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Investigation on Band Segregate Formation during the Electroslag Remelting of H13 Die Steel

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Abstract. Band segregation has been found in the H13 die steel produced by the electroslag remelting (ESR) technology. Chemical and metallographic studies have been carried out on a one ton ESR ingot of H13 die steel, so as to understand the formation mechanism of the band segregation. The results indicate that the T.O content and S content decreased because of cleanliness improvement of ESR process. Transverse macrosegregation of S content decreased after ESR. The overall removal ratio of the inclusion is around 65.8%. The original complex inclusions would be modified to the CaO·Al₂O₃ inclusions. Al₂O₃ and MnS inclusions can be found after ESR. Both of Al₂O₃ and MnS inclusions were found to be the core of primary carbides. The net like structure in ESR ingot and banded structure in the forged steel were observed. V, Mo, Cr and S are rich in the segregation areas of ESR ingot. Besides, black and white segregation bands can be observed on the forged steel samples after etching. Uneven distribution of carbides rich in V, Mo and Cr was observed in banded structure.

1. Introduction

H13 steel is a common hot-working die material, for its good mechanical properties. In order to avoid abnormal failure, it is important to decrease the defects of the H13 steel such as inclusions and segregation^[1-4]. Segregation is unavoidable during the solidification, severe segregation which cannot be completely eliminated by heat treatment would generate adverse effect on the final products. During solidification of H13 steel, elements like C, S and V would diffuse from solidified steel to molten steel causing enrichment of these elements in inter-dendritic, that is what we call dendritic segregation, a kind of microsegregation, large size precipitates can also be found in the segregation areas. After forging or rolling, banded structures always exist in H13 steel. Those precipitates and banded structures mentioned above will lower the impact toughness of H13 steel, the isotropy also can be decreased strongly.

Electroslag remelting (ESR) is usually adapted to produce H13 steel for the excellent ability in removing inclusions and improving solidification structure^[5-7]. However, ESR still can't solve the microsegregation entirely.

Lots of researches have been done to decrease segregation. Li *et al.*^[8] have studied the effect of trace magnesium addition on the inclusions modification and carbide improvement, they found that the disregistry between MgO·Al₂O₃ inclusions and γ -Fe is smaller than that between Al₂O₃ inclusions and γ -Fe, which means MgO·Al₂O₃ inclusions are more effective in promoting nucleation of γ -Fe. Comparing with Al₂O₃ inclusions, the modified inclusions MgO·Al₂O₃ are less possibly to cluster, and the finer and better distributing MgO·Al₂O₃ inclusions would contribute to the improvement of solidification structure. Besides, the primary carbides could be also refined. Liu *et al.*^[9] found that MnS inclusions can be the nucleation agent of primary carbides, the segregation decrease with



desulphurization. MA *et al.*^[10] investigated the thermal homogenization treatment. After being kept in 1200°C for 8 hours, the isotropy of H13 was improved.

Those works mentioned above did show obvious improvement on the segregation in as-cast steel, but there is still lacking of the researches about segregation in forged steel. In the present work, the inclusions in the H13 steel were studied. The segregation in both the as-cast steel and forged steel were investigated.

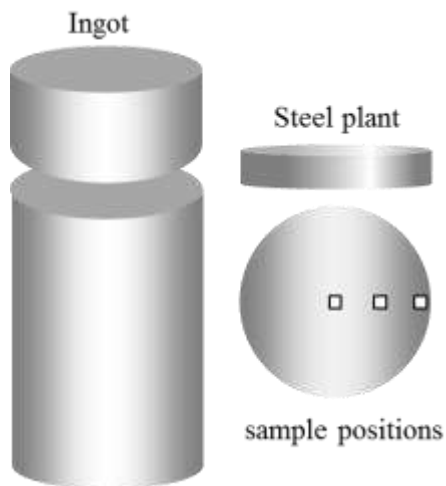


Figure 1. Schematic diagram of the ingot and sample positions

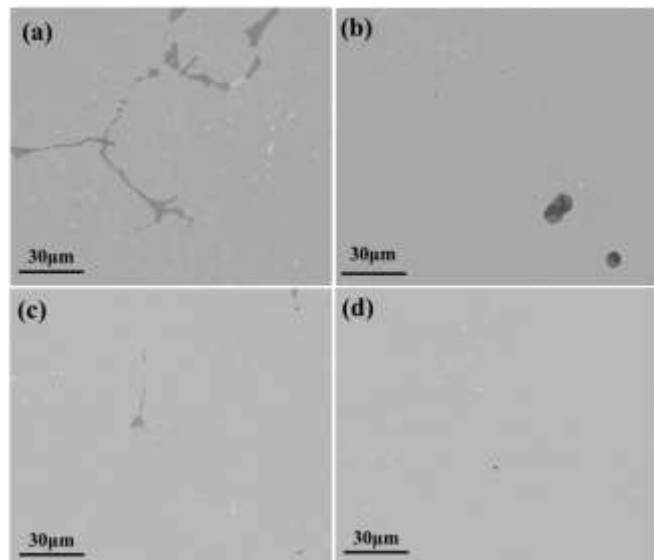


Figure 2. images of the inclusions and precipitates in the steel samples: (a) and (b) electrode, (c) and (d) ESR ingot.

2. Experiment

H13 steel was manufactured with EAF→LF→VD→Ingot Casting→ESR (an one ton electroslag furnace under air atmosphere)→Forging procedure. The slag used in ESR process consists of 70% CaF₂+30%Al₂O₃ (38 Kg). The current of the remelting process is about 9000A, and the remelting process lasted for 4 hours. Steel plants were taken from head of the electrode (200mm in diameter), ESR ingot (350mm in diameter) and forged steel (150mm in diameter). The samples were taken from the center, half of the radius and surface of electrode and ESR ingot with a size of 4×4×4mm³, which were used to analyze the chemical compositions. One sample of 10×10×10mm³ in size was taken from the center of each plant to study the inclusions, and segregation. Figure 1 is the schematic diagram of the ingots and positions where the samples were taken from. The C, S, O and N content and other elements were determined by C-S analyzer, O-N analyzer and ICP-AES. The size, morphology and composition of the inclusions were investigated by SEM (Nova 400 Nano) equipped with EDS (Le350PentaFETx-3). Fifty photos taken randomly by SEM were employed to count the number and size of inclusions by Image Pro-Plus at 2000 magnification. The morphology of segregation areas in ESR ingot and Forged steel was investigated by OM and SEM.

3. Results and Discussion

3.1 Chemical composition

Table 1 shows the chemical composition of electrode and ESR ingot. Comparing the S content in electrode plant with that in ESR ingot plant, there is a significant decrease, which should be attributed to gasifying desulfurization. In air atmosphere, desulfurization process of ESR would happen as Equations (1) and (2) show, and this is gasifying desulfurization mentioned above^[6]. It can be also seen that there are no obviously changes among center, 1/2 radius and surface of the electrode except for the

S content. However, the concentration of sulphur along the transverse of the ESR plant is more homogeneous macroscopically. Thus, the macrosegregation of Sulphur along the transverse of electrode could be solved by electroslag remelting.



N content increased after electroslag remelting which means that the steel was polluted by air. This can also explain the decrease of silicon. It has to be known that the oxidation is detrimental to the desulfurization reaction^[6]. As to the total O content, its average value varied from 46ppm(electrode) to 35ppm(ESR ingot), that should owe to the removing of inclusions by electroslag remelting.

Table.1 Chemical composition of different positions in steel plant of ingot (wt. %).

Electrode	C	Si	Mn	Cr	Mo	V	S	T.O	N
Center	0.39	0.96	0.36	5.24	1.30	1.04	0.0110	0.0044	0.0287
1/2 Radius	0.40	0.97	0.36	5.27	1.33	1.06	0.0093	0.0047	0.0286
Surface	0.40	0.98	0.37	5.31	1.34	1.06	0.0088	0.0047	0.0274
ESR ingot	C	Si	Mn	Cr	Mo	V	S	T.O	N
Center	0.41	0.86	0.36	5.21	1.28	1.06	0.0044	0.0034	0.0315
1/2 Radius	0.40	0.85	0.35	5.16	1.32	1.01	0.0045	0.0035	0.0293
Surface	0.41	0.87	0.37	5.45	1.35	1.08	0.0042	0.0036	0.0295

3.2 Inclusions characteristics and distribution

Figure 2 shows the inclusions in the samples observed by SEM, it can be seen that the size of inclusions are extremely large (Figures 2a, 2b). Inclusions became finer after ESR (Figures 2c, 2d). The number and size were counted, and 50 images with a total area of 0.845mm² were counted for each sample. The average diameter of inclusions measured by Image Pro-Plus is average equal-area-circle diameter. The results are listed in Table 2, about 63.9 percent in number of inclusions were removed, the area fraction (total area of inclusions/total area observed×100%) of inclusions reduced from 0.313% to 0.107%. The effect of electroslag remelting on cleanliness improvement is remarkable.

Table.2 Statistical results of inclusions

Average diameter	Number of observed inclusions				Area fraction of inclusions to total area
	1~2µm	2~3µm	3~5µm	5µm~	
Electrode	189	57	30	29	0. 313%
ESR ingot	52	29	12	17	0. 107%

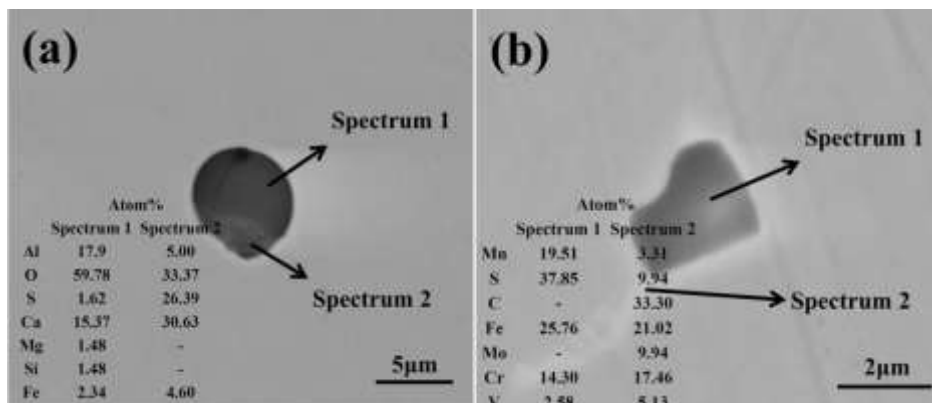


Figure 3. Typical inclusions in electrode sample

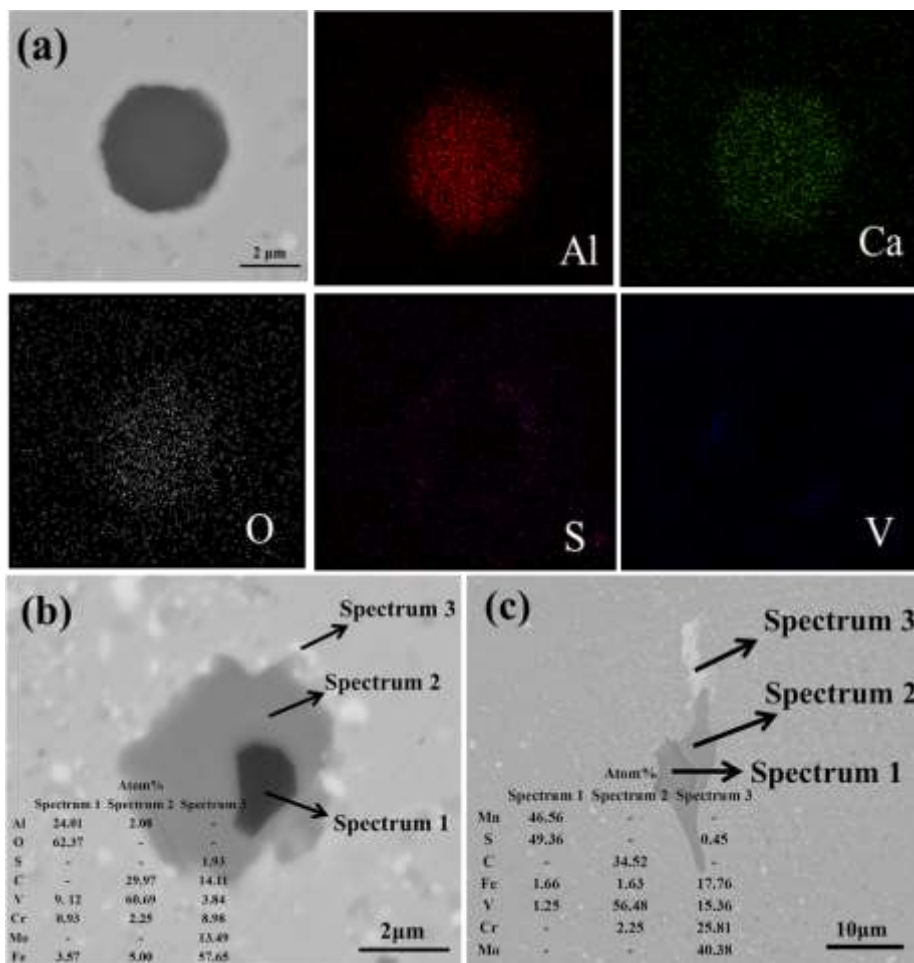


Figure 4. Typical inclusions in ESR ingot sample

Figure 3 shows the typical inclusions in electrode. Figure 3(a) is a complex inclusion with a (Ca-Mg-Al-Si) oxide matrix attached by sulfide. (Ca-Mg-Al-Si) oxide is common inclusion in traditional refining process^[2]. Figure 3(b) shows a sulfide in electrode, the brighter area around sulfide is the primary carbide of Mo and Cr, and this kind of long stripe carbide usually forms at the end of the solidification because of the high content of elements in molten steel. After remelting, complex inclusions like Figure 3(a) disappeared, parts of these were modified into CaO-Al₂O₃ (Figure 4a), and Al₂O₃ can also be seen after ESR.

MnS inclusions would form before primary carbides during solidification^[6]. A solid MnS inclusion is possible to be the nucleation agent of primary carbide, and there are lots of primary carbides with MnS core observed before and after ESR. The disregistry between Al₂O₃ and M(C,N) has been calculated by others^[8]. A larger value of the disregistry mean more supercooling is needed during heterogeneous nucleation, The study shows that Al₂O₃ is effective in promoting nucleation of M(C, N). Thus it can be inferred that both Al₂O₃ and MnS could act the nucleation agent of carbides.

3.3 Microsegregation and banded structure

One of the most important functions of ESR is enhancing the solidification structure. However microsegregation still cannot be solved entirely. Figures 5(a) and 5(b) is the metallographic image of the center part in ESR ingot plant, the sample was etched by 4% nital. Net like structure can be seen in Figure 5(a), in the dark area, much more granular carbides can be observed, and large primary carbides formed in the dark area (Figure 5b). Element map scanning of the segregation area show clearly that the main segregation elements is V, Cr, S and Mo.

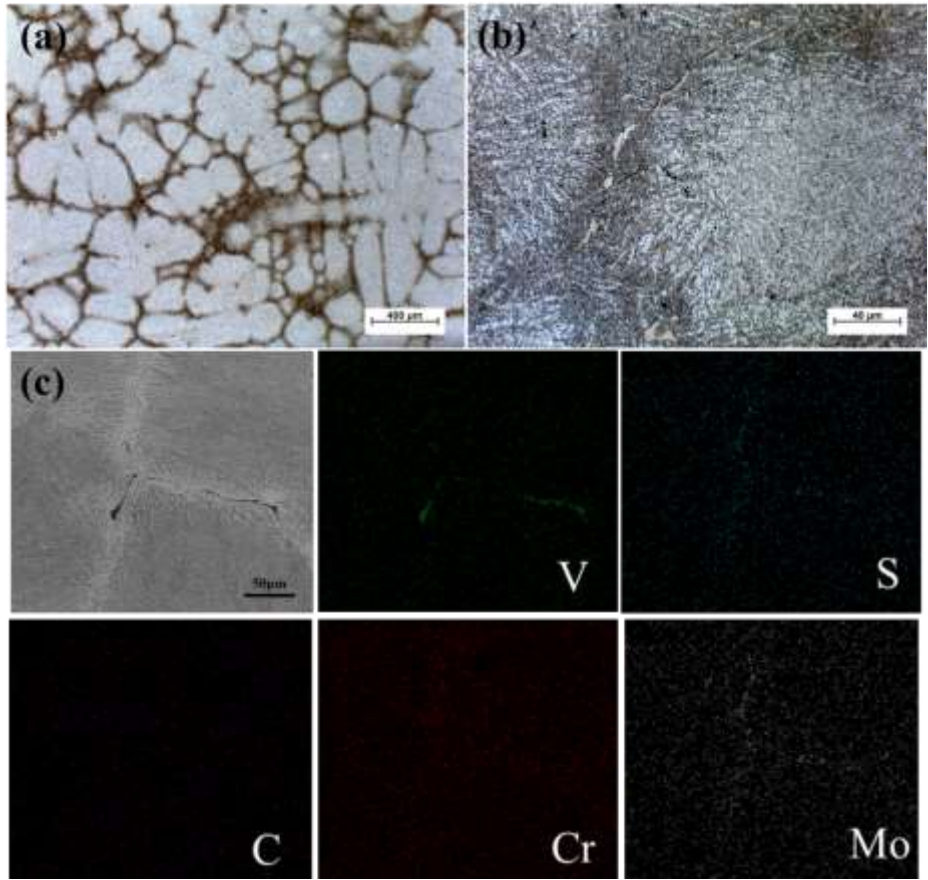


Figure 5. Microsegregation in the ESR ingot

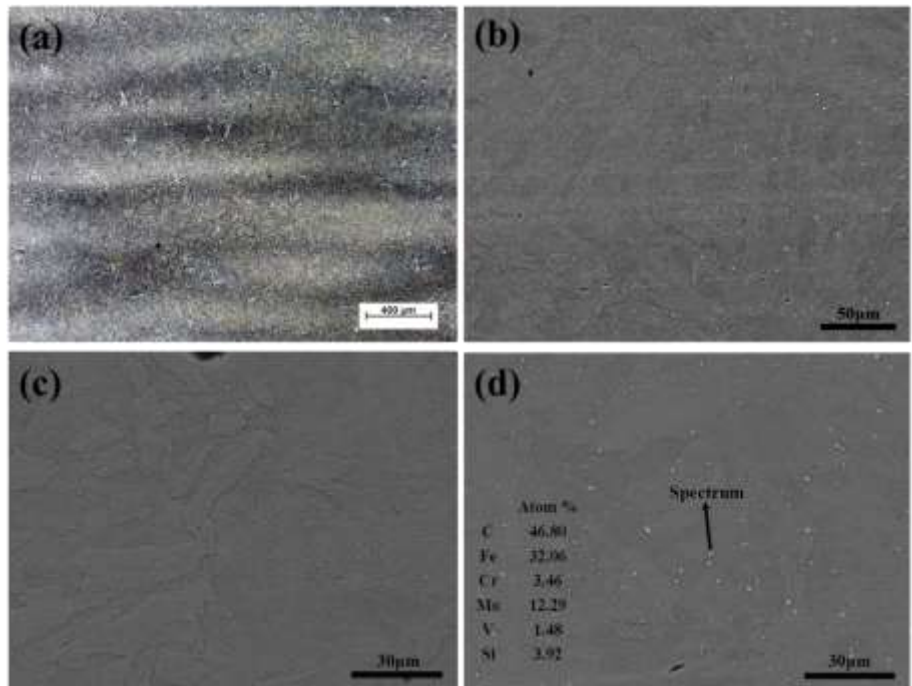


Figure. 6 Banded structure and carbides

The forged ingot sample etched by 4% nital was observed. Banded structure in the sample is showed

in Figure 6(a) taken by OM at 50 magnification. Figure 6(b) was taken by SEM under back-scatter mode, lots of white spots could be observed on the right part of Figure 6(b). The left part (fewer white spots) and right part (more white spots) of Figure 6(b) are shown in Figure 6(c) and Figure 6(d) respectively. The number of the white spots in Figure 6(c) is 15 and in Figure 6(d) is 64. Actually, those white spots are carbides with higher atomic number than the matrix, it can be seen that that kind of carbides are rich in Mo, V, Cr and Si (Figure 6d). The uneven distribution of carbides is the behavior of segregation in forged H13 steel. Mo, V and Cr are rich in both the net like segregation area (as cast ingot) and the white spot (forged ingot), which indicates that the banded structure in forged ingot is related to the microsegregation in as cast ingot.

4. Conclusions

- (1) The S and T.O content, the number, size and area fraction of inclusions decrease owing to the cleanliness improvement of ESR process, N content increase and Si content of ESR ingot decrease because of the influence of air atmosphere. Complex inclusions were modified into CaO-Al₂O₃. Al₂O₃ and MnS can be found in ESR ingot, both of which could promote the nucleation of large carbides.
- (2) The macrosegregation of S content was improved by ESR process, microsegregation and banded structure still can be found. V, Mo, Cr and S are rich in the segregation area in as cast H13 steel, besides, long stripe primary carbide can be also found. Uneven distribution of carbides rich in Mo, V, Cr and Si can be observed in the banded structure.

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