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Precipitation Behaviors of Carbides in High Speed Steel during ESR and Heat Treatment

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Abstract: The microstructure and carbides evolution of high-speed steel after electroslag remelting and solution treatment were studied. The thermodynamic precipitation mechanism of carbides in solid phase was discussed and the characteristic parameters of carbides in different processes were also investigated. The results show that there were large lamellar and fibrous Mo₂C and a small amount of VC in the ESR ingot. Mo₂C are metastable carbides, which can be decomposed into VC and Fe₂Mo₄C during the solution treatment. The average diameter of the carbides is reduced to 1.28 μm and the space distance is reduced to 3.23 μm after forging and hot rolling, which means carbides are completely spheroidal and dispersed in matrix.

Keywords: electroslag remelting; carbide; high speed steel; solution treatment; microstructure

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1. Introduction

High-speed steels (HSS) are widely used in making high-speed cutting tools, which always require high hardness, good wear resistance, and good thermal fatigue resistance at elevated temperatures [1,2]. This kind of wear-resistant and heat-resistant tool steel with secondary hardening characteristics contains a large amount of tungsten, molybdenum, vanadium, chromium, and other alloy elements [3–5].

It is well known that the type, morphology, size, and distribution of carbides have great influence on the mechanical properties of high-speed steel, which are closely related to the as-cast structure of ingots, especially eutectic carbides. M₂C is a typical carbide in W-Mo high speed steel [6]. M₂C is easy to decompose into stable carbides with reaction $M_2C + Fe(\gamma) \rightarrow M_6C + MC$, therefore, it is easy to decompose when heated [7]. High speed steel contains a large amount of reticulated eutectic carbides, which are brittle and easy to crack during forging, which will cause serious segregation and affect its service performance [8]. It has been reported that the presence in carbide distribution of significant fraction of ultrafine particles can improve wear resistance of high-speed steel by 2.13 times compared with the conventional treatment while hardness increases by 7.76% and wear rate decreases [9–11]. It is significant to improve the morphology and distribution of eutectic carbides in high-speed steel.

The evolution of microstructure and carbides in high-speed steel is strongly influenced by the obtainment process. Ideally, depending on the application, some best mechanical properties are expected when the microstructure showed a homogeneous distribution of the carbides in the matrix, but the achievement of this microstructure is very difficult because the carbide formation occurs in several stages of the obtainment process.

The electroslag remelting (ESR) process is usually used to improve the solidification structure, cleanliness, and transverse mechanical properties of high-speed steel [12,13]. It was reported that the consumable electrode and rotation of a mold, respectively, in ESR process can not only reduce the size and alleviate the segregation of carbides in HSS ingot, but also improve the surface quality of ingots and reduce inclusions [14,15]. However, the alloying elements are easy to segregate seriously and form large eutectic carbides in the manufacturing process, which is difficult to eliminate in the subsequent forging and other heat treatment processes [16]. Therefore, continuous directional solidification of electroslag remelting (ESR-CDS) is considered as a widely used secondary refining technology for producing high quality HSS. Li et al. [17] and Fu et al. [18] demonstrate that ESR-CDS could effectively eliminate macro-segregation in as-cast ingot through the shallow molten metal pool controlled by directional solidification.

The present work aims to study the effect of heat treatment and hot deformation on morphology, size, and distribution of carbides in high speed steel after ESR-CDS process. By improving process parameters, thick eutectic carbide can be eliminated, and carbide shape and product performance can be eventually improved.

2. Materials and Methods

Casted HSS used in the present study was melted with pure alloy ingredients in a vacuum induction furnace (Huairou, Beijing, China) with capacity of 50 kg. The liquid steel was cast in a mold, and then forged into electrode with a diameter of 120 mm. Then the electrode was electroslag remelted to produce ingot of 160 mm diameter through ESR-CDS. The remelting process was conducted in the atmosphere with blowing argon at the top of the crystallizer. The chemical composition of the steel is shown in Table 1.

Table 1. Chemical composition of steel (wt%).

C	W	Mo	Cr	V	Si	Mn	Y	Fe
1.2	3.5	8.2	3.9	2.8	0.62	0.3	0.03	Bal.

The experimental process includes heat treatment, forging, and hot rolling. The ESR ingot was held at 1100 °C for 24 h and then forged to bars. The forging start temperatures were controlled in the range of 1060–1100 °C and the forging finish temperatures were controlled to be higher than 910 °C while the forging ratio was 4. Thereafter, the steel was rolled into Φ11 mm by rolling mill. The specimens of ESR ingot were subjected to the solution treatment at the temperatures of 1080 °C and 1130 °C for 1 h, 2 h, and 4 h. The scheme is shown in Table 2 and Figure 1. They were heat treated in an electric resistance furnace, and quenched in water.

Table 2. Solution treatment process.

Specimen Number	Solution Treatment Temperature	Solution Treatment Time
1080-1	1080 °C	1 h
1080-2		2 h
1080-4		4 h
1130-1	1130 °C	1 h
1130-2		2 h
1130-4		4 h

Figure 1. Experimental process for the solution treatment: (a) solution treatment temperature; (b) solution treatment time.

The samples with the dimension of 15 mm × 15 mm × 12 mm were taken from as-cast and ESR ingots, respectively. The microstructure of the samples was observed by optical microscope (Leica DM4M, Leica Microsystems, Wetzlar, Germany, OM), and carbides were characterized by scanning electron microscope (FEI MLA250, FEI, Hillsboro, OR, USA, SEM) equipped with energy-dispersive X-ray spectrometer (XFlash 5030, Bruker, Karlsruhe, Germany, EDS), after grinding, polishing, and etching with 4% nitric acid in alcohol. The chemistry of various precipitates identification at different temperatures and phase equilibrium investigation were calculated by Thermo-calc software (Thermo-calc Software Inc., Solna, Sweden). The samples taken from HSS were machined into a rod of Ø15 mm × 90 mm. Carbides were extracted from steel matrix in organic solution (methanol, tetramethylammonium chloride, glycerin, diethanol amine) by 90. Some were analyzed by X-ray diffraction (Rigaku Dmax-RB, Rigaku, Tokyo, Japan, XRD) to confirm the types, and others were observed by SEM for the three-dimensional morphology.

3. Results and Discussion

3.1. Microstructure of As-Cast and ESR Ingot

The solidification structure and microstructure of as-cast and ESR ingot are shown in Figures 2 and 3, partial primary carbides precipitated along austenite grain boundaries and eutectic ledeburite formed network. The matrix structure in the HSS as-cast ingot was extremely uneven in both morphology and size. The primary and secondary dendrites were coarse, and some relatively coarse tertiary dendrites appeared. The eutectic ledeburite was in the shape of uneven flakes, and the segregation of the ingot was relatively serious. The structure obtained by the ESR-CDS process was relatively uniform and dense, mainly composed of fine dendrites. There was a large amount of white matrix around the eutectic ledeburite. The ledeburite was small, and some of which were short rods, which was more beneficial to the structure and performance of the material in the subsequent deformation processing process [19]. It can be obtained from the above analysis that the level of solidification and shrinkage greatly reduced after the ESR-CDS process.

Figure 2. Cont.

Figure 2. OM images of HSS solidification structure: (a,b) As-cast ingot; (c,d) ESR ingot.

Figure 3. SEM images of HSS solidification structure: (a,b) As-cast ingot; (c,d) ESR ingot.

The morphology of MC carbide shown in Figure 4 was short rod-shaped, while M_2C carbide was disc-shaped with multi-angles. MC carbides precipitated along pre-existed austenitic grain boundaries [20]. The morphology of MC carbide was influenced by local concentration of solute atoms and pre-formed austenite dendritic interface, as well as the solidification rate [21]. It can be confirmed from the EDS results in Figure 5 and XRD analysis in Figure 6 that M_2C is Mo_2C and MC is VC.

Figure 4. Two-dimensional morphology (a) and three-dimensional morphology (b) of carbides.

Figure 5. EDS analyzed results of M_2C (a) and MC (b) carbides.

Figure 6. XRD pattern of HSS at different modes: (a) As-cast; (b) ESR.

3.2. Phases Formation Calculated with Thermo-Calc Software

The equilibrium formation of precipitates in the studied steel was investigated using Thermo-Calc software (TCFE7 database). As shown in Figure 7, five different types of carbides, i.e., M_6C , MC , M_2C , M_7C_3 , and $M_{23}C_6$, precipitated in sequence with decreasing temperature, in high-speed steel under the conditions of equilibrium solidification and cooling. (M represents metallic element atom, C represents carbon atom.) The transformation (precipitation) temperature of precipitates in the investigated steel are listed in Table 3. The temperature of precipitated M_6C , MC , M_2C , M_7C_3 , and $M_{23}C_6$ in sequence during equilibrium solidification condition were 1290 °C, 1250 °C, 1000 °C, 826 °C, and 824 °C, respectively.

Figure 7. The phase equilibrium and transformation during solidification of liquid steel.

Table 3. Transformation temperature of precipitates in steel, calculated by Thermo-Calc.

$T_s / ^\circ\text{C}$					$T_f / ^\circ\text{C}$	
M_6C	MC	M_2C	M_7C_3	$M_{23}C_6$	M_2C	M_7C_3
1290	1250	1000	826	824	920	824

T_s represents phase transformation starting temperature; T_f represents phase transformation finish temperature.

In practical solidification process, the carbides forming elements with solute partition coefficients (k) smaller than 1 would segregate at the solid/liquid interface, which would lead to eutectic reaction in the inter-dendritic regions [19]. To predict phase precipitation during liquid steel solidification in a practical ESR refining process, the Scheil–Gulliver model included in Thermo-Calc software was employed to calculate the non-equilibrium phase precipitation in high speed steel, as shown in Figure 8. MC, M_6C , M_2C , and M_7C_3 carbides precipitated from liquid steel directly in non-equilibrium condition; reactions, phase transformation starting time, and solid fraction are listed in Table 4. However, M_6C and M_7C_3 carbides cannot be found in microstructure, which may be attributed to rapid solidification resulting to precipitation of carbides from austenite phase scarcely [22]. Thus, the microstructure consists of austenite, eutectic Mo_2C , and VC carbides.

Figure 8. Non-equilibrium phase diagram in HSS.

Table 4. Calculation results of non-equilibrium phase precipitation using Thermo-Calc.

Reaction	$T_s/^{\circ}\text{C}$	f_s
$\text{L} \rightarrow \gamma\text{-Fe} + \text{MC}$	1287	0.4
$\text{L} \rightarrow \gamma\text{-Fe} + \text{MC} + \text{M}_6\text{C}$	1245	0.65
$\text{L} \rightarrow \gamma\text{-Fe} + \text{MC} + \text{M}_6\text{C} + \text{M}_2\text{C}$	1219	0.84
$\text{L} \rightarrow \gamma\text{-Fe} + \text{MC} + \text{M}_6\text{C} + \text{M}_2\text{C} + \text{M}_7\text{C}_3$	1180	0.9

T_s represents phase transformation starting temperature; f_s represents solid fraction of liquid steel.

3.3. Effect of Heat Treatment on Carbide Evolution

The microstructure of forging and hot rolling HSS are shown in Figure 9. When the ESR ingot was forged, the grain was stretched and refined. Most of the grain size was between 10–20 μm and the grain was in lath shape. The carbides network near the grain boundary was broken and distributed in a certain direction under the action of forging pressure. Fibrous M_2C carbides were completely decomposed. M_6C carbides were also broken, and became ellipsoidal finer carbides. However, the carbides were not completely disconnected from each other. The shape and size of MC carbides do not change significantly. After hot rolling, the microstructure of HSS had been further refined, the grains were deformed. The carbides were almost completely spherical and were broken into smaller individual particles dispersed in the matrix. The carbide size was basically below 5 μm and the distribution of carbides was homogeneous. There were a lot of dispersed fine carbides in the matrix and the size was 1 μm or even smaller, which strengthened the matrix.

Figure 9. The microstructure of HSS after forging (a,b) and hot rolling (c,d).

Figure 10 shows the XRD pattern of HSS after solution treatment, and the effect of solution treatment temperature and solution treatment time on microstructure and carbide evolution are shown in Figure 11. M_2C is easy to decompose upon heating and decomposed into stable carbides through $\text{M}_2\text{C} + \gamma\text{-Fe} \rightarrow \text{M}_6\text{C} + \text{MC}$ and $\text{M}_2\text{C} + \gamma\text{-Fe} \rightarrow \text{M}_6\text{C} + \text{M}_7\text{C}_3 + \text{MC}$ [23]. Therefore, there were retained lamellar Mo_2C , white rod-like $\text{Fe}_2\text{Mo}_4\text{C}$ and

grey spherical VC as shown in Figure 11. Cr_7C_3 carbides were fine granular shape, which was hard to find in the microstructure. With the solution treatment time increasing, the disconnection of carbides was gradually obvious, while the layers of Mo_2C were also bent and deformed and partial carbides were spheroidized. When solution treatment time was at 4 h, most of the carbides have been spheroidized and transformed into blocks or spheres. The size of the carbides was reduced, and the distribution of the carbides was more homogeneous. Due to the continuous diffusion of alloying elements, the partial carbides have aggregated and grown, but the network structure of carbides still existed. The decomposition rate of M_2C carbide was the fastest at 1100°C [23]. Compared with the carbide size and distribution of solution treatment temperature at 1130°C , solution treatment effect was better at the solution treatment temperature of 1080°C .

Figure 10. XRD pattern of HSS after solution treatment.

Figure 11. Effect of solution treatment temperature and solution treatment time on microstructure and carbide evolution: (a) 1080-1; (b) 1080-2; (c) 1080-4; (d) 1130-1; (e) 1130-2; (f) 1130-4.

Carbides solubility product in austenite can be calculated as follows [24]:

$$\ln[w([V]) \cdot w([C])] = 9.59 - \frac{14,828}{T} \quad (1)$$

$$\ln[w([Cr])^7 \cdot w([C])^3] = 50.76 - \frac{51,516}{T} \quad (2)$$

$$\ln[w([Mo])^2 \cdot w([C])] = 17.35 - \frac{17,788}{T} \quad (3)$$

The relationship of equilibrium solubility product and actual solubility product was shown in Equation (4). $Q_{M_xN_y}^{\circ}$ is equilibrium solubility product and $Q_{M_xN_y}$ is actual solubility product. When $\Delta G < 0$, which means $Q_{M_xN_y}^{\circ} < Q_{M_xN_y}$, the reaction proceeds in the direction of the formation of precipitates. The solubility product of carbides precipitated from the solid phase during the solidification process is shown in Figure 12. Both VC and Mo_2C can be precipitated from the solid phase when the cooling progresses to the solid phase temperature. However, Cr_7C_3 begins to precipitate when the temperature drops to 1115 K (842 °C), which is lower than the solution treatment temperature [24]. Due to the precipitation temperature being low, the time for nucleation and growth is short, Cr_7C_3 carbides exist in the steel in the form of small particles. When the cooling rate is fast, they will not be precipitated.

$$\Delta G = \Delta G^{\theta} + RT \ln \frac{1}{Q_{M_xN_y}} = RT \ln \frac{Q_{M_xN_y}^{\circ}}{Q_{M_xN_y}} \quad (4)$$

Figure 12. Curves of equilibrium solubility product and actual solubility product to temperature of solid HSS.

The basic parameters and microstructure characteristic parameters of carbides were calculated and analyzed by image analysis software. The characteristic parameters include the volume fraction of carbide V_V , the number of carbides per unit volume N_V , the average diameter of carbides $\bar{D}$, and the distance between carbides t_0 . The relation between them is shown in detail in Table 5. A_A is the area fraction of carbide, and N_A is the total number of carbides in the area. In this study, the following parameters could be obtained by software: the number of carbides N_A , the area of carbides A , the average diameter of carbides D , as shown in Table 6. The characteristic parameter of all kinds of carbides in the steel can be calculated based on the microstructure characteristic parameters in Table 5 and the basic parameter values in Table 6. The calculated results are also shown in Table 6.

Table 5. The basic relation between the microstructure characteristic parameters.

Relation Formula	Unit	Spatial Characteristic Parameter
$V_V = A_A$	%	Volume fraction of carbide
$N_V = N_A / \bar{D}$	—	Number of carbides in per unit volume
$t_0 = \sqrt[3]{1/N_V}$	μm	Space distance of carbide

Table 6. Basic parameters and characteristic parameters of carbides.

Status	Basic Parameters			Characteristic Parameters		
	N_A	$A/\mu\text{m}^2$	$\bar{D}/\mu\text{m}$	$V_V/\%$	N_V	$t_0/\mu\text{m}$
ESR	1671	5491	3.84	7.06	5.60×10^{-3}	13.37
Hot-rolling	2334	1817	1.28	9.54	9.57×10^{-2}	3.23
Solution treatment	522	7706	5.03	9.90	1.33×10^{-3}	27.39

Comparing the characteristic parameters of carbides in different process samples, it can be found that the amount of carbides decreased after the solution treatment, while the volume fraction increased. Comparison between Figures 3 and 11 shows that, the lamellar and fibrous M_2C carbides were decomposed into M_6C and MC . M_6C grew into short rod, resulting in the reduction of the amount of carbides and the slight addition of the carbides size. Although the volume fraction of carbides increased from 7.06% to 9.9%, the number of carbide particles per unit volume decreased. The average diameters of carbides were 3.84 and 5.03, respectively, in the ESR sample and the solution treatment sample. After hot rolling, the average diameter of carbides has been reduced to 1.28 μm and the volume fraction varies little, from 7.06% to 9.54%. However, the carbides particle number increased a lot, from 1671 to 2334, and the distance between carbides particles was reduced from 13.37 to 3.23. Therefore, the carbides in hot-rolled high-speed steel were more dispersed in the matrix.

4. Conclusions

1. The carbides of high-speed steel after electroslag remelting are of a large size and mainly lamellar and fibrous precipitates along the grain boundary. After solution treatment, the size of carbides is obviously reduced and the distribution of carbides are uniform, and the morphology of carbides are mainly short-rod, irregular spherical, and fine particles.
2. The carbide types both in as-cast and ESR high-speed steel are metastable carbides Mo_2C and VC . The types of carbides after solution treatment, forging, and hot rolling are mainly small composite carbides containing molybdenum, vanadium, and chromium, including Mo_6C , VC , and Cr_7C_3 . Mo_2C carbides in ESR ingot can be decomposed into VC and Fe_2Mo_4C during the solution treatment.
3. After forging and hot rolling, carbides are completely spheroidal and dispersed in the matrix. The average diameter of the carbides can be reduced to 1.28 μm and the space distance reduced to 3.23 μm . A uniform distribution of spheroidal shaped carbides was achieved.

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