Research Article

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Behavior of MnS inclusions during homogenization process in low-alloyed steel FAS3420H

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Abstract: Sulfur is added to low-alloyed steel FAS3420H to improve the free-cutting property of the steel. Elongated MnS inclusions usually deteriorate the mechanical and process performance of steels, so the control of shape and size of MnS is essential. The statistical analysis of MnS inclusions in as-cast and rolled steels at different positions shows that the distribution and aspect ratio of MnS in rolled steel are closely related to the quantity and size of MnS in as-cast one. The *in situ* observation and experimental results reveal that homogenization treatment effectively reduced MnS inclusions size and transformed their shape to globule or ellipsoid. Significant change of quantity and size of MnS inclusions was identified only when the soaking time exceeds 5 h at 1,250°C. Additionally, a kinetic model is presented to quantitatively characterize the behavior of MnS inclusions during homogenization process.

Keywords: MnS inclusions, low-alloyed steel FAS3420H, homogenization, *in situ* observation

1 Introduction

The steel FAS3420H is a low carbon low-alloyed steel that is applied to manufacture components, such as transmission gear, gear shaft, and so on, for heavy-duty truck or coach. The gear components play an important role in transferring power and changing velocity and direction.

Therefore, the gear steel is required to have high fatigue strength [1,2] under the action of long-term alternating load. Also, high quality gear steel not only presents high purity and narrow hardenability bandwidth, but also requires good free-cutting performance to save costs for manufacturing and to strictly control the nonmetallic inclusions in steel to ensure excellent fatigue resistance. MnS is a common type of nonmetallic inclusions in steel, but plays an important role in free-cutting steel [3,4]. For the difference between MnS and the steel matrix, cracks are generated at the tip of the elongated MnS when applying an external force, resulting in a reduced cutting resistance. On the contrary, this characteristic of MnS will reduce the gear fatigue life and the elongated MnS causes steel anisotropy, decreasing its mechanical and process performance [5].

Sims and Dahle [6] classified manganese sulfides in as-cast steel into three different types according to their geometry and sulphide morphology would appear to be closely related to fatigue crack propagation in low-alloyed steel. In particular, type II inclusions, which distribute along the grain boundaries in a form of dot chain, would substantially reduce ductility of steel [7]. To improve the free-cutting performance and mechanical property of steel FAS3420H, the control of size and shape of MnS in steel must be conducted [8].

Ferrite/pearlite banding is a common phenomenon in hot-rolled low-alloyed steel, which usually reduces the mechanical properties of steel, and the prerequisite for its formation is the micro-segregation of the alloying elements [9]. In general, high temperature soaking before hot-rolling process is used to downgrade banded microstructure of gear steel after being rolled. Type II MnS precipitates at grain boundaries due to the micro-segregation in solidification. To control the shape, size, or type of MnS, it is hard to decrease the micro-segregation of solute elements; the behavior of MnS in high temperature soaking and subsequent hot-rolling process is also worthy of attention. Numerous studies [10–13] have been carried out to investigate the behavior of the elongated

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MnS in rolled steel during homogenization. Significant shape change of MnS from slender to spindle-like or spherical was identified during soaking after hot-rolling process, but these methods to change MnS morphology in rolled steel are not practical. At the same time, there were few studies on effects of soaking temperature and time on the morphology and size of manganese sulfide in as-cast steel during high temperature soaking before hot-rolling process.

Better understanding of the behavior of MnS in as-cast steel during soaking could optimize parameters of homogenization process, thereby obtaining appropriate morphology of MnS such as spherical or spindle-like. Moreover, this optimization can promote more uniform distribution of alloying elements in the steel, which can downgrade the banded microstructure after hot-rolling process.

In this study, the influence of different temperatures and homogenization times on the shape, size, and distribution of MnS in continuous casting bloom was studied. The *in situ* observation directly revealed the process by which MnS changed during heat treatment. In particular, a kinetic simulation model will be established to analyze the mechanism of MnS inclusions shape evolution during homogenization for a sufficient time. We thus hoped to gain a better insight into the evolution of MnS inclusions in bloom during heat treatment.

2 Materials and methods

The experimental materials were from commercial steel FAS3420H and the chemical composition is shown in Table 1.

Small specimens (10 mm × 10 mm × 8 mm) were cut from a bloom at three different positions (Edge, 1/2R, Center of the bloom), and they were homogenized in a resistance furnace at respective temperatures of 1,050 and 1,250°C for different times. The thermal schedule is shown in Figure 1(a). According to the research by Shao [18], rapid heating and low temperature are not beneficial to the break up of large-sized elongated MnS. The heating rate was chosen to be 5°C/s and soaking temperature was chosen to be 1,000 and 1,250°C. After being heat-treated, the samples were water-cooled and observed with optical microscope (OM) after being ground and polished and 50

Figure 1: The thermal schedule of (a) homogenization treatment and (b) *in situ* observation.

fields of view were taken for each sample to count the quantity and size of MnS inclusions. The microstructure of FAS3420H bloom was revealed by etching with a 4 vol% Nital solution for 5–10 s and the distribution of MnS was observed based on scanning electron microscope (SEM).

Cylindrical specimens with a diameter of 7 mm and a height of 3 mm were taken from the bloom. The samples were ground and polished and placed in an alumina crucible, and then the behavior of MnS during heating was observed by use of VL2000DX-SVF17SP ultra-high temperature confocal microscope; the thermal schedule is shown in Figure 1(b). The heating rate was 1.67°C/s (100°C/min), the soaking temperature 1,250°C, the duration 20 min, and then the samples were quickly cooled. The entire process was recorded in a video file.

3 Results

3.1 Analysis of inclusions in as-cast steel

The observation of the samples at three different positions of the bloom by SEM revealed that type II MnS inclusions were predominant at all positions. Figure 2 illustrates the morphology of MnS inclusions of three different types in the bloom. Type I MnS inclusions uniformly distribute at the edge, which is spherical or ellipsoidal, shown in Figure 2(a), and type III MnS inclusions have sharp angles, shown in Figure 2(c), and they are irregularly distributed; nevertheless, the dot chain-shaped type II MnS with relative large size distributes along grain boundaries, shown in Figure 2(b). All of these inclusions are almost pure MnS, shown in Figure 3, in which Fe and C

Table 1: Chemical composition of FAS3420H (wt.%)

C	Si	Mn	P	S	Cr	Ni	Mo	N	Al _(t)	O
0.2	0.25	0.85	0.01	0.02	0.55	0.55	0.2	0.008	0.03	0.0009

Figure 2: Morphology of MnS in as-cast steel (a) type I, (b) type II, (c) type III.

Figure 3: Morphology (a) and EDS results (b) of MnS in as-cast steel.

signals from the matrix. Type II MnS inclusions were not beneficial to improve the mechanical and process properties of the experimental steel.

Figure 4: The statistical analysis of MnS at different positions of the experimental steel.

The statistics of overall mean area and quantity of MnS in as-cast steel are illustrated in Figure 4 along with the overall mean aspect ratio of MnS in rolled steel. In general, MnS at the edge has the smallest size, but that at the 1/2R has the biggest size, and there is little difference for quantity of MnS at three positions. The facts show that it is positively related for aspect ratio of the long-strip-shaped MnS in rolled steel to the size of MnS in as-cast steel, so it is possible to change the morphology and distribution of the long-strip-shaped MnS in rolled steel by controlling the size and quantity of MnS in as-cast steel.

3.2 Homogenization treatment

Figure 5 illustrates the variation of morphology of MnS at the 1/2R of as-cast steel with homogenization time at 1,250°C. During homogenization treatment, the morphology of MnS transformed from irregular shape to globule; its number increased but size decreased. Fifty fields

Figure 5: Variation of morphology of MnS at the 1/2R of as-cast steel with homogenization time; (a) 3 h, (b) 5 h, and (c) 10 h.

of view were taken from each sample by use of OM, and the statistical results of the sizes of MnS inclusions were shown in Figures 6 and 7. As seen from Figure 6, the size

of MnS inclusions at 1/2R and center increased to the maximum firstly with homogenization time and then gradually decreased, while that at the edge was slowly

Figure 6: Overall mean area and area distribution of MnS at different positions without and with homogenization at 1,250°C; (a) overall mean area of MnS, (b–d) area distribution of MnS at the edge, 1/2R and center, respectively.

Figure 7: Overall mean area and area distribution of MnS at different positions without and with homogenization at 1,050°C; (a) overall mean area of MnS, (b–d) area distribution of MnS at the edge, 1/2R and center, respectively.

increasing. The homogenization times corresponding to the maximum of MnS size at 1/2R and center, respectively, were different, resulting from different initial sizes of MnS.

With the increase of homogenization time, the size distributions of MnS at different positions have various trends. At the edge, although the size of MnS increased, the number of MnS inclusions in different size ranges per square millimeter gradually increased with homogenization time, and there were few large-size MnS whose area was greater than $20 \mu\text{m}^2$. At 1/2R, the size of MnS reached maximum when the homogenization time was 3 h, but the number of MnS inclusions in all size ranges dropped to a minimum. After that, the number of manganese sulfide increased with homogenization time, but the number

of MnS whose area was greater than $20 \mu\text{m}^2$ decreased. After homogenization, the morphology of MnS changed from large irregular shape to small spherical shape as shown in Figure 5. The behavior of MnS at the center was similar to that at the 1/2R. However, because the initial size of MnS at the center was smaller than that at the 1/2R, the time required to reach the maximum value of the size was longer than that at the 1/2R.

When the soaking temperature became 1,050°C, the behavior of MnS during homogenization treatment changed. The size of MnS inclusions at the edge almost did not change with increasing homogenization time, but the number of MnS per square millimeter increased. At the center, the size of MnS still reached its maximum at 5 h, but then decreased slowly beyond 5 h. The heating

time at which the size of MnS at the 1/2R reached maximum was postponed to 5 h, while that was 3 h when heated at 1,250°C. In summary, MnS inclusions have larger size and smaller quantity after the homogenization process at 1,050°C, which indicates that the effect of homogenization at 1,050°C is not so good as that at 1,250°C.

In general, the area of single particle of MnS in the tested steel is mainly distributed in 0–5 μm^2 , and the homogenization process has a large influence on the size and quantity of MnS inclusions. After homogenization treatment for enough time, the size of MnS shows a tendency to decrease, quantity increases instead. When the homogenization process was at 1,250°C for 5 h or more, suitable MnS was gained with small sizes and big quantity.

3.3 *In situ* observation of MnS during heating

The morphology of type II MnS inclusions at the 1/2R during heating and holding was observed by using an

ultra-high temperature confocal microscope and the results were plotted in Figures 8 and 9.

These type II MnS inclusions precipitated by eutectic reaction were distributed at grain boundaries in a form of chain and close to each other. It was not until 700°C that the morphology of MnS changed and two MnS inclusions started to merge with each other, as indicated by the arrow in the Figure 8(a). This phenomenon occurred more often with increasing temperature. When the temperature rose to 1,250°C, most of the MnS showed a tendency of approaching each other, except for a few MnS which gradually became spherical since they were far away from these neighboring type II MnS, as shown in Figure 8(d). When soaked at 1,250°C for 120 s, these MnS inclusions have almost grown into a long strip which had been shown in Figure 8(e).

Figure 9 showed the morphology of MnS in the sample cooled after *in situ* observation. Some type II MnS inclusions at grain boundary aggregated and grew up, and eventually became short-rod or cylindrical shape, which was consistent with the result of *in situ* observation of the sample. Therefore, after a brief heating, MnS of the

Figure 8: The morphology variation of MnS during heating and holding process (a–c) heating process, (d–f) sample held at 1,250°C for 0, 120, and 390 s, respectively. (a) 700°C, (b) 900°C, (c) 1,100°C, (d) 1,250°C, (e) 1,250°C, (f) 1,250°C.

Figure 9: The morphology of MnS in sample cooled after *in situ* observation.

uniformly distributed type I and irregularly distributed type III gradually spheroidized; some of the aggregately distributed type II MnS grew up, and the size of these MnS increased with short heating.

Based on previous research [6], in the subsequent homogenization process, the two ends of the rod-like and cylindrical MnS would bulge firstly, then axial contraction occurred, eventually the whole MnS would split up and spheroidize. Therefore, the size of MnS would reduce after the split-up of MnS.

4 Discussion

4.1 Controlling elements of MnS for Ostwald ripening behavior

For the steel FAS3420H, the process of heating and homogenization had obvious effects on the morphology modification of MnS inclusions and the effect of homogenization at higher temperatures was distinctly better than that at lower. During the heating and short-term thermal insulation, the type II MnS grew up into long-strip or short-rod-shaped inclusions and other MnS inclusions gradually spheroidized; these results clearly indicated that MnS coarsening was the mechanism responsible for the observed morphology modifications of the inclusions. However, after homogenization treatment at 1,250 or 1,050°C for a sufficient long time, followed by quenching, the size of MnS decreased, but the number increased, which was due to the re-precipitation of MnS by dissolution and diffusion of solute elements throughout the matrix [14].

According to the Ostwald ripening theory [15,16], with the action of interfacial energy, small-sized second-phase particles were dissolved and large-sized particles grew up,

eventually the free energy of the system decreased along with the interface per unit mass. However, if the distance between two MnS inclusions exceeds a certain value, the inclusions would not grow up; finally, these inclusions spheroidize.

The diffusion of atoms is the main controlling factor in the Ostwald ripening process [17]. In order to determine which element is the controlling factor, Yong *et al.* [18] derived the Ostwald ripening theory formula,

$$[\text{Mn}] - k[\text{S}] = C = \sqrt{\frac{D_{\text{Mn-}\gamma}}{D_{\text{S-}\gamma}} \times k \times 10^{5.02-11.625/T}} \cdot \left(\frac{D_{\text{S-}\gamma}}{D_{\text{Mn-}\gamma}} - 1 \right) \quad (1)$$

$$D_{\text{Mn-}\gamma} = 0.16 \cdot \exp\left(-\frac{26,150}{RT}\right) \quad (2)$$

$$D_{\text{S-}\gamma} = 1.7 \cdot \exp\left(-\frac{222,000}{RT}\right) \quad (3)$$

Mn and S have the same effect on Ostwald ripening process in this case. Where $D_{\text{Mn-}\gamma}$ and $D_{\text{S-}\gamma}$ are the diffusion coefficients of Mn atom and S atom, respectively, and at the same temperature, the diffusion coefficient of S atom is two orders of magnitude higher than that of Mn atom. R is the ideal gas constant and T is the absolute temperature, k is the relative atomic weight ratio of Mn and S (A_{Mn} and A_{S} are 54.938 and 32.06 respectively), $k = A_{\text{Mn}}/A_{\text{S}}$, C is the derived diffusion control element criterion, a function independent of the matrix. The relationship between $[\text{Mn}] - k[\text{S}]$ and C is shown in Figure 10, where $[\text{Mn}]$ and $[\text{S}]$ are both mass percentage of the contents of Mn and S in steel, respectively. It can be seen from Figure 10 that the control elements of Ostwald ripening behavior would change if the temperature is different for the same steel. When $C > [\text{Mn}] - k[\text{S}]$, S atom is the control element, when $C < [\text{Mn}] - k[\text{S}]$, Mn atom is the control element.

Figure 10: The relationship between $[Mn]-k[S]$ and the diffusion control element criterion.

According to the chemical composition of the experimental steel, $[Mn]-k[S] = 0.816$, $C_{1050} = 0.335$, $C_{1250} = 0.998$. When samples were homogenized at $1,250^{\circ}\text{C}$, $C_{1250} > [Mn]-k[S]$; from Fick's first law, it can be concluded that the diffusion flux of Mn atoms, J_{Mn} , was greater than the diffusion flux of S atoms, J_S , so S atom was the control factor for Ostwald ripening behavior of MnS in this experimental steel at $1,250^{\circ}\text{C}$, but that was Mn instead when samples were homogenized at $1,050^{\circ}\text{C}$ since that $C_{1050} < [Mn]-k[S]$. It is possible that the different control factors lead to the discrepancy of effect of homogenization at different temperatures.

4.2 Kinetic simulation of homogenization process

In order to understand the distribution of solute atoms in the matrix, the DICTRA [19] kinetic software was used to simulate the solidification process and homogenization process of the experimental steel.

To perform simulation using DICTRA, both thermodynamic (tcfe8) and kinetic descriptions (mobfe3) are needed. For simplicity, a superior alternative is to store atomic mobility in the database, rather than diffusion coefficients. From absolute-reaction rate theory arguments, the mobility coefficient for an element B, M_B , may be defined as follows [20]:

$$M_B = \exp\left(\frac{RT \ln M_B^0}{RT}\right) \exp\left(-\frac{Q_B}{RT}\right) \frac{1}{RT} \Gamma \quad (4)$$

both $RT \ln M_B^0$ and $-Q_B$ are in general dependent upon the composition, temperature, and pressure. Γ is a factor taking into account the effect of the ferromagnetic transition, and it is a function of the alloy composition. By assuming that the vacancy concentration be governed by thermodynamic equilibrium, the diffusional flux of an element k can be expressed as follows [21]:

$$\bar{J}_k = -c_k \gamma_{Va} \Omega_{kVa} \frac{\partial \mu_k}{\partial z} \quad (5)$$

here c_k is the amount of k per unit volume, γ_{Va} is the fraction of vacant lattice sites where k is dissolved, Ω_{kVa} is a kinetic parameter which describes the exchange rate if there is a vacancy adjacent to an atom k and μ_k is the chemical potential of k. The mobility M_k is defined as:

$$M_k = \gamma_{Va} \Omega_{kVa}, \quad \text{when k is substitutional} \quad (6)$$

$$M_k = \Omega_{kVa}, \quad \text{when k is interstitial} \quad (7)$$

The homogenization process of the experimental steel after it has been cooled from $1,600$ to $1,250^{\circ}\text{C}$ at a cooling rate of 2°C/s was simulated in this model. The geometry used in simulation process is shown in Figure 11 and its length of the geometry is half the secondary dendrite arm spacing whose mean value is $18.65 \mu\text{m}$, measured in Figure 12 by image processing software ImageJ. BCC first precipitates from the liquid phase, and a peritectic reaction, $\text{BCC} + \text{Liquid} = \text{FCC}$, occurs when the

Figure 11: The schematic diagram of the geometry used in simulation process.

Figure 12: Dendritic morphology at the edge of as-cast steel and sketch map of the secondary dendrite arm spacing.

Figure 13: Variation of segregation factor of main element in as-cast steel along with homogenization time (a) as-cast, (b) 1 h, (c) 3 h, (d) 5 h.

temperature continues to decrease; eventually the phase of the entire model is FCC, and diffusion and counter-diffusion of solute elements of the experimental steel have taken place throughout the process.

The simulation results of DICTRA are plotted in Figure 13. Distance 0 represents the branch position of the dendrite and distance 18.65×10^{-6} m represents the inter-dendritic position. A new parameter, Segregation Factor (SF), is defined as follow,

$$SF = \frac{C_{\text{local}}}{C_{\text{average}}} \quad (8)$$

where C_{local} and C_{average} are the local and average concentration of solute elements, respectively. The SF at the inter-dendritic position is counted in Table 2.

Figure 13 shows that as the homogenization time increases, the distribution of solute elements within the entire dendrite gradually becomes uniform. Generally, C and S in steel are extremely more easy than Mn to segregate, but actually, due to the back diffusion of solute elements during solidification process, the degree of segregation of Mn is much larger than that of C and S, as shown in Figure 13(a). Compared with Figure 13(a–d), the distribution of C and S in the

Table 2: Segregation factor of Mn at different times

SF	As-cast	1 h	3 h	5 h
Mn	1.26691	1.03822	1.00253	1.00122

dendrite was uniform after homogenization for 3 h, while Mn needed 5 h. It is concluded that S needs less time than Mn to reach uniform distribution.

Table 2 summarizes the segregation factors of Mn at different homogenization times at the inter-dendritic position. The diffusion of Mn was a little fast at the initial stage of heating. After 3 h, the segregation factor of Mn changes less significantly with the extending of the homogenization time.

The controlling factor of Ostwald ripening behavior at 1,250°C is S, but that is Mn when the temperature is 1,050°C; diffusion coefficient of S is much larger than that of Mn.

Atomic diffusion leads to the growth, dissolution, and re-precipitation of manganese sulfide inclusions in steel. It was at 3 h that S became uniform in the whole dendritic when samples were heat-treated at 1,250°C. S combined with the enriched Mn at the inter-dendritic, leading to the precipitation of manganese sulphide. Therefore, the size of MnS reached to the maximum at 3 h, as shown in Figure 6(a). With the homogenization of Mn in the subsequent heat treatment after 3 h, the precipitation of MnS occurred at other high energy sites, such as grain boundaries, grain angles or internal defects, and so on. At the same time, the long-striped MnS inclusions broke into several segments by dissolution, diffusion through the matrix, and then gradually spheroidized [22]. Eventually, after homogenization for 5 h or more, the size of MnS inclusions became smaller, but the number increased; simultaneously the morphology became spherical and the distribution was improved slightly.

When homogenized at 1,050°C, Mn is the controlling factor for Ostwald ripening behavior of MnS. It was the slow diffusion of Mn that delays the coarsening of MnS; as a result, the size of MnS reached to the maximum till 5 h, as shown in Figure 7(a). Meanwhile, the dissolution and diffusion of solute elements, by which the long-striped MnS broke, need more time.

5 Conclusions

In this paper, the type, quantity, and distribution of manganese sulphide inclusions in low-alloyed steel FAS3420H were studied, and the morphology modification of MnS after homogenization was observed. At the same time, the

variation of size and number along with homogenization time has been analyzed systematically and the behavior of MnS during heating and homogenization was observed based on an ultra-high temperature confocal microscope. The conclusions can be drawn as follows.

- (1) The aspect ratio of long-striped MnS in rolled steel is positively correlated with the size of MnS in as-cast steel. After homogenization treatment at 1,250°C for a sufficient time, such as 5 h or more, the size of MnS in as-cast steel decreased, the quantity increased, the morphology became spherical, and the distribution was improved slightly. In that way, the aspect ratio and distribution of MnS in rolled steel can also be ameliorated.
- (2) Atomic diffusion of Mn and S is the key control factor of shape evolution of MnS for steel FAS3420H when the samples are homogenized at 1,250 and 1,050°C, respectively. Compared to manganese, sulfur promoted the process of growth, dissolution, and re-precipitation of MnS by its rapid diffusion.
- (3) With appropriate increase in soaking temperature or time, such as 1,250°C for 5 h, not only the solute element homogenization can be achieved, but also MnS with reduced aspect ratio gained in steel after being hot-rolled.

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Data availability statement: The data that support the findings of this study are available from the corresponding author upon reasonable request.

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