

Use of Metallographic Analysis for Evaluating Microstructures in Quenched and Tempered High-Strength Steel

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The experiment described herein focused on quantitative evaluation of carbides in the martensitic microstructure of X105CrMo17 steel. Carbides form as a result of the varying solubility of carbon and alloying elements during solidification and cooling. They possess high strength and hardness and their morphology plays a major role in mechanical properties of steel. In order to determine the relationship between carbide morphology and mechanical properties, several characteristics must be measured accurately. In this case, quantitative evaluation of carbides by means of optical microscopy and image analysis software were employed. Samples of the above-identified steel were quenched from various temperatures in order to obtain various levels of chromium and carbon dissolved in the martensitic matrix. They were then tempered at various temperatures to provide secondary hardening through precipitation. Furthermore, volume fractions and equivalent diameters of particles and the surface area density of particles were measured by metallographic techniques. Hardness was measured to assess the impact of heat treatment on strength. From the values collected in this manner, relationships between heat treatment and hardness were derived and an optimal heat treatment sequence was proposed for obtaining maximum hardness.

Keywords: High-strength steel, heat treatment, quantitative metallography, precipitation hardening.

1 Introduction

The desired properties in high-strength steels are obtained by appropriate heat treatment (HT). In martensitic steels, this consists of quenching from the austenitizing temperature and subsequent tempering. The quenched steel exhibits high strength and hardness but low toughness. Tempering restores toughness but slowly reduces strength and hardness. At certain tempering temperatures, carbide precipitation may occur in some alloy steels, leading to secondary hardening. Quenched and tempered materials show high strength and hardness, good toughness and thermal stability of properties up to the temperature of the last HT step. The processes which take

place during such HT depend on chemical composition and HT method and conditions [1]. Microstructural changes brought about by HT can be evaluated and analysed by means of metallography [2]. Relationships and dependences between the heat treatment sequence, microstructure and resultant mechanical properties can thus be identified.

The experiment described here was carried out on X105CrMo17 high-strength corrosion-resistant steel (Tab. 1) [3]. Thanks to high carbon level, this steel achieves high hardness (up to 60 HRC upon HT [3]) and high abrasion resistance. It is therefore often used for making parts of ball bearings (such as in this case). Chromium provides enhanced corrosion resistance.

Tab. 1 Chemical composition of X105CrMo17 steel

C	Mn	Si	P	S	Cr	Mo
0.95-1.2	≤1.00	≤1.00	≤0.040	≤0.03	16-18	0.4-0.8

2 Experimental schedule

The experiment involved several heat treatment sequences applied to specimens (bearing balls of 16 mm diameter) of X105CrMo17 high-strength corrosion-resistant steel. Various austenitizing and tempering temperatures were selected in order to obtain and compare material conditions with different mechanical properties and evolution of carbide phases. The treated specimens were examined using metallography, optical microscopy and hardness testing. Measurement of metallographic characteristics was employed to track dissolution of carbides at various austenitizing temperatures, the presence of fine carbide precipitates formed at higher temperatures and the size distribution of primary carbides. From these and the hardness data, the relationships between heat treatment, microstructure and mechanical properties were

derived.

2.1 Heat treatment

Unused bearing balls were soaked at the austenitizing temperatures of 980, 1030 and 1080 °C for 45 minutes and oil quenched. The soaking was carried out in laboratory furnaces without any protective atmosphere. Three specimens were used for each temperature. They were then tempered in a furnace for 120 minutes at 175, 300, 400 and 500 °C. The heat treatment sequences (Fig. 1) were based on the steel's tempering diagrams [4], with emphasis on secondary precipitation hardening. After microstructure examination, the HT sequences for specimens quenched from 1080 °C were expanded to include the tempering temperature of 700°C. The purpose of this increased tempering temperature was to confirm the hypothesis of the presence of fine precipitates in specimens

tempered at 400 and 500 °C in which black dots were observed (Fig. 2a). In the specimens tempered at 175°C, only hardness was measured, whereas the other examination techniques were omitted because microstructural

changes that take place at this low temperature cannot be detected by optical microscopy.

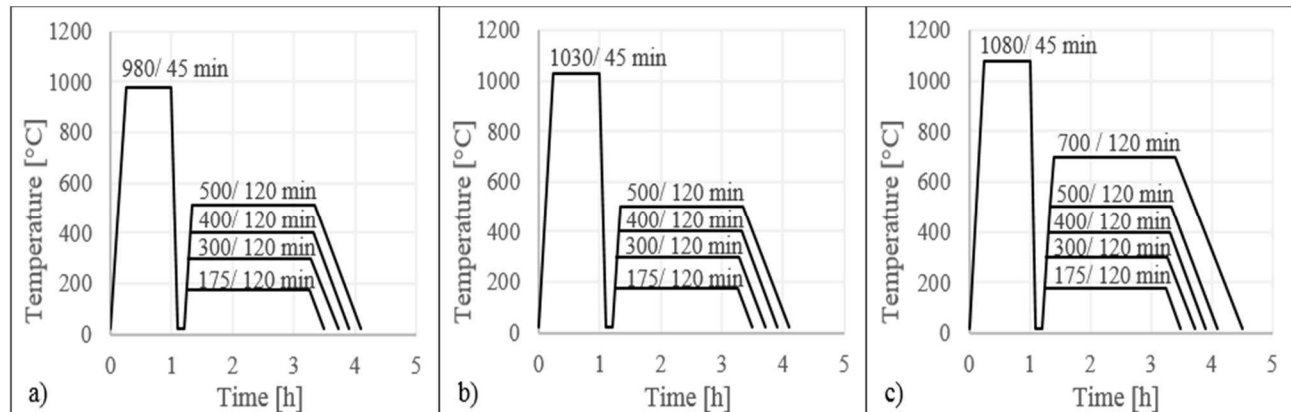


Fig. 1 Heat treatment diagrams showing the quenching temperatures: a) 980 °C, b) 1030 °C and c) 1080 °C with subsequent tempering at 175, 300, 400, 500 °C and 700 °C.

2.2 Metallographic analysis

Metallographic sections were prepared by the usual procedure on the specimens of bearing balls. The etchants used for revealing carbides were Nital 3% and the Villela-Bain reagent [5]. A Nikon Epiphot 300 metallographic microscope was used for metallographic examination. The data was processed and evaluated using the NIS-Elements image analysis software. On each specimen, 10 measurements were taken from images recorded in randomly-selected parts of the metallographic section. In these measurement runs, the thresholding for primary chromium carbides was set at 0.45 µm in the image analysis software (fine precipitates were not included due to inadequate resolving power). Using binary images, the following characteristics were calculated:

- Equivalent carbide diameter and size distribution.
- Surface area density of particles (S_V) calculated from the equation [6]:

$$S_V = 4/\pi \cdot L_A, \quad (1)$$

Where L_A is the sum of circumferences of images of particles in a unit area.

- The volume fraction of particles in the matrix V_V using the well-known stereological formula $V_V = A_A$, where A_A is the areal fraction of images of particles in the metallographic section. This formula is based on the assumed random arrangement of particles and the assumed equivalence between the volume fraction of particles and their areal fraction (essentially, one can thus track the dissolution of carbides at various austenitizing temperatures and, at the same time,

chromium depletion of the matrix) [6].

$$V_V = A_A, \quad (2)$$

2.1 Measurement of hardness

Several hardness measurements were taken during the experiment. The Wolpert Wilson Instruments CK-A3 hardness tester was employed, using the load of 1 kg, to obtain readings on the HV10 scale. At least three readings were obtained for each specimen. The mean values were then converted to values on the HRC scale by means of tables (Tab. 2).

3 Results

Microstructures of the specimens consisted of a martensitic matrix tempered to various degrees, and carbides. Two categories of particles were found: uniformly-distributed small globular carbides and large or even very large irregularly-shaped carbides (with equivalent diameters up to 24 µm). The latter were scattered at random across the entire metallographic section in view; in all specimens under examination (Fig. 2). Visual comparison between microstructures of specimens upon different treatments revealed no appreciable differences in the morphology, size or counts of these large particles (in either category).

Black dots were observed in etched specimens tempered at 400 and 500 °C. These dots were identified (in accordance with the tempering diagram for the steel) as fine chromium carbide precipitates. Their size was less than 1 µm (Fig. 3a). In specimens tempered at 700 °C, these precipitates were considerably coarser. Their appearance ranged from black dots to small white particles (Fig. 3b). At the particular magnification, these particles had inadequate areas for thresholding. Their measurement would therefore be very inaccurate and they were omitted from the measurement.

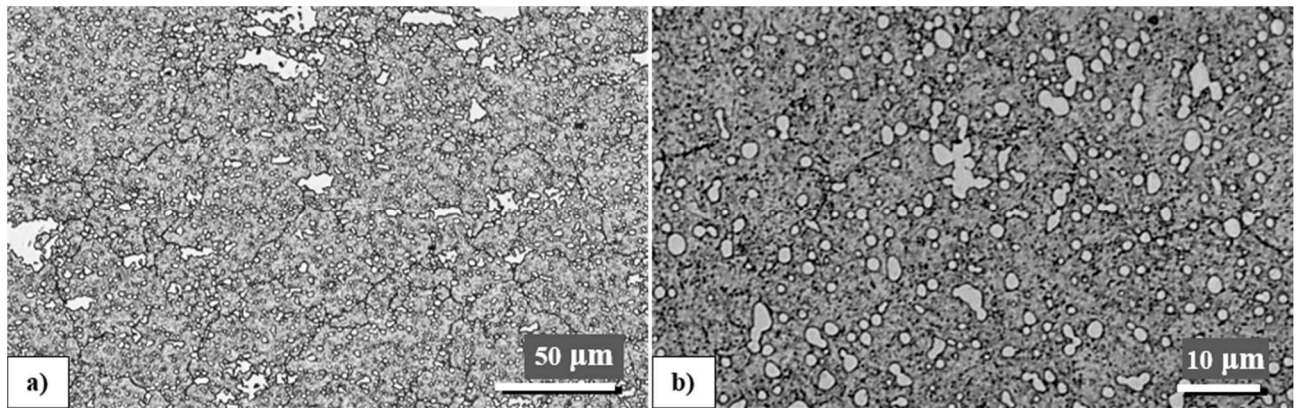


Fig. 2 Specimen quenched from 1030 °C and tempered at 500 °C – Villela Bain (15 s) + Nital 3 % (3 s); a) morphology of carbides at a general-view magnification; b) micrograph at the magnification used for measuring

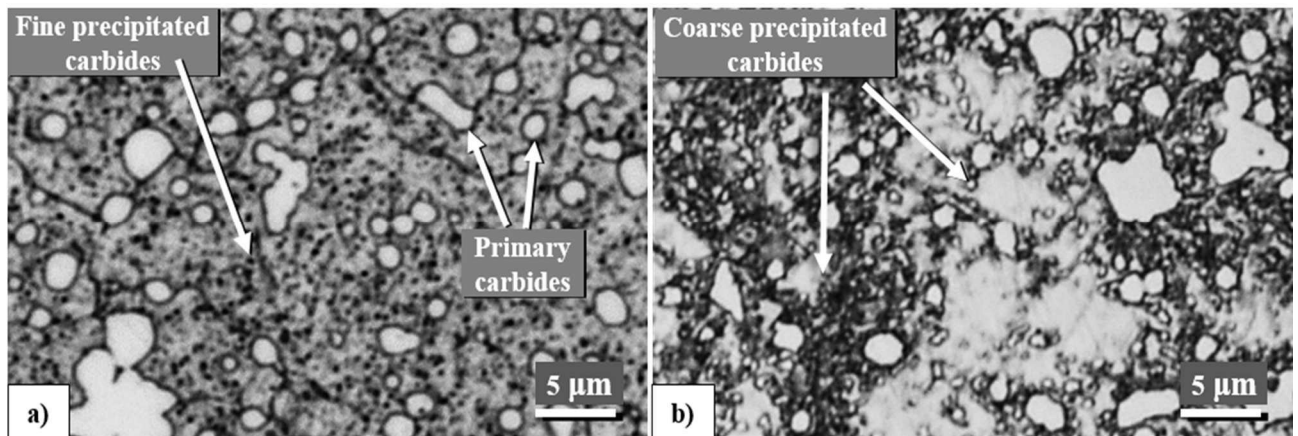


Fig. 3 Fine chromium carbide precipitates in a tempered martensitic matrix; a) specimen quenched from 980 °C and tempered at 500 °C – Villela Bain (15 s) + Nital 3 % (3 s) – fine precipitates shown as black dots within the martensitic matrix; b) specimen quenched from 1080 °C and tempered at 700 °C – Villela Bain (9 s) – precipitates which coarsened due to higher tempering temperature, 700 °C.

Tab. 2 Hardness readings and characteristics calculated from formulae (1) and (2) (areal fraction of particles and surface area density of particles) for individual HT sequences.

Heat treatment [°C]		Hardness HV10		Hardness HRC	Volume fraction of particles [%]		Surface area density of particles [mm ⁻¹]	
Austenitization	Tempering	Hardness HV10	Standard deviation		Mean	Standard deviation	Mean	Standard deviation
980	300	599	3	55	16	3	459	20
	400	592	7	55	18	2	498	10
	500	576	5	54	17	2	496	10
1030	300	618	1	56	16	2	462	20
	400	625	7	57	17	2	468	20
	500	630	8	57	18	2	530	30
1080	300	617	10	56	14	3	362	30
	400	623	5	57	15	2	398	20
	500	659	4	58	11	2	338	20
	700	289	3	28	14	3	299	20

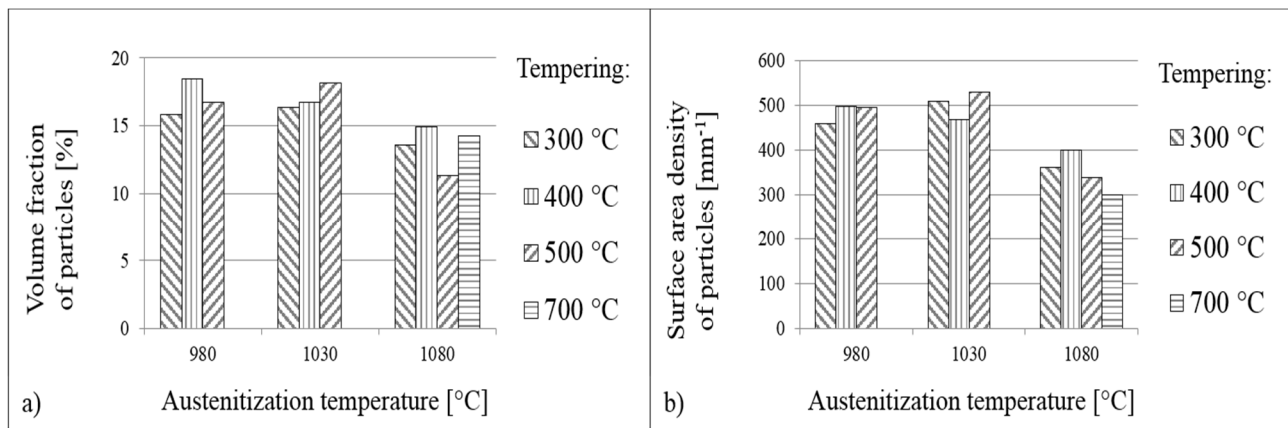


Fig. 4 Characteristics of interest vs. austenitizing and tempering temperatures; **a)** volume fraction of particles; **b)** surface area density of particles.

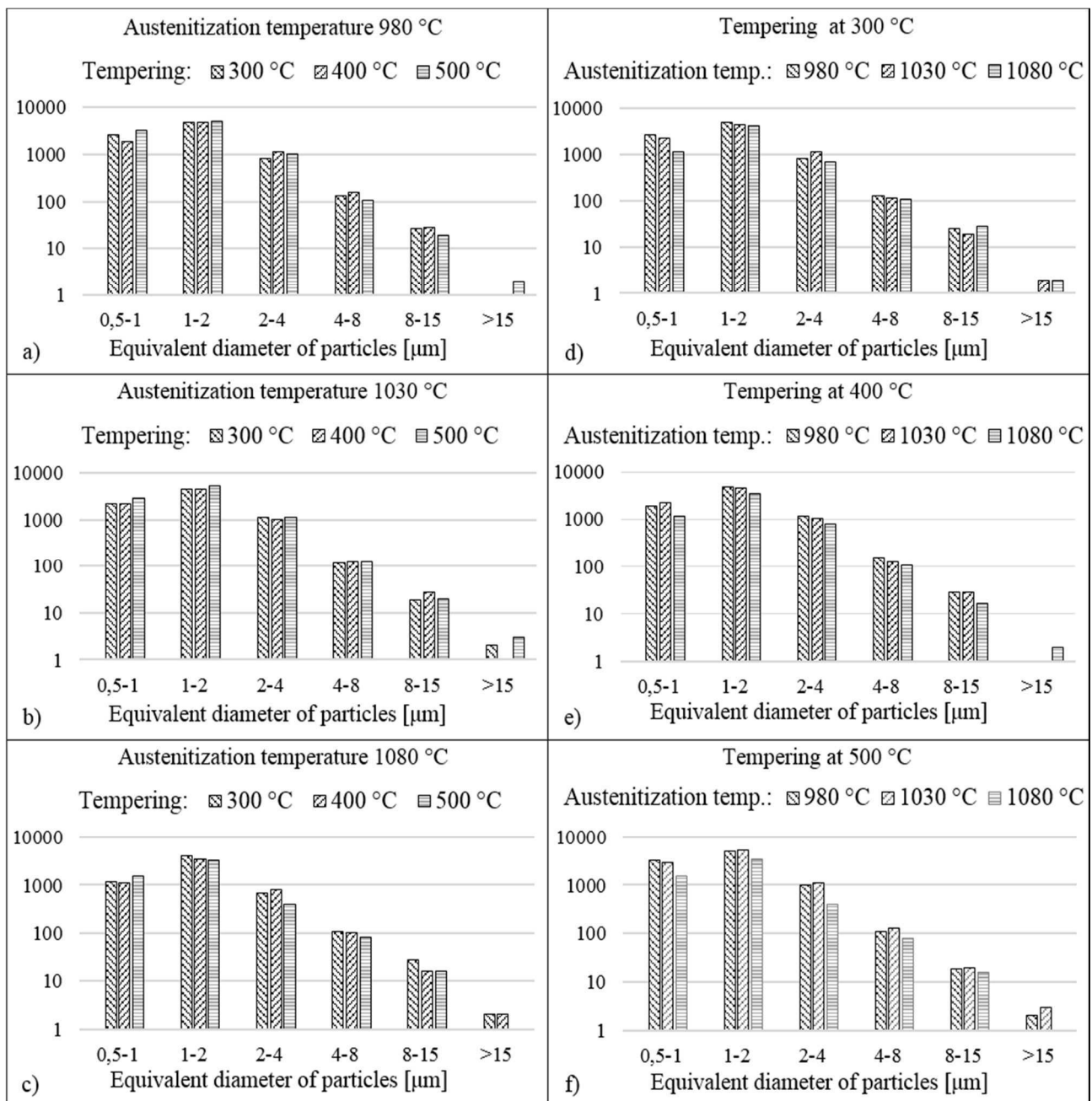


Fig. 6 a - f Histograms of particle counts per size class for various austenitizing and tempering temperatures.

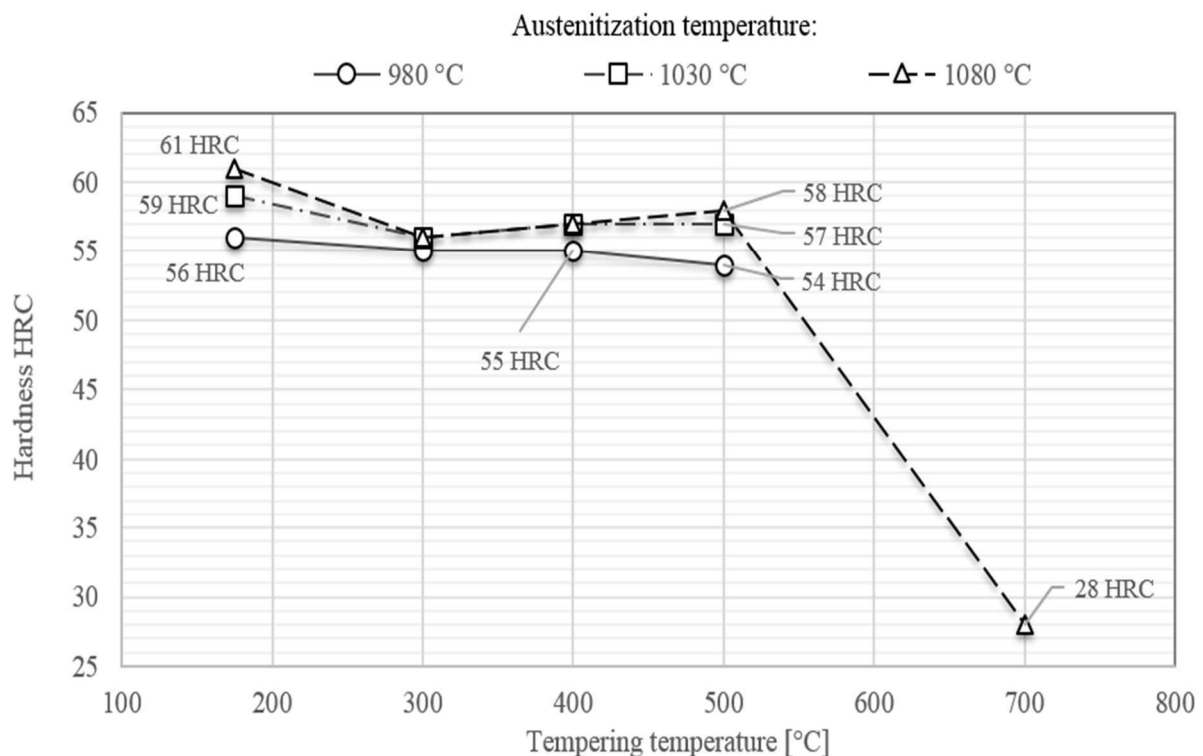
Using image analysis, no readily-apparent relationship between the amount of particles or their areal fraction and the tempering temperature was found. There are no major differences between the measured values. This is obviously caused by the high temperature stability of Cr-carbides [7]. The sizes mostly fall within the standard deviation (2-3 %). In the specimens quenched from 980 and 1030 °C, the measured volume fractions of carbides were above 15 %, whereas in those quenched from 1080 °C, the fractions were just below this value (Fig. 4 and Tab. 2). What was obvious, however, was the effect of the dependence of particle fraction on the austenitizing temperature. At the highest austenitizing temperature, 1080 °C, the fraction of carbides decreased visibly. This is in line with the assumed partial dissolution of carbides at this high temperature.

Even the size distribution of particles (Fig. 6) showed no appreciable changes with the tempering temperature. In terms of globular carbides, there were slight indications of an increase of their count with the tempering temperature. The count of large irregularly-shaped particles (with equivalent diameters of 15 μm and more) is not relevant due to the large scatter at this magnification. Histograms that show the dependence of the counts of particles of various sizes on the austenitizing temperature again showed an appreciable decrease in the amount of carbides in all specimens quenched from 1080 °C (effectively in all size classes). This can be attributed to more intensive dissolution of carbides at higher austenitizing temperatures. More intensive precipitation upon subsequent tempering can be assumed, leading to higher hardness.

In order to assess the precipitation at various tempe-

ring temperatures, electron microscopy would be an appropriate technique. As the resolving power of optical microscopes is insufficient for measuring fine secondary carbides, they are not useful for such a microstructures. Microstructural evolution in relation to tempering temperatures was therefore assessed on the basis of measured mechanical properties (hardness) and visual observation.

After comparing the above-described results and hardness readings (Graph 1 and Tab. 2), it became clear that hardness decreased with increasing tempering temperature within the range from 300 to 400 °C. At the tempering temperature of 500 °C, secondary hardening by fine precipitates occurred, which was in agreement with the above-presented results of metallographic observation. The contribution of this hardening mechanism to strength depends on the amount of chromium dissolved in the matrix which increases with rising austenitizing temperature. At the same time, the effects of this strengthening are offset to some extent by softening during tempering (recovery or recrystallization). An appreciable increase in hardness was therefore only achieved at an austenitizing temperature as high as 1080 °C, upon which increased hardness became apparent starting from 400 °C. When the quenching temperature was reduced to 1030 °C, there were only slight increases or no changes in hardness with increasing tempering temperature. As the austenitizing temperature was further reduced to 980 °C, secondary hardening became almost negligible and a gradual decrease in hardness occurred with increasing tempering temperature. The specimen which was tempered at 700 °C showed a hardness of no more than 28 HRC. Here, the effects of softening processes prevailed completely.



Graph 1 Hardness vs. quenching and tempering temperatures

4 Conclusion

This experiment on bearing balls of X105CrMo17 steel dealt with the effects of heat treatment on the morphology of carbides in martensite, and on hardness. The purpose was to obtain a quenched and tempered microstructure with maximum hardness. Specimens were quenched from various austenitizing temperatures (980, 1030 and 1080 °C, upon soaking for 45 minutes) and tempered (175, 300, 400, 500 and 700 °C, for 120 minutes). Tempering temperatures were proposed with respect to carbide precipitation and secondary hardening. After the treatment, the specimens were examined using metallographic techniques, optical microscopy and image analysis. The features of interest included the volume fraction of primary carbides in the matrix, the surface area density of the carbides, equivalent particle diameter and size distribution. Hardness on the HV10 scale was measured.

Among the specimens tempered at the higher temperatures, those quenched from 1080 °C and annealed at 500 °C showed the highest hardness. At this austenitizing temperature, the amount of dissolved primary carbides was sufficient and the level of dissolved chromium and carbon led to greater super-saturation of martensite on quenching. During subsequent tempering, intensive precipitation occurred, leading to a more appreciable hardening.

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