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Evaluation and Modification of Inclusions Characteristics in HK40 Heat-Resistant Cast Steel

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Abstract: In this investigation, specifications of inclusions such as type, morphology, number, composition, size, and place of inclusions in HK40 heat-resistant casting steel were studied by optical microscopy and scanning electron microscopy equipped with energy-dispersive spectroscopy system. Then, the effects of calcium silicide (CaSi) addition on these characteristics were evaluated in two pouring temperatures of 1420°C and 1470°C. Results showed that most of the appeared inclusions were in type of chromium oxide and addition of CaSi had a significant effect on the modification of morphology, size, distribution of inclusions, and changing the composition to oxide-sulfide compounds.

Keywords: Heat-resistant steel, inclusions, Calcium silicide, Modification

1. Introduction

Castings are classified as heat resistant if they are capable of sustained operation while exposed, either continuously or intermittently to operating temperatures that result in metal temperatures in excess of 650°C [1]. Due to maintaining its strength at high temperatures, HK40 (H, K, and 40 stand for heat-resistant steel, Cr and Ni, and carbon content, respectively) or 25Cr20Ni is widely used for stressed parts in structural applications up to 1150°C; offers good resistance to corrosion by hot gases, including sulfur-bearing gases, in both oxidizing and reducing conditions; and is used in air, ammonia, hydrogen, and molten neutral salts. It is also can be applied for tubes and furnace parts [1-2].

Microstructure of HK40 consists of austenite in matrix and carbides distributed in boundaries. Carbides can be solved at high working temperature of these steels, but inclusions as inherent parts of each casting parts will remain even at high temperatures, so it is important to identify inclusions and specify them in these groups of steels [2-4].

The product of oxidation process may be Cr_2O_3 or $FeO{\cdot}Cr_2O_3$ according to chromium activity in molten iron. It was shown that chromite tends to form solid solution phase with other spinels' constituents, that is, the solid solution of $(Mn,Fe)O{\cdot}Cr_2O_3$ will be formed in the presence of manganese [5].

Two major types of inclusions in stainless steel were reported: (1) chromium-rich manganese sulfide and (2) Cr-Mn-Fe-enriched globular oxide inclusions. The sulfide inclusions were found to coexist with oxide inclusion at most of the sites [6].

Due to their high affinity to absorb oxygen, rare earth or alkali metals, which are in the form of either pure or alloyed with silicon, such as calcium silicide (CaSi), are usually used for oxidizing process in steelmaking. They can be added in the form of powder or through a hollow wire into the molten metal [7-10].

Researchers have revealed that high content of nickel in austenitic stainless steel castings can increase the solubility of the calcium in melt, therefore its efficiency is promoted by changing more solid inclusions to liquid inclusions [11-12].

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The aim of this investigation is the identification of common inclusions and their compositions in HK40 steel.

2. Experimental

Melting process was done in a high-frequency induction furnace (inductotherm model) with a capacity of 50 kg. CaSi (70% Ca and 30% Si) was immersed in the melt in ladle. Castings were prepared in standard test coupons (Y-block), which are produced by investment casting method [12]. The casting conditions of specimens and composition of the produced samples are presented in Tables 1 and 2, respectively.

Samples for metallographic observation were etched with a solution containing 10 mL HCl, 10 mL H_2O, and 5 g $FeCl_3$ for microstructural analysis [13]. No etchant solution was used for the investigation of inclusions. Optical microscopy (Olympus, Japan) and scanning electron microscopy (SEM; TESCAN, Czech) equipped with energy-dispersive spectroscopy (EDS) system were applied for surveying the microstructure and chemical composition of inclusions, respectively [14]. Interpretation of optical microscopy's images was carried out by an image analysis software.

Table 1. Sample castings condition

Sample code	Pouring and molding condition
1	Pouring temperature 1420°C without CaSi
2	Pouring temperature 1420°C with CaSi
3	Pouring temperature 1470°C with CaSi

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Table 2. Chemical composition of HK40 steel (wt.%)

C	P	S	Mn	Mo	Si	Cr	Ni	Fe
0.35	0.02	0.005	1.2	0.3	1.6	24.8	20.1	Balance

3. Results and discussion

3.1. As-cast structure

The optical microscopy image of as-cast microstructure of HK40 is shown in Fig. 1. Due to the presence of nickel as an austenite stabilizer element, austenite can be found in the matrix. A small amount of carbide compounds in grain boundaries and intergranular areas were reported in the previous research [15]. Carbide precipitation in interdendritic areas also can be found because of the segregation of carbon and chromium and their high concentration in the mentioned area.

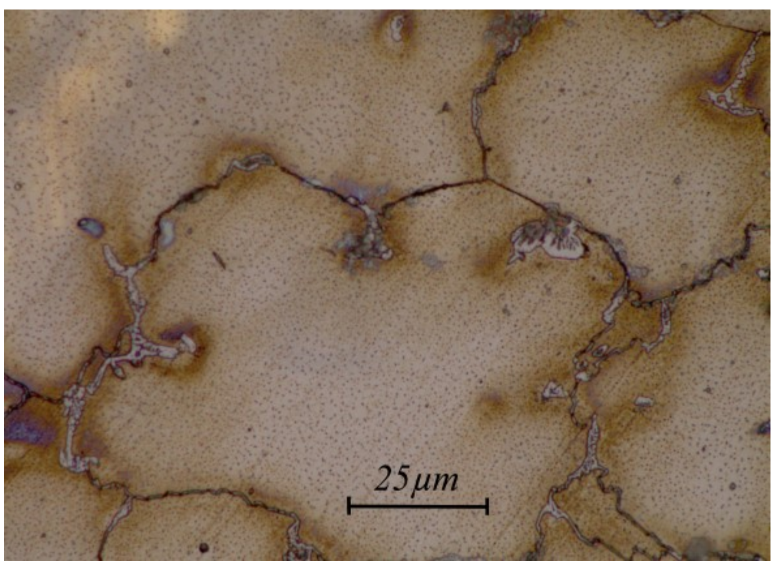

Fig. 1. Microstructure of HK40 steel.

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3.2. Inclusion behavior in the liquid steel

Typical inclusions found in sample 1 are shown in Fig. 2(a). It can be seen that the morphologies of inclusions are sharp, and due to the formation of compounds with high melting point, the coalition of inclusions did not happen completely. Although individual inclusions reached together, they did not have the opportunity to become union.

Fig. 2(b) shows typical inclusions found in sample 2. It should be noted that the coalition of inclusions began, but the complete joining of inclusions cannot occur due to lack of sufficient heat, so the morphology changed to semispherical shape.

Spherical inclusions can be seen in Fig. 2(c) and Fig. 3. The size of inclusions also became smaller in sample 3. Individual inclusions with size smaller than 5 μm can be found in Fig. 2(c). It can be seen that an even distribution of inclusions was made by calcium, which affected chemical composition and morphology modification. Better efficiency of calcium treatment at 1470°C can be found by comparison of Figs. 2(b) and 2(c). At higher temperature and in the presence of CaSi, there is an opportunity of formation of compound with lower melting point along with the changing solid inclusion to liquid inclusion in melt. CaSi addition can decrease the wettability of inclusions in this case too [16].

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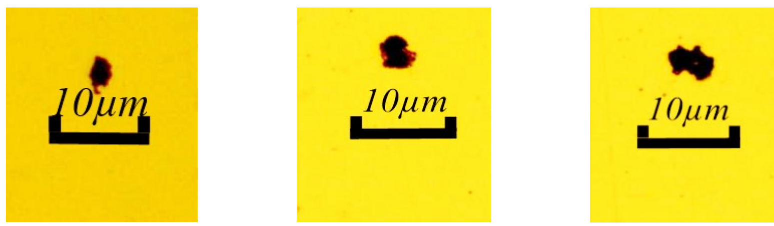
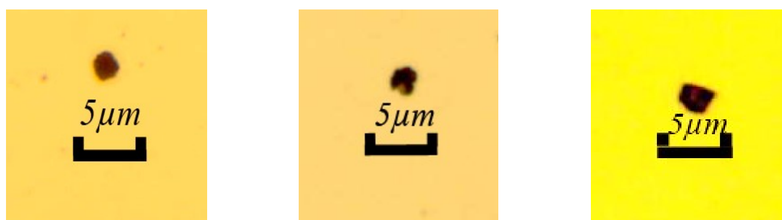
(a) Sample 1



(b) Sample 2



(c) Sample 3

Fig. 2. Typical inclusions found in samples.

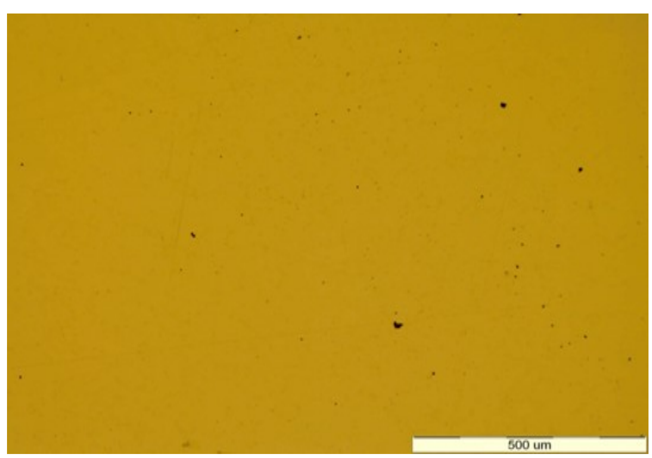


Fig. 3. Optical microscopy image of typical inclusions in sample 3.

Interpretive results (area and diameter) of optical microscopy images of samples, which were extracted by the image analysis software, are summarized in Table 3. It can be found that, at the same scanned area, the maximum area of inclusions was reduced from 694 μm^2 in sample 1 to 309 and 126 μm^2 in samples 2 and 3, respectively, in the presence of CaSi.

The number of inclusions reported was 32 in sample 2. It can be inferred that large inclusions were broken into smaller types as a cause of lowering the melting point through CaSi addition. The number of inclusions in sample 3 is 18. This can be happened through the simultaneous effect of high-temperature liquid treatment and CaSi addition, which made more inclusions to be transformed to liquid inclusion [9-10, 17].

The maximum diameter of inclusions was reduced from 30 μm in untreated state to 20 μm in treated state at 1420°C and 12 μm in treated state at 1470°C as shown in Table 3. It must be noted that we cannot be contented with these numbers exactly, but generally it can be said that CaSi treatment is successful and more effective at 1470°C.

Table 3. Interpretive results of optical microscopy images of samples

Sample	Quantitative charctristics of inclusions		
	Maximum area/ μm^2	Count	Maximum diameter/ μm
1	694	24	29.7
2	309	32	19.9
3	126	18	12.7

The following equation known as Stock's equation shows that segregation of inclusions in molten metal can be done based on their differences in their densities:

$$V = 2r^2 g(\rho_m - \rho_i) / 9\eta \quad (1)$$

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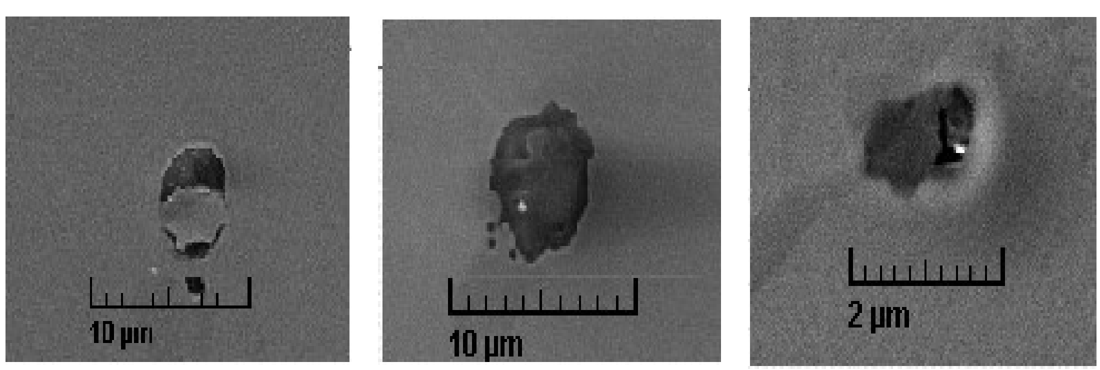
where g is gravity constant (N/kg), V is velocity of solid particle segregation (m/s), r is radius of solid particle (m), ρ_m is density of melt (kg/m^3), ρ_i is density of inclusion (kg/m^3), and η is viscosity of melt (Pa·s).

When calcium is added to molten metal in the form of CaSi or lime, it can make inclusions form new compounds, which are lighter, and at the same time, the viscosity of melt can be reduced [17]. This process will accelerate segregation of inclusions. It can be noted that the formation of oxide films in the layers between inclusion and melt will be effective in decreasing the wettability of inclusions. Uniform distribution of globular and small-size inclusions can promote mechanical properties [18-19].

3.3. Chemical composition of inclusions

SEM images of inclusions and their compositions are shown in Fig. 4. It can be seen that untreated inclusions have sharp and polyhedral morphology. At 1420°C and in the presence of CaSi, inclusions started to join together. This phenomenon can be seen in Fig. 4(b). At 1470°C, the same phenomenon happened followed by reducing in the size of inclusions (Fig. 4(c)).



(a) (b) (c)

Fig. 4. SEM images of typical inclusions in (a) sample 1, (b) sample 2, and (c) sample 3.

Table 4 shows the EDS data of inclusions. It can be noted that oxide inclusions including 80% Al_2O_3, 18% Cr_2O_3, and 2% CaO. Treated inclusions in sample 2 consists of 60% Al_2O_3, 15% Cr_2O_3, and 25% CaO. The appearance of CaO in this sample can be inferred by oxidization process in the presence of CaSi.

Table 4. EDS data of oxide inclusions (at%)

Melt sample	Al_2O_3	Cr_2O_3	CaO	CaS, MnS
1	80	18	2	
2	60	15	25	
3	54	14	26	6

The composition of inclusions in sample 3 was changed to some extent. It consisted of 54% Al_2O_3, 14% Cr_2O_3, and 26% CaO. There is also 6% of sulfide compounds. It can be concluded that oxide and sulfide inclusions appeared in the inclusions of sample 3. These sulfide compounds included CaS and MnS. CaS is 3.5%, whereas MnS can be found 2.5%. The appearance of sulfide compound can be explained by the formation of CaS in the presence of CaSi, which is a stable compound. With attention to higher content of oxide inclusions than sulfide inclusions, it can be concluded that oxide inclusions can be regarded as a place of nucleation for sulfide constituent; therefore, it can be inferred that inclusion composition is oxsy-sulfide (oxide-sulfide) type.

Elemental mapping (elemental analysis) results of inclusions in samples 2 and 3 are shown in Figs. 5 and 6, respectively. Charging chromium in the form of ferrochrome compound to molten metal is common in heat-resistant castings process, so they can be

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regarded as source of oxide inclusions. Figs. 5 and 6 indicated that chromium oxides existed in inclusions.

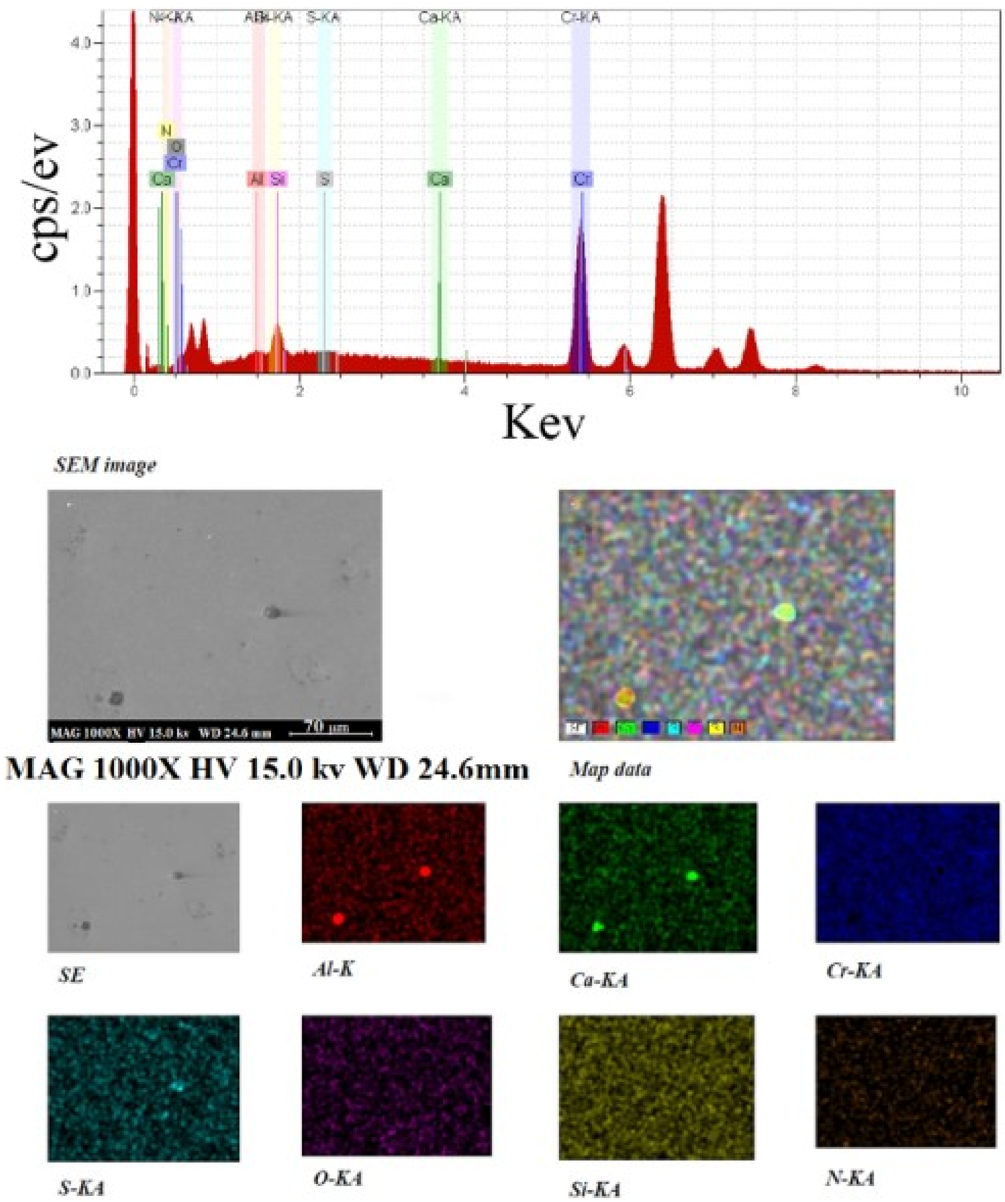

Fig. 5. Elemental map analysis of typical inclusions in sample 2.

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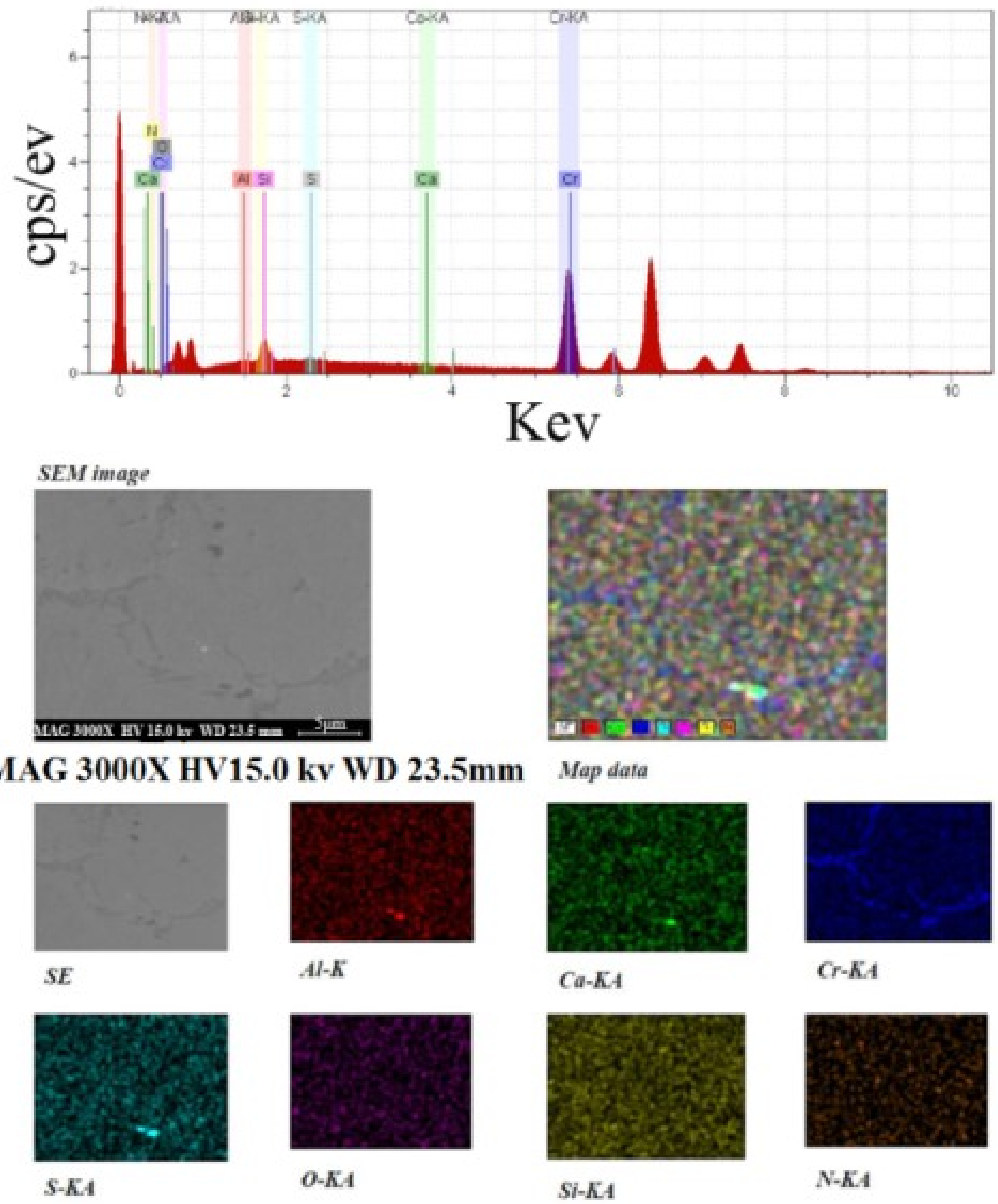

Fig. 6. Elemental map analysis of typical inclusions in sample 3.

Figs. 5 and 6 also confirmed that oxide inclusions' composition including $Al_2O_3{\cdot}Cr_2O_3$, which is mentioned in Fig. 4 too. Hence, the morphology of inclusions changed from sharp state to globular shape by the addition of CaSi into molten metal, whereas stable sulfide-type inclusions were also formed in the vicinity of oxide inclusions.

4. Conclusions

Final conclusions obtained from this investigation can be mentioned as below:

(1) Appeared inclusions in HK40 heat-resistant steel were based on alumina-chromite with nonuniform distribution.

(2) Shape, size, and composition of inclusions were changed to oxy-sulfide with uniform distribution, smaller size, and globular shape by CaSi treatment process of HK40 melt.

(3) CaSi treatment was more effective at 1470°C melt temperature.

(4) Maximum area and maximum diameter of inclusions can be reduced to 80% and 60%, respectively, by CaSi treatment.

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