate high-melting compounds, thereby decreasing the degree of segregation and improving the mechanical properties of steel at high temperatures [15,16]; on the other hand, REs first react with inclusions that are already present in steel, and then affect the existing state of trace elements [17]. It has been proven that the additions of lanthanum and cerium can successfully reduce the degree of segregation for the trace element arsenic [17–19]. Reactions between REs and refractories are also an important issue to consider when manufacturing RE-containing steels [20,21].

The aim of this work is first to check the evaporation effect of arsenic before the addition of lanthanum in the liquid steel, and then to study the evolution of arsenic- and lanthanum-containing inclusions during the whole steelmaking process, and to discuss the effect of reactions between lanthanum and alumina crucibles on the formation of arsenic inclusions, as well as the content of arsenic in steel.

2. Materials and Methods

The industrial billets of high carbon steel were used as raw materials, offered by an iron and steel company from Chongqing, China. The billets, with a cross-section size of 150 mm × 150 mm, were forged to rods with a diameter of 65 mm to make the chemical compositions uniform. Table 1 shows the chemical compositions of the obtained rods. The arsenic- and lanthanum-added steels were prepared with an induction furnace under the protection of argon with a purity of 99.999 mass%. About 550 g steel rods without rust were used for each sample. After the raw materials were melted and held under 1600 °C for 10 min, arsenic particles with a purity of 99.9 mass% were added. We used high-purity iron sheets to wrap the arsenic particles, together with about 5 g steel rods. Then, we put them in a quartz tube with one head closed and used magnets to hold them. The arsenic particles were added to molten steel when the magnets were removed. It can be directly observed that the alloy sunk into the molten steel. After another 30 min, lanthanum metal with a size of 0.5 to 2 mm was added in the same way. Eight samples for each steel were taken from the liquid steel with quartz tubes with an inner diameter of 4 mm, followed by water-quenching. The power of the induction furnace was turned off after the final sampling from the liquid steel, which was furnace-cooled to room temperature. Figure 1 shows the process of sample preparation.

| Elements | C   | Si  | Mn  | P   | S   | Al  | O   | As  |
|----------|-----|-----|-----|-----|-----|-----|-----|-----|
| Concentrations | 0.79 | 0.21 | 0.61 | 0.011 | 0.015 | 0.001 | 0.0024 | 0.005 |

![Figure 1](image.png)

**Figure 1.** Details of the sample preparation process.

The mass reduction caused by sampling was accurately calculated by weighing the samples extracted before alloying. Then, the initial additions of arsenic were set to 0.02% (0.025% in content), and the initial lanthanum additions were set to 0.148% and 0.054%, respectively. Lanthanum needs
to consume most of the oxygen and part of the sulfur before reacting with arsenic in steel. According to the ideal stoichiometric ratio, the consumed amount of lanthanum as the form of \( \text{La}_2\text{O}_3 \) and LaS was calculated to be 0.086% in total in this work. Therefore, we chose the La additions of 0.148% (higher than 0.086%) and 0.054% (lower than 0.086%) as a comparison.

Except for the samples extracted directly from the molten steel, we also took samples at the 1/2 height and 1/2 radius of the ingots to measure their chemical compositions. The concentrations of lanthanum, arsenic, and acid-soluble aluminum were measured with an inductively coupled plasma optical emission spectrometer (Optima 8000, Waltham, MA, USA). An optical microscope (OM, Sunny XD30M, Ningbo, Zhejiang, China) and a scanning electron microscope (SEM, JEOL 7800F, Tokyo, Japan) were used to observe the inclusions. The accelerating voltage of SEM was set to 20 keV and their chemical compositions were identified with an energy dispersive spectrometer (EDS, 80 mm² X-Max³ Silicon Drift Detector, Oxford, UK). For the water-quenched samples, we chose at least twenty fields of view and no less than 50 inclusions randomly to analyze the chemical compositions of the inclusions for each sample.

3. Results and Discussion

3.1. Evolution of Inclusions in Molten Steel

The results show that the evolution of the inclusions in water-quenched samples taken from liquid steel mainly involves Si–Mn–Al–La–O inclusions, as shown in Figure 2. Before adding lanthanum, the inclusions in both steels were Si–Mn–Al–O composite inclusions; after adding lanthanum, their compositions changed dramatically.

![Figure 2. Evolution of inclusions in molten steel. (a–e) With the initial La addition of 0.148%; (f–j) with the initial La addition of 0.054%.](image-url)
After adding 0.148% lanthanum into the molten steel for 5 min, almost all the Si–Mn–Al–O inclusions were modified by lanthanum and changed to La-containing oxides. The content of Al₂O₃ in these inclusions increased with the extension of constant temperature time; the content of La₂O₃, however, decreased at the same time. The inclusions finally changed back to mostly Si–Mn–Al–O composite inclusions in 20 min, and the amount of Al₂O₃ was dramatically more than that of the inclusions before adding lanthanum.

The evolution of the inclusions in the 0.054% La-added steel shows the same trend with that of the 0.148% La-added steel, but the effect of 0.054% lanthanum on the evolution was significantly weaker. It is worth noting that if the sampling time is within 5 minutes after the addition of lanthanum, the La₂O₃ content in the inclusions could be higher. However, in order to ensure that the metal particles of lanthanum are fully dissolved and well-distributed in molten steel, shortening the sampling time takes risks. Many researchers set 5 min after the RE addition as the first sampling point [20,22].

3.2. Changes in Concentrations of Lanthanum, Arsenic, and Acid-soluble Aluminum in Steel

The changes in the concentrations of arsenic, lanthanum, and acid-soluble aluminum during the whole steelmaking process are shown in Figure 3. It can be seen that 10 minutes was long enough for the arsenic particles to dissolve into the molten steel completely. The content of the arsenic was relatively stable, with only slight evaporation before adding lanthanum, but it fluctuated significantly after adding 0.148% and 0.054% lanthanum, respectively.

![Figure 3](image)

**Figure 3.** Changes in concentrations of As, La, and acid-soluble Al in steel (Arsenic additions were made at 10 min, La additions at 30 min, and the points at 60 min represent the samples taken from ingots). (a) the 0.148% La-added steel; (b) the 0.054% La-added steel.

The arsenic content was reduced from 0.0151% to 0.0059% in 5 min after adding 0.148% lanthanum to molten steel. This indicates that, within five minutes, the lanthanum reacted with arsenic and As-containing inclusions formed. They floated up to the surface of the molten steel, resulting in a sharp decrease in arsenic content. According to the previous research, we consider these arsenic inclusions as La–S–As and will discuss their formation in Section 3.3 [23].

From 5 to 20 min after the addition of 0.148% lanthanum, the content of the arsenic gradually increased unexpectedly. At the same time, the content of the aluminum gradually increased also. Since there was no other source of aluminum, it can be inferred that the increase in the aluminum content was due to the reactions between lanthanum and alumina crucibles [20,21], as well as the ones between lanthanum and the existing Si–Mn–Al–O composite inclusions. These reactions significantly increased the aluminum content in steel and decreased the activity of lanthanum, leading to the partial decomposition of the La–S–As inclusions and the increase in the arsenic content.

The dynamic conditions of lanthanum-related reactions account for the unexpected increase in arsenic content. Within 5 min after the addition of lanthanum, the lanthanum particles quickly dissolved into the molten steel uniformly and reacted with arsenic and sulfur to form La–S–As inclusions. For this reaction, the reactants lanthanum, sulfur, and arsenic were evenly distributed in molten steel, providing perfect reaction dynamic conditions. The reaction kinetics of dissolved
lanthanum with the alumina crucible, however, was slower compared to the formation of La–S–As, since the former is a solid–liquid reaction, but the latter a liquid–liquid reaction. The formation of La–S–As, therefore, was the dominant lanthanum-related reaction within the first 5 min. After 5 min or earlier, most of the sulfur was consumed, and the lanthanum started to mainly react with the alumina crucibles, giving rise to the increase in the content of aluminum and arsenic and a decrease in the effective concentration of lanthanum acting on arsenic.

The evolution of the inclusions mentioned in Section 3.1 shows that the addition of lanthanum made the inclusions change from (SiO₂–MnO)-rich Si–Mn–Al–O inclusions to La-containing ones, then to Al₂O₃-rich Si–Mn–Al–O ones. The addition of lanthanum made the Si–Mn–Al–O inclusions contain higher Al₂O₃ content that shows the reaction sequence of lanthanum-related reactions. This is in good accordance with the evolution of concentrations of lanthanum, arsenic, and aluminum in the molten steel.

For the 0.054% La-added steel, the arsenic content only slightly decreased, as shown in Figure 3b, indicating that the lanthanum did not significantly react with the arsenic and the crucible. Nevertheless, the addition of lanthanum has the same effect on the evolution of the Si–Mn–Al–O inclusions with that of the 0.148% La-added steel.

It is worth noting that, after solidification, the arsenic content in the ingot was slightly higher than that in the molten steel for the 0.148% La-added steel. The continuing decomposition of La–S–As after sampling at 50 min accounts for this. However, the arsenic content was eventually the highest level through the whole steelmaking process for the 0.054% La-added steel. We will carry out further investigation into this, since there is no mature theory to explain it.

![Figure 4](image-url)

**Figure 4.** Morphologies, distribution, and compositions of inclusions on the profile of the ingot (backscattered electron images). (a) Top surface of the ingot; (b) an enlarged image of La₂O₃S and LaAlO₃ inclusions; (c) side surface of the ingot; (d) an enlarged image of La–S–As inclusions near the top surface; (e) energy dispersive spectrum (EDS) of point C; (f) bottom of the ingot.

### 3.3. Types and Distribution of Inclusions in Ingots

Figure 4 shows the distribution of the inclusions on the profile of the ingot for the 0.148% La-added steel. Large concentrations of the inclusions were found near the top, side, and bottom of the ingot, as shown in Figure 4a,c.f. From outside to inside on the top surface, the inclusions were LaAlO₃ (and La₂O₃), La₂O₃S, and La–S–As together with a few LaS inclusions. The first three distributed at the surface, as shown above the yellow dotted line (Figure 4b). La–S–As and LaS, however, mainly distributed below the yellow dotted line (Figure 4a,d). The most significant difference among the top,
side, and bottom surfaces was the amount of cluster-shaped La–S–As inclusions, most of which distributed near the top surface, owing to their floating up.

Furthermore, La–S–As inclusions distributed unevenly near the ingot surface, but the LaAlO₃ inclusions distributed uniformly at the surface with a thickness of around 100 µm that also showed a significant directionality, growing from the surface to inside, as shown in Figure 5. As the LaAlO₃ inclusions formed at the side and bottom surface, resulting from the reactions between lanthanum and alumina crucibles, they can float up to the top surface of the liquid steel. The ones generated during and after solidification, however, cannot move and stayed at the ingot surface. This is the formation process of the LaAlO₃-containing layers. Except for the inclusions mentioned above, inside the ingots, a few La–As, La–S–As, and LaAsO₃ inclusions were also discovered. We discussed the formation mechanism in the previous work [17]. They do not affect the content of arsenic left inside the ingots.

![Figure 5. Morphologies of the LaAlO₃-containing layer at the side surface of the ingot (secondary electron image).](image)

As for the inclusions mentioned above, LaAlO₃ and La₂O₃ are mainly the products of the reactions of lanthanum with alumina crucibles, La₂O₃S and LaS the products of deoxidization and desulfurization, and La–S–As the product of dearsenification. They are mainly generated in molten steel of a large size. Most of them floated up to the surface of the molten steel or moved to the vicinity of the crucible wall to be captured. Among them, the movement of La–S–As to the surface of the molten steel is the core theory of adding arsenic to remove arsenic. In the industrial production, the surface layers containing these inclusions can be removed by dropping off the surface oxidation layers during hot rolling. As for the La–S–As and La–As inclusions generated during the solidification and cooling process, caused by the decreasing solubility of lanthanum, sulfur, and arsenic, they have relatively small sizes and can improve the existing state of the residual arsenic in steel, thereby reducing its hazards to steel products.

Compared with the inclusions in the 0.148% La-added steel, the types of inclusions turned out to be simpler when 0.054% La was added. The most La-containing inclusions were LaS, as shown in Figure 6. They gathered and distributed on the top surface randomly, except the corner between the top and side surfaces (Figure 6b). The corner was the ideal area for capturing cluster-shaped inclusions. At the side and bottom surface, only a few LaAlO₃ inclusions were discovered (Figure 6d–f), indicating that the 0.054% addition of lanthanum slightly reacted with the alumina crucibles, which was in good accordance with the slight increase in aluminum content after adding lanthanum; only a small amount of La₂O₃ and La₂O₃S were found on the top surface, also.

According to the ideal stoichiometric ratio, the lanthanum content required to consume 0.015% of sulfur in the form of LaS is 0.065%; lanthanum needs to consume most of the oxygen in the steel before it can react with arsenic. The lanthanum addition of 0.054% was too low to react with arsenic. Therefore, no cluster-shaped La–S–As inclusions were discovered in the 0.054% La-added steel.
Figure 6. Morphologies, distribution, and compositions of inclusions on the profile of the ingot with the initial La addition of 0.054% (backscattered electron images). (a) Top surface of ingot; (b) corner between the top and side surfaces of ingot; (c) energy dispersive spectrum (EDS) of point D; (d) bottom of ingot; (e) side surface of ingot; (f) EDS of point E.

Many spotted MnS inclusions were found in the water-quenched La-free samples (before the La addition) and the 0.054% La-added samples; even the time consumed before quenching was only around 3 s. These MnS inclusions were possibly generated during the solidification and cooling process. A small amount of La–As inclusions with a size of less than 1 μm were also found in the 0.148% La-added water-quenched samples. These two kinds of inclusions did not generate in the liquid phase.

3.4. Effect of Reactions between Lanthanum and Alumina Crucibles on the Existing State of Arsenic

Possible reactions that existed before the formation of La–S–As are expressed by Equations (1)–(5) [17,24,25]. Their standard Gibbs free energy changes per mole lanthanum or aluminum are shown in Figure 7. As we can see, LaS was the last to form when the content of soluble oxygen was not low, and Al₂O₃ was hard to form when the content of lanthanum was high. LaAlO₃, LaO, and La₂O₂S were the first three inclusions to form. In this work, we discovered these three inclusions, and LaAlO₃ had the most significant amount. After the existing soluble oxygen was consumed, LaS generated together with La–S–As. Unfortunately, no Gibbs free energy data of La–S–As inclusions were reported.

\[
\begin{align*}
2[\text{La}] + 3[\text{O}] &= \text{La}_2\text{O}_3 & \Delta G^\circ &= -1511520 + 379.2T \\
2[\text{La}] + 2[\text{O}] + [\text{S}] &= \text{La}_2\text{O}_2\text{S} & \Delta G^\circ &= -1425820 + 351.0T \\
[\text{La}] + [\text{S}] &= \text{LaS} & \Delta G^\circ &= -490000 + 171.0T \\
2[\text{Al}] + 3[\text{O}] &= \text{Al}_2\text{O}_3 & \Delta G^\circ &= -1225000 + 393.8T \\
[\text{Al}] + [\text{La}] + 3[\text{O}] &= \text{LaAlO}_3 & \Delta G^\circ &= -1188616 + 310.6T \\
[\text{La}] + [\text{S}] + [\text{As}] &= \text{La-S-As} & \Delta G^\circ &= -36384 - 83.2T \\
\end{align*}
\]


\[ 2[\text{La}] + \text{Al}_2\text{O}_3 = \text{La}_2\text{O}_3 + 2[\text{Al}] \quad \Delta G^\circ = -286520 - 14.6T \quad (8) \]

We consider that the La–S–As inclusions were generated in the form of Equation (6). Because the reactions between lanthanum and alumina crucibles did not involve sulfur and arsenic, the key factor was dissolved lanthanum. Although Equation (6) was one of the dominant reactions within the first 5 min after the addition of lanthanum, the reactions of lanthanum with alumina crucibles became the dominant ones after 5 min or earlier, as illustrated by Equations (7) and (8). REAl_{11}O_{28} inclusions may also generate when RE addition is high [20]. The two steel-refractory reactions have slower kinetics, but they can continuously offer oxygen to react with lanthanum, leading to an increase in the aluminum content in steel and a decrease in the effective concentration of lanthanum acting on arsenic. Thereby, some La–S–As inclusions decomposed, which put some arsenic back into the steel. The profile morphologies and element distribution at the steel-refractory interface of the 0.148% La-added steel ingot are illustrated in Figure 8.

![Figure 7](image1.png)

**Figure 7.** Gibbs free energy of La-related reactions per lanthanum or aluminum in molten steel.

![Figure 8](image2.png)

**Figure 8.** Morphologies and element distribution on the profile of the side surface of the ingot. (a) Backscattered electron image of morphologies.

3.5. Evolution of the Existing State of Arsenic in the Smelting and Solidification Process

The vital issue that we focused on to decrease the harm of arsenic to steel products is its existing state, content, and distribution in ingots. Five steps can be used to illustrate this issue, as shown in Figure 9.
Step 1: After added to molten steel, arsenic metals with a size of around 0.5 to 2 mm dissolved into the molten steel within 10 min.

Step 2: At the same time, the arsenic started to evaporate due to its low partial pressure under the protection of argon gas. The evaporation proportion of arsenic in this work was around 10% within 30 min.

Step 3: After adding lanthanum, the arsenic reacted with the lanthanum and sulfur to form La–S–As inclusions. These inclusions then mostly floated up to the top surface of the molten steel, leading to a steep decrease in the arsenic content.

Step 4: Lanthanum can also react with alumina crucibles if its addition is high, which refers to the 0.148% in this work. These steel-refractory reactions dramatically decreased the activity of lanthanum, leading to the decomposition of some existing La–S–As inclusions and the increase in the arsenic content.

Step 5: When the power of the induction furnace was turned off, the decomposition of La–S–As still existed due to the continuous steel-refractory reactions during the cooling and solidification process.

![Figure 9](image_url)

**Figure 9.** The existing state of arsenic in the steelmaking process and its distribution in the ingot.

In summary, the addition of 0.148% lanthanum significantly decreased the content of arsenic by generating La–S–As inclusions, which floated up to the top surface or moved to the side and bottom surfaces with the flow of the molten steel. At the same time, a serious issue that needs attention is the constant temperature time after adding lanthanum because lanthanum can react with alumina crucibles. This will significantly reduce the removal effect of arsenic.

4. Conclusions

The evolution of inclusions in the whole steelmaking process of rare earth steels containing arsenic with alumina crucibles was investigated, as well as its effect on the existing state of arsenic. The following conclusions were obtained:

1. The content of arsenic steeply decreased after adding 0.148% lanthanum by generating La–S–As inclusions, most of which floated up to the top surface of the molten steel. The addition of 0.054% La did not dramatically affect the content of the arsenic.

2. Both the 0.148% and 0.054% lanthanum additions modified the existing Si–Mn–Al–O inclusions, making them first change to La-containing inclusions, then change back to Si–Mn–Al–O inclusions. During this process, the compositions of the inclusions changed from (SiO₂–MnO)-rich to Al₂O₃-rich ones, owing mainly to the reactions between the lanthanum and alumina crucibles.

3. The addition of 0.148% lanthanum resulted in a relatively severe reaction with the alumina crucible. This led to the decomposition of some existing La–S–As inclusions and a slight increase in
the arsenic content. An appropriate holding time after adding lanthanum had a significant effect on the removal of arsenic and the consumption of rare earth elements.

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