Influence of Heat Treatment on Content of the Carbide Phases in the Microstructure of High-Speed Steel

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Abstract

This article presents the results of investigations of the effect of heat treatment temperature on the content of the carbide phase of HS3-1-2 and HS6-5-2 low-alloy high-speed steel. Analysis of the phase composition of carbides is carried out using the diffraction method. It is determined that with increasing austenitising temperature, the intensification of dissolution of M_6C carbide increases. As a result, an increase in the grain size of the austenite and the amount of retained austenite causes a significant reduction in the hardness of hardened steel HS3-1-2 to be observed. The results of diffraction investigations showed that M_7C_3 carbides containing mainly Cr and Fe carbides and M_6C carbides containing mainly Mo and W carbides are dissolved during austenitisation. During austenitisation of HS3-1-2 steel, the silicon is transferred from the matrix to carbides, thus replacing carbide-forming elements. An increase in a degree of tempering leads to intensification of carbide separation and this process reduce the grindability of tested steels.

Keywords: Heat treatment, Metallography, Carbide phase, High-speed steel, Microstructure

1. Introduction

High-speed steel (HSS) is complex iron-based alloy containing carbon, tungsten, molybdenum, vanadium, chromium and substantial amounts of cobalt [1]. To ensure applicable quality requirements during production of tools made of HSSs, it is very important that their cutting ability is not lower than comparable tools made of classic high-speed steels [2].

In the case of cutting tools, the maximum efficiency is determined mainly by hardness, strength and temperability of the tool material [3, 4]. These properties of the tool material can be adjusted within a relatively wide range by the parameters of the heat treatment [5]. Optimum heat treatment parameters ensure that the highest hardness, strength and temperability are achieved at the same time [6]. Determination of the optimum austenitising temperature requires special attention because it regulates a

number of important processes. On the one hand, with an increase in austenitising temperature higher dissolution of secondary carbides is observed, which increases temperability and strength. On the other hand when steel material is overheated to too high a temperature an excessive growth of grains is observed, which increases the amount of retained austenite. However, in the case of smaller carbon content in steel, ferrite can be formed or even part melting of the grain boundaries can occur. HSSs should be used in a state of high hardness, which involves the determination of the optimum temperatures of austenitising and tempering [2, 7-9]. Austenitising temperature of a tool steel set in the process of tool manufacture should produce a grain size determined by the Snyder-Graff of no less than 10, a bending strength of 2500–3000 MPa and a hardness of about 63 HRC.

Microstructural changes occurring in tool during grinding process depend mainly on the chemical composition of material,

thermal treatment and grinding parameters. The investigations of the effect of heat treatment on the coefficient of grinding [1] showed that this coefficient depends more on the fold of tempering than on the austenitization temperature. Both of these parameters determine the number and size of carbide phases leading to a decrease in the coefficient of grinding. The aim of this research is phase analysis and determination of the content of elements in carbide phases in HS3-1-2 and HS6-5-2 steels subjected to different heat treatment conditions.

2. Methods

Analysis of the phase composition of carbides is carried out using the diffraction method. The research material was prepared in the form of isolates containing carbon bound into carbides and unbounded carbon in solid solution. These isolates were obtained by electrolytic dissolution of samples. Conditions of electrolysis and the chemical composition of the electrolyte are shown in Table 1.

The isolate was removed from the sample by ultrasonic method. Then the isolate was washed in a centrifuge and dried in an evaporating dish at 105°C. Isolates were tested using the diffractometer. Copper and chromium lamps with a nickel filter were used. The wavelength of CuKα radiation was λ = 0.229092 nm. An analysis of the phase composition was performed for both grades of steel after annealing, hardening and tempering at 620°C for 4 hours. The HS3-1-2 steel was hardened at temperatures of 1080°C, 1120°C and 1150°C. The HS6-5-2 steel was hardened at a temperature of 1200°C and tempered at 550°C for 2 hours. The grain size was determined using Snyder-Graff (S-G) intercept method.

Determination of element contents in carbides was carried out via atomic absorption spectrometry (AAS), using a Perkin-Elmer model 503 atomic absorption spectrometer. The AAS method was used to determine the content of Fe, Cr, Mn, W, V, Mn and Si in the HS3-1-2 steel after the heat treatment processes. Isolates were dissolved in a solution of HNO_3 concentrated by 4% solution of NaF in the proportion 1:1, with later addition of hydrofluoric acid. The resulting percentages of elements in the isolate x were converted to percentages in carbides y according to the relationship:

$$a = \frac{x}{b} \cdot 100\% \qquad (1)$$

$$y = \frac{x}{B} \cdot 100\% \qquad (2)$$

where :
x – the content of the element in isolate determined by AAS method (%),
y – the content of the element in carbides (%),
a – the content of the element in isolate (%),
b – isolate mass (g),
B – mass of the specimen part dissoluted electrolytically (g).

3. Results and discussion

The results of the phase analysis for tested steels according to the austenitising temperature are shown in Table 2. A significant dissolution of the M_7C_3 and M_6C carbides in HS3-1-2 at austenitising temperatures of 1120°C and 1150°C was observed, while no visible dissolution of the MC carbide at these temperatures was observed. Simultaneously, no M_2C primary carbide, besides MC carbide in HS3-1-2 steel, was revealed. The M_2C carbide did not occur in the microstructure in spite of the high carbon content in the HS3-1-2 steel, as this steel was additionally enriched by nitrogen. The phase analysis of HS6-5-2 steel revealed the dissolution of $M_{23}C_6$ type carbide at temperatures below 1210°C. The microstructure of tested steels is composed of alloying ferrite and carbides.

The composition of the carbide phase is similar after all heat treatment operations. Diffraction studies did not reveal significant differences between the carbide phases in hardened steel and carbide phases after tempering. This is due to two factors:
- the intensity of the deflected diffracted X-ray line for the family of planes affects not only the volume fraction of the specific phase in the solution but also has an influence on factors such as the chemical composition of the phases and the phase texturing.
- carbide precipitates are very small, so they can be revealed by the diffraction pattern.

It is known that when heating the steel for tempering, first M_7C_3 carbides are dissolved and M_6C and MC carbides inhibit the grain growth. M_6C carbide dissolution begins to intensify with an increase in austenitising temperature and as a result the size of austenite grains (Fig. 1) and the amount of residual austenite (Table 3) increase, causing a significant reduction in the hardness of HS3-1-2 hardened steel.

The grains in the samples hardened at a temperature of 1120°C are greater than in the samples hardened at 1160°C (Fig. 3), despite having the same annealing time and batch size.

Table 1.
Electrolysis conditions and chemical composition of the electrolyte

Steel grade	Composition of the electrolyte	Current density, A/cm²	Time of electrolysis, min.
HS3-1-2	150 g KCl + 10 g of citric acid + 15 ml of HCL per 1 dm³ of H_2O	30	8
HS6-5-2	1150 ml CH_3OH + 15 g of citric acid + 50 ml of HCL per 1 dm³ of H_2O	3	90

Table 2.
Results of phase analysis

Steel grade	Heat treatment	Occuring carbides	Comments
HS6-5-2	soft annealing	M_6C, M_7C_3, MC, $M_{23}C_6$	M_7C_3 – trace amount
	austenitization at 1210°C	M_6C, MC	isotale contain very small particles of martensite
	austenitization at 1210°C + tempering at 550°C for 2 hours	M_6C, MC	lack of martensite
	austenitization at 1210°C + tempering at 550°C (2 hours) + tempering at 620°C (4 hours)	M_6C, MC	
HS3-1-2	soft annealing	M_6C, MC, M_7C_3	
	austenitization at 1080°C	M_6C, MC, M_7C_3	
	austenitization at 1120°C	M_6C, MC, M_7C_3	significant reduction of content of carbides M_6C and M_7C_3 compared to austenitization at 1180°C is observed
	austenitization at 1150°C	M_6C, MC, M_7C_3	
	austenitization at 1120°C + tempering at 550°C (2 hours)	M_6C, MC, M_7C_3	based on the data the variation of fraction of individual phases in isolate cannot be determined
	austenitization at 1120°C + tempering at 550°C (2 hours) + tempering at 620°C (4 hours)	M_6C, MC, M_7C_3	

Table 3.
Physico-mechanical properties of HS3-1-2 steel

Austenitizing temperature, °C	Hardness after hardening HRC	Grain size	After twice tempering (550°C, 2 hours)		After triply tempering (550°C, 1 hour)	
			Hardness HRC	Retained austenite, %	Hardness HRC	Retained austenite, %
1080	61	13	61	15	62	-
1100	61	12-13	62	16.5	62	-
1120	60	11-12	62	18	62.5	2
1140	59	10-11	63	19.5	63	4
1160	57	8	60	21	62	14.5
1180	53	7	55	30	61.5	21

Table 4.
The chemical composition of carbide isolate in HS3-1-2

Heat treatment of isolate	Chemical composition in isolate, % wt.						
	Fe	Mn	Cr	W	Mo	V	Si
soft annealing	33.79	0.65	20.54	13.25	5.28	8.12	0.83
austenitization at 1080°C	21.33	0.31	13.21	11.75	4.08	9.75	5.84
austenitization at 1120°C	11.16	0.12	6.34	9.84	3.75	9.84	9.26
austenitization at 1150°C	5.13	0.05	3.68	8.43	2.97	9.74	8.65
austenitization at 1120°C + tempering at 550°C for 2 hours	15.24	0.18	9.36	13.15	4.27	9.40	5.86
austenitization at 1120°C + tempering at 550°C for 2 hours + tempering at 620°C for 4 hours	88.64	0.76	16.54	12.14	4.57	9.27	1.01

After the third tempering the hardness of the steel significantly increases (Table 4) as a result of the evolution of a large number of carbides. As the sizes of these carbides are several times smaller than the primary carbides, their total area is significantly higher, which can result in a reduction of the grindability of the tested steels.

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several times smaller than the primary carbides, their total area is significantly higher, which can result in a reduction of the grindability of the tested steels. The results of the analysis of elements content in the carbide isolates in HS3-1-2 steel are shown in Table 4. During the austenitisation, dissolution of both the M_7C_3 carbides containing Cr and Fe and the M_6C carbides containing mainly Mo and W is observed.
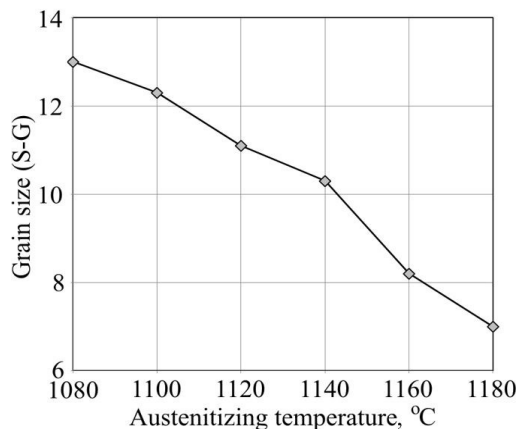

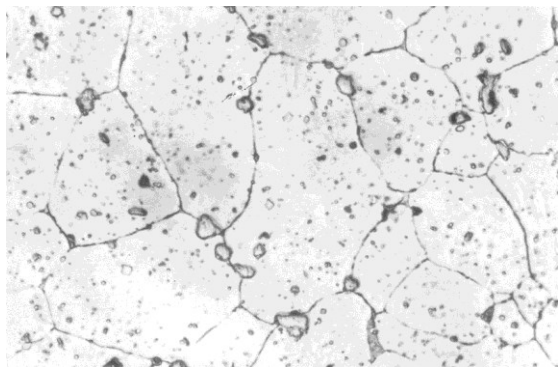Fig. 1. The effect of austenitising temperature on the size of the austenite grain

Fig. 2. Microstructure of the HS3-1-2 steel austenitized at 1160°C; etching using reagent of 8% HNO_3; magnification 500×

This is related to the transition of Cr, Fe, Mo and W carbides into the matrix. Vanadium is an element that transfers to matrix in a very small amount, which indicates no dissolution of the MC carbide. An increase in the content of elements in carbides after tempering is proof that the processes of separation or growth of carbides occurred. Of particular importance in this context is the change in silicon content in the steel during heat treatment processes (Table 4). The percentage of silicon increases with an increase in austenitising temperature. However, the percentage of silicon carbide decreases during the tempering. Thus, it should be concluded that during austenitisation the silicon is transferred into the carbides. Silicon is incorporated into the network of the M_6C carbide, replacing the atoms of elements such as Cr, Fe, Mn, Mo, V and W.

4. Conclusions

From the experimental results, the following conclusions can be drawn:
1. An increase in austenitising temperature intensifies the dissolution of M_6C carbide causing an increase in the austenite grain size.
2. During austenitization the process of dissolution of M_7C_3 and M_6C carbides occurs and, as a result of this process, elements such as Cr, Fe, Mo, and W are separated from the matrix.
3. During austenitization silicon separates from the matrix to the carbides and simultaneously the carbide-forming elements in matrix are replaced.
4. An increase in a degree of tempering leads to intensification of carbide separation and this process can reduce the grindability of tested steels.

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