Cerium Addition Effect on Modification of Inclusions, Primary Carbides and Microstructure Refinement of H13 Die Steel

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A comprehensive study of the Ce addition effect on microstructure, inclusions, and primary carbides in H13 steel was carried out. 3D morphology of inclusions and primary carbides was assessed by the non-aqueous electrolytic method. The addition of 0.0038 mass% Ce had no obvious refinement effect on dendritic structures in H13 steel. With Ce content increase from 0.0038 to 0.019 mass%, dendritic structures of H13 steel were refined. Al2O3 and MnS inclusions in original H13 steel promoted heterogeneous nucleation of primary carbides. Al content increased, and S content decreased with increasing Ce content. When the latter was increased from 0.0038 to 0.019 mass%, the original inclusions were modified first to Al11O18Ce and CeAlO3, then to Ce2O3, and, finally, to Ce2O2S. Numerous small-sized Ce2O2S inclusions were found in steel with 0.019 mass% Ce, which promoted nucleation of γ-Fe during solidification and contributed to the refinement of as-cast dendritic microstructures of H13 steel. Besides, Ce2O2S inclusions suppressed heterogeneous nucleation of primary carbides. The size of primary carbides decreased, and their morphology became less developed due to the finer microstructure hindering their growth. Finally, banded structures in forged Ce-bearing H13 steel were improved.

KEY WORDS: H13 steel; cerium addition; microstructures; inclusions; primary carbides.

1. Introduction

Hot-working die steel used in the metal forming process is exposed to high-temperature and mechanical cyclic loading during its service life.1,2) Large-size second-phase particles, such as oxide inclusion, sulfide inclusion, and primary carbide, act as the initial sites for the crack development in the alloy matrix.3–7) Furthermore, the above harsh environment greatly promotes crack growth. The medium-carbon H13 (4Cr5MoSiV1) die steel, which combines good hardenability, high-temperature hardness, and toughness, is now widely used as a raw material for producing hot-working dies.8,9) Dendritic segregation in the H13 steel contributes to generating primary carbides in it.10) On the other hand, the dendritic segregation will be inherited after the hot forming process, creating banded structures and violating the steel matrix isotropy.11) The above banded structures and primary carbides would negatively affect the H13 steel toughness and reduce its service life.

Several ways have been proposed to mitigate the dendritic segregation and primary carbides. The high-temperature homogenization treatment is one of the most effective methods because the enriched solutes in the inter-dendritic regions would sufficiently diffuse during the long soaking time under a high temperature, and the primary carbides would decompose and dissolve at the same time.11,12) However, the method is energy-intensive and would give rise to grain coarsening.12) Increasing the cooling rate can also achieve the refinement of dendritic structures and suppress the dendritic segregation since a higher cooling rate could reduce the secondary dendrite arm spacing, which is used to measure the scale of dendritic segregation.13) Meanwhile, the refinement of dendritic structures would restrain the growth of primary carbides.13,14) Due to the casting process’s thermal nature, the cooling rate of the commercial-scale casting is limited. Numerous studies have shown that the common
oxide inclusions in Al-killed steel, such as MgO·Al₂O₃ and Al₂O₃ inclusions, would act as nucleation agents for the heterogeneous nucleation of primary carbide and promote the nucleation and growth of primary carbides.\(^{10,15–17}\) According to our previous study, sulfide inclusion could notably motivate the primary carbide nucleation in H13 steel.\(^{10,15}\) It was found that transforming the MgO·Al₂O₃ and Al₂O₃ inclusions into calcium-aluminate inclusions by calcium treatment inhibited the heterogeneous nucleation of primary carbides in high-carbon tool steel due to the low melting point of calcium-aluminate inclusions.\(^{16,17}\) Nevertheless, the high-temperature homogenization treatment and increased cooling rate method mentioned above cannot prevent the inclusions from becoming the nucleation agents of primary carbides.\(^{10,18}\) In contrast, calcium treatment has no known dendritic structures improvement in H13 steel.\(^{17}\)

As is known, rare earth (RE) elements are favorable for enhancing the solidification structure due to their extremely strong affinity for oxygen and sulfur in steel to generate high-melting-point RE-oxide and RE-oxysulfide inclusions, which can effectively promote the nucleation of δ-Fe and γ-Fe during the steel solidification.\(^{19–23}\) Noteworthy is that RE elements also improve primary carbides in steel, not only because of the solidification structure enhancement. A study on the changes of primary carbides in D2 tool steel with Ce addition showed that the lamellar-like primary carbides transformed into globular-shaped ones.\(^{24}\) This has been explained based on the first principle. The formation of Ce-oxide and Ce-oxysulfide inclusions contributed to the elimination of enriched oxygen and sulfur in grain boundaries, which improved the molten steel’s isotropy. And then, the improved isotropy was the factor controlling the morphology change of primary carbides. The term “disregistry” implies a nucleating agent’s capability to promote the nucleation of solid: the smaller the disregistry, the stronger the capability.\(^{25}\) Diseregistries between the nucleating agent Ce-oxide as well as Ce-oxysulfide inclusions, and nucleated solid primary carbides in H13 steel were calculated. The results confirmed that Ce-oxide and Ce-oxysulfide inclusions were ineffective in heterogeneous nucleation of primary carbides.\(^{26}\)

The addition of RE elements can refine the dendritic structures and modify inclusions simultaneously, both of which may be beneficial for controlling primary carbides. Thus, the authors were motivated to carry out a comprehensive investigation of the evolution of dendritic structures and inclusions within as-cast H13 steel with Ce addition, as well as their effects on primary carbides. Besides, the banded structure of forged H13 steel with Ce addition is also considered in the present work, aiming to provide guidance on solidification structure and primary carbides’ improvement for manufacturing the H13 steel.

## 2. Experimental Procedure

To obtain H13 steel with different Ce contents, raw materials were first taken from a commercial H13 steel ingot, as indicated in Fig. 1. The addition of Ce was carried out using a high-frequency induction furnace, as shown in Fig. 2.

A 500 g slab of as-received H13 steel was polished and charged into an alumina crucible with a 40 mm inner diameter and then heated to 1 823 K. After the H13 steel was

![Fig. 1. Schematic of investigated materials’ preparation and sampling procedures. (Online version in color.)](image)

![Fig. 2. Schematic diagram of remelting experiments. (Online version in color.)](image)

| Table 1. Chemical composition of five remelted samples (mass%). |
|---|---|---|---|---|---|---|---|---|---|
|   | C   | Si  | Mn  | V   | Mo  | Cr  | Al  | T. O | S   | Ce   |
| S1 | 0.41 | 0.87 | 0.36 | 1.05 | 1.32 | 5.27 | 0.019 | 0.0033 | 0.0034 | –    |
| S2 | 0.41 | 0.86 | 0.36 | 1.05 | 1.32 | 5.27 | 0.017 | 0.0042 | 0.0033 | 0.0038 |
| S3 | 0.40 | 0.87 | 0.36 | 1.05 | 1.31 | 5.27 | 0.021 | 0.0049 | 0.0026 | 0.0074 |
| S4 | 0.41 | 0.86 | 0.37 | 1.05 | 1.31 | 5.27 | 0.024 | 0.0057 | 0.0017 | 0.0130 |
| S5 | 0.41 | 0.86 | 0.37 | 1.05 | 1.31 | 5.27 | 0.022 | 0.0051 | 0.0017 | 0.0190 |
fully melted, Ce metal with oxidation layer was carefully ground, covered by iron foil, and placed into the molten steel. One steel sample (S1) was remelted without Ce addition, while 0.066, 0.162, 0.324 and 0.660 g Ce were added to the other four samples (S2 to S5), respectively. This yielded 0, 0.0038, 0.0074, 0.013, and 0.019 mass% contents of Ce in specimens S1 to S5, respectively. More detailed chemical compositions of the above five samples are listed in Table 1, in which T. O stands for total oxygen. The C and S contents were determined by C-S analyzer (CS600,

Fig. 3. Comparison of dendritic structures in the remelted H13 steel samples with different Ce contents: (a) S1; (b) S2; (c) S3; (d) S4; (e) S5. (Online version in color.)

Fig. 4. Typical inclusions in S1 sample: (a) Al₂O₃ inclusions with attached primary carbides; (b) EDS analysis of (a); (c) sulfide inclusions with attached primary carbides; (d) EDS analysis of (c). (Online version in color.)
LECO, USA). The T. O content was determined by O-N analyzer (TC500, LECO, USA), and other chemical compositions were determined by ICP-AES (Agilent 725, Agilent Technologies Inc. USA).

After a 20 min induction melting, the five samples of the remelted H13 steel with different Ce contents were produced. And once again, new subsamples were collected from the above five samples at the same position (center of the ingot and 10 mm height from the bottom) for the following examination. The new subsamples used for dendritic structure observation were etched by 4% nital and studied via an optical microscope (OM, DSX 510, Olympus Co., Japan). The morphology and composition of primary carbides and inclusions were determined by scanning electron microscopy (SEM, Nova 400 Nano, FEI Co., USA) equipped with an energy-dispersive spectrometer (EDS, Le350 PentaFETx-3, Oxford Instruments Co., UK). Fifty photos of each subsample were taken by SEM at 1000X magnifications for number and size statistics of primary carbides and inclusions. The three-dimensional (3D) morphology of primary carbides and inclusions was obtained through a non-aqueous electrolytic method.

To investigate the effect of Ce addition on the banded structure of H13 steel after the forming process, the remaining remelted H13 steel samples with different Ce contents were forged into a square rod with a 15 mm × 15 mm cross-section. Then, the forged square rod was heated to 1 123 K at a hold time of 2 h, then cooled in the furnace to 773 K, and finally, was cooled in the air to room temperature. The forged and annealed steels were then sampled at the same position. After etching by 4% nital, each sample’s banded structures were examined via the OM, and the microhardness of each sample was determined by a Vickers hardness tester (HV-1000B, Xinyun Co., Ltd., China).

3. Results and Discussion

3.1. Evolution of Dendritic Structures

Primary carbides are mainly distributed in the inter-dendritic region of dendritic structures produced by dendritic segregation, and their size is directly related to the dendritic structures. Figure 3 shows the evolution of dendritic structures in the remelted H13 steel with increasing Ce content. These pictures were acquired from the dark-field model of OM, and the black net-like structures in pictures correspond to inter-dendritic regions of as-cast H13 steel.

It can be seen from Fig. 3 that the dendritic structures of S1 and S2 samples differ only slightly. However, dendritic structures get finer with the Ce content increase from 0.0038 mass% in the S2 sample to 0.019 mass% in the S5 sample. This implies that adding a small amount of cerium (0.0038 mass%) fails to improve the solidification structure of H13 steel. Dendritic structures will only be promoted when the added Ce amount exceeds a certain threshold level. Thus, the addition of 0.0074–0.019 mass% of Ce enhances dendritic structures, especially in the S5 sample with Ce content of 0.019 mass%.

3.2. Modification of Inclusions

The typical inclusions in the S1 sample are Al2O3 and MnS, which are reported in previous studies to be potent in promoting the nucleation of primary carbides in H13 steel. Inclusions attached to primary carbides are easily found in the S1 sample, as shown in Fig. 4.

The primary carbides nucleating on Al2O3 inclusions are usually rich in vanadium (Fig. 4(a)), while those nucleating on MnS inclusions have an enrichment of molybdenum (Fig. 4(c)). Al2O3 inclusions have lower disregistry with V-rich primary carbides, as compared to MnS inclusions, and MnS inclusions have lower disregistry with Mo-rich primary carbides in comparison with Al2O3 inclusions. Figure 5 shows the primary carbides nucleating on inclu-

![Fig. 5. 3D morphology and elemental mapping of primary carbides with inclusion cores: (a), (b) primary carbides nucleating on Al2O3 and elemental mapping; (c), (d) primary carbides nucleating on MnS and elemental mapping. (Online version in color.)](image-url)
sions. Figures 5(a) and 5(c) depict the 3D morphology of the above two kinds of complex precipitates. It can be observed that peaks of elemental S and Mo are overlapped in the EDS analysis. MnS inclusions in Fig. 5(c) can be distinguished by the aggregation of elemental Mn in Fig. 5(d). Noteworthy is that none of MnS inclusions are seen to exist alone, which is due to that the formation timing and location (interdendritic region) of MnS inclusions, which are located close to primary carbides.\(^{15}\)

After 0.0038 mass% Ce was added to H13 steel, most of the \(\text{Al}_2\text{O}_3\) inclusions transformed into Ce–Al–O system inclusions in the S2 sample (Fig. 6(a)). It can be inferred that a small amount of Ce addition can effectively modify the \(\text{Al}_2\text{O}_3\) inclusions into the Ce–Al–O system inclusions, which has also been proved in previous study.\(^{27}\) MnS inclusions are also found in the S2 sample. Besides, complex inclusions (shown in Fig. 6(c)) appear, consisting of MnS and Ce–Al–O system inclusions bearing less Ce content than that mentioned above. Primary carbides can still nucleate on the complex inclusions, as shown in Fig. 6(c). The Ce–Al–O system’s stability diagram in the S2 sample was calculated using the Factsage 6.1 software (Databases: FToxide and FTmisc, Phase Diagram module) and plotted in Fig. 7, where \(w[\text{Al}]\) and \(w[\text{Ce}]\) stand for the mass fraction of Al and Ce content, respectively. The red spot in Fig. 7 stands for the chemical composition of the S2 sample, indicating a coexistence area of \(\text{CeAlO}_3\) and \(\text{Al}_{11}\text{O}_{18}\text{Ce}_x\), which should be Ce-rich Ce–Al–O system inclusions and low-Ce Ce–Al–O system inclusions in S2, respectively. The 3D morphology of typical oxide inclusions in S1 and S2 samples is shown in Fig. 8. Figure 8(a) depicts separate \(\text{Al}_2\text{O}_3\) inclusions in the S1 sample. Both the \(\text{Al}_2\text{O}_3\) inclusions in S1 and Ce–Al–O system inclusions in S2 are approximately spherical (globular).

Continuing increase of Ce content, the typical inclusions in the S3 sample are shown in Figs. 9(a) and 9(c), which are identified as Ce–O and Ce–O–S inclusions according to EDS analysis of Figs. 9(b) and 9(d). As to inclusions in S4 and S5 samples, except for Ce–O inclusions (as shown in Fig. 9(a)), larger numbers of small-sized inclusions exist as depicted in Fig. 9(c), and the EDS analysis as shown in Fig. 9(f) illustrates that they are Ce–O–S inclusions. None of the Ce–O and Ce–O–S inclusions in S3, S4, and S5 samples are nucleation sites for primary carbides. The 3D morphology of Ce–O–S in S3, S4, and S5 samples is shown in Fig. 10. Ce–O–S inclusions in S4 and S5 samples are finer than those in S3 one. The number and size of the oxide, sulfide

![Fig. 6. SEM images and EDS analyses of inclusions in S2: sample (a), (b) Al–Ce–O inclusions and EDS analysis; (c), (d) Al–Ce–O–MnS complex inclusions and EDS analysis. (Online version in color.)](image-url)

![Fig. 7. Stability diagram of oxide inclusions in the investigated H13 steel. (Online version in color.)](image-url)
and oxysulfide inclusions in each sample were counted and summarized in Table 2.

It should be noted that the thermodynamic calculations of S3 to S5 were not carried out due to the lacking of Ce-oxysulfide data in the present databases. However, similar evolution behavior of Al2O3 inclusion that modified by Ce is also observed in previous study.28) When Ce content is raised to 0.0038 mass%, the number of inclusions with added Ce increases, and their average size first increases slightly and then drops until Ce content reaches 0.013 mass%. Al2O3 has a high melting point and should remain solid under the test conditions. Thus, it can be assumed that as trace Ce is added to the investigated H13 steel, it will first participate in the transformation of original Al2O3 inclusions, first from Al2O3 to Al11O18Ce and then to CeAlO3. With a further increase in Ce content, inclusions are modified to Ce2O3 and Ce2O2S.28) Therefore, the size of the inclusions would be influenced by the size of the original Al2O3 inclusions. However, if the amount of Ce is sufficient for modifying original inclusions, dissolved O and S elements can also form Ce2O2S. The newly-formed Ce2O2S inclusions have finer size than the modified ones.

To achieve a visualized recognition of the modification degree of inclusions in the five samples with varying Ce content, the composition of fifty random inclusions in each sample was detected by SEM-EDS. The distribution of various inclusion types in five tested samples is plotted in Fig. 11. As seen in Fig. 11, the addition of 0.0038 mass% Ce can modify most of the Al2O3 inclusions into CeAlO3 ones, while MnS will remain and promote the formation of Al11O18Ce–MnS complex inclusions. As Ce content is increased to 0.0074 mass%, Ce2O3, and Ce2O2S inclusions appear and are accompanied by unmodified CeAlO3, MnS, and Al11O18Ce–MnS complex inclusions, which means a further addition is needed. Meanwhile, 0.013 mass% Ce completely modified all inclusions into Ce2O3 and Ce2O2S ones, and the percentage of Ce2O2S inclusions increases when 0.019 mass% Ce is added.

The evolution of inclusions also explains the variation of Al and S contents, as shown in Table 1. The T.O content of samples with increasing Ce addition is due to the oxidation of pure metal Ce. In the S2 sample, the amount of Ce cannot transform all inclusions, brings oxygen into steel, and reduces Al content from 0.019 to 0.017 wt.%. However, Al content increases when Ce content is raised from 0.0038 to 0.019 wt.%, although T. O increases too. This occurs because the elemental Al in Al2O3 inclusions will be replaced by Ce, which contributes to an increase of Al content. It has to be noticed that, the influence of Al2O3 crucible on the Al content in steel can be ignored, though Al2O3 phase exists in the crucible as well. Because there is no obvious increase in Al content as Ce content increases form 0.0038 mass% to 0.019 mass%. As to S content, there is no obvious difference between S1 and S2 samples. The S content starts to drop as Ce content increases from 0.0038 mass% in S2 to 0.013 mass% in S4 for the generation of Ce2O2S inclusions and does not change when Ce is further increased to 0.019 mass% in S5 due to the complete modification of inclusions.

Fine Ce2O2S inclusions can act as effective nucleation agents of γ-Fe during the solidification of H13 steel,23) improving the nucleation rate and degree of undercooling and finally contributing to finer dendritic structures of steel.25) The heat release behavior of high-speed steel with and without Ce addition during solidification through DSC has been investigated by Wang et al.,29) who reported that
the phase-transition starting temperature of the liquid phase to the $\gamma$-Fe phase in steel was increased after Ce addition. The dissolved sulfur in molten steel is accumulated in the liquid phase before the solidification front due to micro-segregation. The enriched sulfur will decrease the solidus temperature and extend the local solidification time, aggravating micro-segregation in turn. If a sufficient content of Ce is added, the dissolved S is fixed in the Ce–O–S inclusions, which can potentially prevent micro-segregation of sulfur.

3.3. Effect of Ce Addition on Primary Carbides’ Refinement

Primary carbides generated from dendritic segregation and growing in inter-dendritic regions are greatly affected by dendritic structures, for they provide primary carbides with growth space. Figure 12 depicts the 3D morphology of primary carbides in the matrix of S1 and S5 samples acquired by the non-aqueous electrolytic method. Primary carbides lie in the matrix hollows (pits), which are distributed along with small granular secondary carbides. Figure 12(b) shows that secondary carbides distributed in the pits are preferentially located in the matrix. This indicates that hollows are supposed to be inter-dendritic areas rich in solutes due to micro-segregation, which results in precipitation of primary carbides in the residual liquid phase during solidification and precipitation of secondary carbides in the solid phase during the subsequent cooling process. Primary carbides in the S1 sample, as shown in Fig. 12(a), are more developed and have complex morphologies like stretching arms into the surrounding inter-dendritic areas. However, primary carbides in the S5 sample are less developed, and the morphologies are short bands (strips).

The two-dimensional average, maximum, and minimum diameters of each primary carbides in the observation area are measured on SEM images at 1000X magnifications by the Image Pro-Plus software. Results in Fig. 13(a) present...
the distribution of the average diameter of all primary carbides in the observation area of each sample fitted by the normal distribution. It indicates that the primary carbides in the S5 sample, with added 0.019 wt.% Ce, are finer than those in the other four samples. Figure 13(b) describes the distribution of the maximum-to-minimum diameter ratios for primary carbides in each sample, which is also fitted by the normal distribution law.

The highest probability of the primary carbides’ average diameter corresponds to about 4 μm in all five samples. However, this probability increases as Ce content is raised from 0.0038 to 0.019 mass%, which means that primary carbides become less developed with increased Ce content. The main reason for the improvement of primary carbides is the refinement of the solidification structure. Because the inter-dendritic areas provide primary carbides with growth space,10,14) shrinking of inter-dendritic areas will limit the growth of primary carbides, as shown in Fig. 12.

3.4. Evolution of Banded Structure and Microhardness

Banded structures are reported to be a manifestation of dendritic structures in forged steels. In forged H13 steel after the annealing process, banded structures consist of unevenly distributed secondary carbides.11) Figure 14 shows the banded structures in forged and annealed five samples. Bright bands in the matrix, as shown in Fig. 14, are distributed with more secondary carbides than dark bands. The bright bands in photos F1 and F2 (Fig. 14) are similar but get narrower with increasing Ce content, while F5 contains the narrowest bright bands of all five cases depicted in Fig. 14. The distribution of secondary carbides gets more homogenous after adding 0.019 mass% Ce.

Microhardness values of bright and dark bands were determined using the Vickers hardness tester. Ten random (both bright and dark) bands in each sample were tested. The average microhardness values of bright and dark bands

<table>
<thead>
<tr>
<th>Sample</th>
<th>Average diameter (μm)</th>
<th>Number</th>
</tr>
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<tbody>
<tr>
<td>S1</td>
<td>1.96</td>
<td>75</td>
</tr>
<tr>
<td>S2</td>
<td>2.06</td>
<td>91</td>
</tr>
<tr>
<td>S3</td>
<td>1.79</td>
<td>100</td>
</tr>
<tr>
<td>S4</td>
<td>1.48</td>
<td>106</td>
</tr>
<tr>
<td>S5</td>
<td>1.49</td>
<td>119</td>
</tr>
</tbody>
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Fig. 10. 3D morphologies of Ce–O–S inclusions in various samples: (a) S3, (b) S4, and (c) S5.
in five samples are plotted in Fig. 15. The average microhardness of bright bands in the S1 sample was the highest due to the enrichment of secondary carbides. That of dark bands in S1 was the lowest due to the scarcity of secondary carbides. However, the microhardness of bright bands gets lower, and that of dark bands gets higher as Ce content increases, which is the result of uniformly distributed secondary carbides. In other words, the isotropy of H13 steel is improved with Ce addition. The main reason is the improvement of dendritic segregation contributed by the refinement
of dendritic structures through Ce addition. Additionally, the S content reduction also enhances banded structures in the steel under study.30)

4. Conclusions

(1) Trace Ce addition (0.0038 mass%) fails to refine dendritic structures in H13 steel. However, the dendritic structures are greatly improved if the Ce content is increased from 0.0038 to 0.019 mass%.

(2) Numerous small-sized Ce₂O₂S inclusions are generated in H13 steel with a 0.019 mass% Ce content, promoting the nucleation of the γ-Fe phase during solidification and preventing the heterogeneous nucleation of primary carbides. The formation of Ce₂O₂S inclusions is also beneficial for reducing the S content. The effective nucleation of γ-Fe phases and the S content reduction jointly contribute to the refinement of dendritic structures.

(3) Due to the refinement of the solidification structure, the growth of primary carbides is limited. Primary carbides in H13 steel with a 0.019 mass% Ce content have a smaller size and less developed morphology.

(4) If a sufficient amount of Ce is added, the bright bands in the banded structure of forged and annealed H13 steel samples with more distributed secondary carbides get narrower due to the refinement of dendritic structures, while the H13 steel isotropy is improved.

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