Influence of Al deoxidation on the formation of acicular ferrite in steel containing La

Yu-Min Xie, Ming-Ming Song*, Fang-Fang Wang, Zheng-Liang Xue, Run-Sheng Xu, and Wei Wang*

Abstract: The formation of acicular ferrite (AF) in steel containing Lanthanum (La) with different contents of Aluminum (Al) has been investigated. It has been found that the amount of AF in La treated steel killed by Al is less than the one with little content of Al. The reasons are as follows: first, Al can change the type of La-containing inclusions and may reduce the effectiveness of La inclusion to induce the nucleation of AF. second, Al can make the cooling rate range of AF nucleation narrower in La treated steel. What’s more, Al can increase the decomposition temperature of austenite, and all these make AF formation difficult. In this study, the content of Al should be controlled to less than 0.018 wt% to obtain a more effective inclusion to induce the formation of AF.

Keywords: La, acicular ferrite, inclusion, cooling rate, Al-killed steel

1 Introduction

Microstructure refinement is the only way to strengthen steel with no reduction in toughness [1–3]. Acicular ferrite, formed within an austenite grain on an inclusion, can refine the microstructure dramatically [4,5]. Thanks to its fine chaotic interlocking nature [6–8], which can obstruct the propagation of cracks [9,10], AF can enhance the performance of steel. Researchers [11,12] found that a bit of rare earth (RE) makes a great difference in the microstructure and performance of steel. Wen [12] affirmed that about 0.02 wt% RE could make a large amount of AF formed in 16Mn steel. Our previous work [13,14] compared the content of AF within the steels having different types of RE inclusions and found that the complex of RE oxysulfide and MnS has the largest capacity to induce the formation of AF. As is well known that RE has very great chemically active properties, during the process of steelmaking, deoxidizing must be done before the addition of RE in order to improve the yielding rate and stabilize the treatment function of RE [15]. Al is one of the most common deoxidizing elements. Lee et al. [16] and Huang et al. [17] have found that Al can reduce the formation of AF. They believe that Al can change the composition of effective inclusion into inert inclusion in Ti-killed steel. While both Li et al. [18] and Wu et al. [19] believed that Al can facilitate the formation of AF in Zr- and Ti-containing steel. Until now, Al has great uncertain and complicated influence on the formation of AF. Moreover, almost all researchers focus their interests on Al dioxide function to modify the inclusion. Besides this, Al may also have an alloying effect on the austenite decomposition process. It is a pity that there are few reports on this impact in the field of rare earth-treated steel. Lanthanum (La) is a typical rare earth element. At present, we chose La to research the formation of AF after killing by Al. In this study, several attempts have been made to investigate the influence of Al content on the formation of AF in La treated steel, comprehensively.

2 Experimental procedure

Three steels with different compositions were obtained by a vertical resistance furnace. The furnace was equipped
with a thermocouple of PtRh30–PtRh6 and an automatic temperature controller. During the whole experiment procedure, pure argon was pumped from the bottom to provide a protective atmosphere. Raw materials were melted in a crucible with a size of $\varnothing 45 \times 100$ mm. The melt was held at 1,600°C for 5 min to homogenize composition. After that, the power was turned off. When temperature fell to 1,100°C, the ingots were picked out and water quenched quickly. The chemical compositions of four ingots are listed in Table 1.

Samples with size of $\varnothing 10 \times 10$ mm were sectioned from the middle of the cooled ingots. They were machined into metallographic specimens after being ground and polished. Inclusions' observation in samples was carried out on a scanning electron microscope (SEM) with an energy-dispersive spectrometer (EDS) attachment. Then the specimens were etched by 4 vol% nital to view microstructure. The characteristics of microstructure were studied by the electron backscatter diffraction (EBSD) technique.

The ingots were hot forged into $\varnothing 10$ mm rods. The forging temperature was between 1,200 and 900°C. Then the rods were cooled to room temperature by air. Samples with size of $\varnothing 4 \times 10$ mm were picked up from the rods for dilatometer test by DIL805A thermal dilatometer under vacuum condition. During the dilatometer test, samples were heated up to 1,100°C at a speed of 10°C/s. After maintaining the temperature uniform for 5 min, the samples were then cooled to room temperature at series rates (1, 2, 3, 5, 8, 10, 15, 25, and 30°C/s). The microstructures of cooled samples were detected by optical microscopy (OM).

### 3 Experimental results

#### 3.1 Microstructures

Figure 1 shows the as-cast microstructures of three steels. The most microstructure in steel no. 1 is martensite (M) and in addition a small amount of bainite (B), as shown in Figure 1(a). When about 0.020 wt% La was added, the microstructure in steel no. 2 showed a significant change, as shown in Figure 1(b). The microstructures contain a lot of AF and a little of ferrite side plate (FSP) and pearlite. However, when the content of Al increased to 0.027 wt% in steel no. 3, the microstructure is mainly parallel bundle-like bainite after La was added, as shown in Figure 1(c). The AF amount in steel no. 3 was decreased obviously, compared to that in steel no. 2.

#### 3.2 Microstructure transfer during continuous cooling process

The microstructures of steels cooled by different rates are shown in Figures 2, 3, and 4. In steel no. 1, when the cooling rate is slow, the microstructure is mainly boundary ferrite (GBF) and FSP. When the cooling rate is faster, the B increases. As the cooling rate further increases, the microstructure changes into M. There is little visible AF at each cooling rate. In steel no. 2, it can be seen that AF forms in the range of 1–8°C/s, besides some GBF and FSP. However, for steel no. 3, there is only a little of AF when the cooling rate is 2–5°C/s, and the main microstructures were FSP and GBF. Under the same cooling speed correspondingly, the amount of AF in steel no. 3 is less than that in steel no. 2 significantly when the cooling rate is 2–5°C/s. Compared with the quenching results, it can be roughly estimated that the quenching cooling rate was 3–8°C/s during $\gamma \rightarrow \alpha$ at the sampling position according to microstructure.

SEM with EBSD attachment was used to explore the microstructure features in steel no. 2 and 3. The grain orientation and grain boundary angle in two steels were obtained, as shown in Figure 5. Ferrite laths in steel no. 3 were parallel bundle-like, while in steel no. 2 the ferrite was chaotic, as shown in Figure 5(a and d). The ferrite grain orientation distribution is shown in Figure 5(b and e). The ferrite grain orientation distribution in AF microstructure is pellmell, but that orientation in bainite is more or less the same. The size distributions of the grain boundary angle of ferrite in steel no. 2 and 3 are shown

### Table 1: Chemical compositions of experimental ingots (wt%)

|    | C   | Si  | Mn  | P   | S   | O   | Al  | La  | Fe  |
|----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| No. 1 | 0.17 | 0.22 | 1.31 | 0.018 | 0.025 | 0.0085 | 0.003 | — | Bal. |
| No. 2 | 0.17 | 0.25 | 1.31 | 0.018 | 0.025 | 0.0065 | 0.003 | 0.020 | Bal. |
| No. 3 | 0.18 | 0.26 | 1.32 | 0.015 | 0.022 | 0.0045 | 0.027 | 0.022 | Bal. |

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in Figure 5(c and f). Thick red lines are those grain boundary angles greater than 15°, while those grain boundary angles less than 4° are marked with thin black lines. The grain boundary angles of ferrite laths in FSP are very small (mainly <4°), and these grain boundaries are mostly subgrain boundaries. While for AF, the grain
boundary angles of ferrite are mostly very high. The distributions of grain misorientation angle of ferrite in two kinds of microstructures are shown in Figure 5(g): the grain misorientation of ferrite in steel no. 2 is larger than that in steel no. 3, as the percentage of low grain misorientation of ferrite (<5°) in steel no. 2 is less than that in steel no. 3, and the percentage of high grain misorientation of ferrite (>40°) in steel no. 2 is more than that in steel no. 3. This is why AF can refine the microstructure and improve the performance of steel obviously. So, it is very important to understand the formation of AF.

4 Discussion

From the experimental results, it can be found that AF has a chaotic and fine morphology, Al makes the content of AF to decrease and the formation cooling rate narrower, which indicates that the formation of AF in C–Mn steel with La becomes hard when Al content increased. To make clear the influence of Al on the formation of AF, two factors will be briefly discussed below: Al functions of (i) inclusion modify and (ii) a certain degree of alloying effect.

4.1 Inclusion modify

Figure 6 shows the composition and morphology of inclusions in three as-cast steels. In steel no. 1, the typical compositions of the inclusion are MnS and complex Mn–Si–Al–O, as shown in Figure 6(a and b). Some of MnS can cover around Mn–Si–Al–O. In steel no. 2, after La was added, the composition of the inclusion changed into composite La–Mn–Si–O–S with some MnS attached as shown in Figure 6(c). In steel no. 3, as shown in Figure 6(d and e), the non-metallic inclusion compositions are La–Al–O and La–S, and some La-containing inclusions also have MnS precipitated on the surface.

To further determine the chemical composition of inclusions, FactSage 6.3 thermal software was used to calculate the types of non-metallic inclusions formed in three steels, as shown in Figure 7. In steel no. 1, the primary inclusions are MnS and Mn–Si–Al–O, as shown
in Figure 7(a). Figure 7(b) shows that the main inclusions in steel no. 2 are La$_2$O$_2$S and MnS, besides a bit of Mn–Si–O and LaAlO$_3$ after La was added. In steel no. 3 as the content of Al increased, the inclusions change into MnS and LaAlO$_3$, and a little of La$_2$S$_3$, as shown in Figure 7(c). The compositions of inclusion calculated are consistently well with the EDS results found in three specimens, correspondingly.

In this study, it was found that the main microstructure in steel no. 1 is martensite and a few bainite, which means that Mn–Si–Al–O or MnS has little effectiveness to induce the formation of AF. When La is added into steel no. 2, the inclusions change into La$_2$O$_2$S and MnS, and the microstructure contains a lot of AF. The typical nucleation of AF on the surface of inclusion in steel no. 2 is shown in Figure 8. There are more than five ferrite plates nucleated on this inclusion, which means that the effectiveness of inclusion in steel no. 2 is very high. While in steel no. 3, when La is added into steel after killed by Al, the main inclusions are LaAlO$_3$ and MnS, and a little of La$_2$S$_3$, and the content of AF reduces greatly. It can be inferred that the effectiveness of inclusion in steel no. 3 is less than that in steel no. 2.

Lattice epitaxial matching between ferrite and inclusion can reduce the interfacial energy, which is considered as an important way to cut down the resistance of inclusion to induce the nucleation of AF heterogeneously [20]. The smaller lattice mismatch, the activation energy barrier is lower under the transformation temperature of γ → α (about 912°C). The mismatch between ferrite and inclusion can be calculated using Bramfitt’s theory, as shown in equation (1) [21].

\[
\delta_{(hkl)_s}^{(hkl)_n} = \sum_{i=1}^{3} \left( \frac{|d_{[uvw]_s} - d_{[uvw]_n} \cos \theta|}{3 \times d_{[uvw]_n}} \right) \times 100\%
\]

where $s$ is the substrate, $n$ is the nucleated solid, $(hkl)_s$ is a low index plane of the substrate, $[uvw]_s$ is a low index direction in $(hkl)_s$, $(hkl)_n$ is a low index plane of the nucleated solid, $[uvw]_n$ is a low index direction in $(hkl)_n$, $d_{[uvw]_n}$ is the interatomic spacing along $[uvw]_n$, $d_{[uvw]_n}$ is the interatomic spacing along $[uvw]_n$, and $\theta$ is the angle between the $[uvw]_s$ and $[uvw]_n$.

In conventional arc welding, Thewlis et al. [11] found that effective inclusions for AF nucleation had an exceptionally low misfit with ferrite when the Bain orientation relationship was obeyed, i.e., $\langle 100 \rangle$ planes of ferrite were parallel to $\{100\}$ planes of the inclusion and, within $\{100\}$ planes, $\langle 100 \rangle$ directions in ferrite were...
parallel to \langle 110 \rangle \) directions in the inclusion. In the present work, the lattice misfits between ferrite and inclusion were calculated under two orientation relationships. One is in obeying the Bain orientation relationship and the other is a low index orientation relationship depending on the crystal system where the lattice misfit is the smallest. The calculated results are shown in Table 2. It is evidently that LaAlO\(_3\) or MnS does not have very low lattice misfit with ferrite whatever obey the Bain orientation relationship or not. Nevertheless, the lattice misfit between La\(_2\)O\(_2\)S and ferrite is very low. For an La\(_2\)S\(_3\) particle, the lattice misfit with ferrite is between that of La\(_2\)O\(_2\)S and LaAlO\(_3\). So, La\(_2\)O\(_2\)S has the highest effectiveness to induce the nucleation of AF, La\(_2\)S\(_3\) is the second, and LaAlO\(_3\) and MnS are the lowest. Therefore, the amount of AF in steel no. 2 is the largest. In steel no. 3, the main inclusion is LaAlO\(_3\) and there is only a little of La\(_2\)S\(_3\). The low content of AF in steel no. 3 may have been caused by a lack of effective inclusions. The calculation result agrees consistently well with the experimental results.

According to the lattice epitaxy theory, the most important condition for AF formation is in having a lot of effective inclusion as a nucleus. If there were little effective inclusion, AF would not form. The greater the effectiveness of inclusion to induce the nucleation of AF is, the more the number of that inclusion is, the more the content of AF in steel is. The effectiveness of inclusion to induce the nucleation of AF is determined by the type of inclusion. Since equations (R1) and (R2) react easier than equation (R3), Al can change the inclusion composition after La is added easily [22].

\[
[\text{La}] + [\text{Al}] + 3[\text{O}] = \text{LaAlO}_3(s), \quad (R1)
\]

\[
\Delta_r G^0 = -801616 + 28.9\cdot T \text{ J/mol}
\]

\[
[\text{La}] + \frac{1}{2}\text{Al}_2\text{O}_3(s) + \frac{3}{2}[\text{O}] = \text{LaAlO}_3(s), \quad (R2)
\]

\[
\Delta_r G^0 = -188200 - 166.1\cdot T \text{ J/mol}
\]

\[
[\text{La}] + [\text{O}] + \frac{1}{2}[\text{S}] = \frac{1}{2}\text{La}_2\text{O}_2\text{S}(s), \quad (R3)
\]

\[
\Delta_r G^0 = -712978 + 175.6\cdot T \text{ J/mol}
\]

Besides the type change of effective inclusion, Al may alter the number density and size of inclusions, which may also have a great influence on the formation of AF. In order to well interpret the influence of Al...
content on the number density and size of inclusions in La steel, 60 photographs were taken randomly under ×1,000 magnification using OM in the polished samples of steel no. 2 and 3, counting the inclusion number and size statistically. The result shows that the inclusion number densities are about 203 and 210/mm²,
respectively, in steel no. 2 and 3, and the difference is very small. The size statistical result shows that as the content of Al increases, the mean size of inclusion becomes smaller, as shown in Figure 9. This is caused by the quantity percentage of inclusion size of <1 µm increased, while the quantity percentage of inclusion size of >1 µm decreased. It means that the increase in Al can fine-tune the size of inclusions in La treated steel.

However, it is worth mentioning that the size of effective inclusion, which can nucleate AF, mainly falls in 1–4 µm according to the previous research results [13]. The increase in Al content reduces the number of effective inclusions, which is not conducive to the nucleation of AF either.

4.2 Alloying effect

The cooling process starting from austenite is an important factor considering the formation of AF. In the present study, after austenization for 5 min at 1,100°C, three samples almost have the same austenite grain size, about 140–160 µm. Figure 10 reveals the microstructure evolutions in three samples as the cooling rate increased from 1 to 30°C/s. The shades in the figure mark the cooling rate and temperature range of AF formation. In steel no. 2, the AF formation temperature range is 570–660°C and the cooling rate range is 1–8°C/s, as shown in Figure 10(b). It should be noted that the addition of La has little influence on the
La is extremely low in melt, about 10 wt% according to thermodynamic calculation suggested that the content of La is very weak within the scope of this study. Thermodynamic analysis of the CCT curve, which means that the alloying effect of La is very weak, is presented. Figure 9 shows the effect of Al on the size of inclusions in steels.

### Table 2: Lattice misfit values between particle constituent phases and ferrite

| Compound   | Crystal system | Lattice parameter, nm | Planar parallelism            | $a/a_{\alpha,Fe}$ | Misfit, % |
|------------|----------------|-----------------------|-------------------------------|-------------------|----------|
| LaAlO$_3$  | Cubic          | $a = 0.3807$          | (100)$_{a}$/[100]$_{inclusion}$, (100)$_{a}$/[100]$_{inclusion}$ | 1.328             | 32.8$^\dagger$ |
| La$_2$S$_3$ | Cubic          | $a = 0.8616$          | (111)$_{a}$/[111]$_{inclusion}$, (110)$_{a}$/[110]$_{inclusion}$ | 3.006             | 0.2$^\dagger$ |
| La$_2$O$_2$S | HCP            | $a = 0.40509, c = 0.6943$ | (100)$_{a}$/[100]$_{inclusion}$, [110]$_a$/[110]$_{inclusion}$ | —                | 6.3$^\dagger$ |
| MnS        | Cubic          | $a = 0.523$           | (111)$_{a}$/[100]$_{inclusion}$, (100)$_{a}$/[100]$_{inclusion}$ | 1.825             | 8.8$^\dagger$ |

$^*$Lattice parameter of $\alpha$-Fe $a = 0.28665$ nm. $^\dagger$Low index orientation relationship between ferrite and inclusion. $^\ddagger$Bain orientation relationship between ferrite and inclusion.

#### 4.3 The restriction of Al content

Based on the above analysis, it can be found that Al has a great influence on the formation of effective inclusion (type, size, and quantity) and the formation of AF during cooling process in C–Mn steel treated by La. However, more work is still needed to distinguish which one of these two factors is the main aspect. In view of obtaining AF in steel, Al content should be controlled in a suitable range strictly [23,24]. Given the formation of effective inclusion, thermodynamic calculation was used to explore the restriction of Al content at 1,600°C, as shown in Figure 11. It can be seen that Al content has a great effect on the types of La-containing inclusions in molten steel at 1,600°C. When the content of Al in the steel is less than 0.0015 wt%, the inclusions are La$_2$O$_3$, LaAlO$_3$, and La$_2$O$_2$S. As the Al content increased to 0.0015–0.018 wt%, the inclusions changed into LaAlO$_3$ and La$_2$O$_2$S. When Al content augmented to more than 0.018 wt%, only LaAlO$_3$ inclusion existed. It is thus obvious that to obtain the effective inclusion for AF nucleation, the Al content in the steel should not be higher than 0.018 wt%.

#### 5 Conclusions

The effect of Al on microstructure evolution in C–Mn steel with La treatment has been investigated. The conclusions are summarized as follows:

1. After La is added, inclusions in steel turn from MnS and a little Mn-Si-Al-O to La$_2$O$_3$S and MnS, besides a small number of Mn–Si–O + LaAlO$_3$. While the increase of Al makes the inclusion change into LaAlO$_3$ and MnS and some La$_2$S$_3$ in La treated steel.
La$_2$O$_2$S has great effectiveness to induce the formation of AF. At present, making Al less than 0.018 wt% can form La$_2$O$_2$S.

(2) The increase in Al content makes AF formation difficult. The amount of AF in La steel with high Al content is less than that of low Al content. And the range of cooling rates when AF nucleation in La steel turned narrower after the Al content increased.

(3) There are two reasons for Al to be adverse to the nucleation of AF: one is the modification to La-containing inclusion, which reduces the effectiveness to induce the formation of AF; the other is the alloying function of Al, which is beneficial on ferrite forming on grain boundary but is bad for AF formation.

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