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Regularities of Formation of Optimal Structures of High-Carbon and Alloy Tool Steels during Double Heat Treatment

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Abstract: *This paper proposes two-stage hardening modes for U11, 9XVG, and XGS steels. In this case, the first hardening is performed with heating above Ac3, and the second hardening with heating above Ac1s. Such heat treatment ensures the production of dispersed secondary carbides, and the service life of the product increases by 3-4 times. It is shown that a high level of defectiveness of the crystalline structure of hardened steel is achieved only after heating to 1000-1200 °C. In this case, the austenite grain grows sharply. Repeated heat treatment using conventional technology ensures not only crushing of the austenite grain, but also inheritance of an increased level of defectiveness of the crystalline structure. Due to this, wear resistance increases. An increase in wear resistance is possible from 20-25% to 2 times or more. Similar heat treatment modes (double hardening, when the first hardening is high-temperature) can provide an increase in the crack propagation resistance of steel.*

Keywords: *tool steels, extreme temperatures, quenching, tempering, hardness, dislocation density.*

Introduction. Standard heat treatment modes for martensite-hardened tool steel include heating to a temperature of Ac_1 +30-50 °C, quenching in water or oil (or in water with transfer to oil) and subsequent low tempering to the required hardness. In this case, the structure contains fine-needle martensite, secondary carbides of hypereutectoid steel and residual austenite.

High-alloy steels are hardened from temperatures significantly exceeding the phase transformation point in order to dissolve the carbides of the alloying elements. Tempering is carried out either for primary or secondary hardness.

In some cases, preliminary heat treatment is carried out in order to improve the service properties of finished products. This includes all modes of heat treatment with multiple phase recrystallization. Three main directions in the use of preliminary treatment for additional improvement of properties can be established: treatment associated with the preparation of the main structure of the matrix in order to obtain ultrafine grain with repeated heating, the creation of a certain substructure and the preservation of its elements during the subsequent $\alpha_f - \gamma - \alpha_m$ transformation, the effect on the second phase, which does not undergo significant changes, with repeated lower heating.

The most acceptable are heat treatment modes with double phase recrystallization, when the first is high-temperature.

These modes are the most technologically acceptable and have been widely studied for structural steels. The essence of the method is that the first phase recrystallization is carried out from high heating temperatures. Subsequent cooling is carried out either in air, or in oil or water. At the same time, there are extreme heating temperatures, when after cooling a structure with a maximum of crystalline structure defects is formed. Research in this direction was started by L. I. Mirkin. In later works it was established that during austenization to various temperatures, the time factor plays a significant role in the formation of lattice defects. The reason for the existence of extreme heating temperatures is the formation of a heterogeneous solid solution in microvolumes at the beginning of the dissolution of refractory impurity phases of steel (carbonitrides , nitrides, oxysulfides). The beginning of dissociation and dissolution of these phases occurs at extreme temperatures, which was shown experimentally by the gas evolution of Armco iron during heating in a high vacuum. Chemical microheterogeneity of the γ-phase leads to the creation of "zone" structures during cooling, and as a consequence, to the fragmentation of coherent scattering regions (CSR) and the growth of microdistortions of the crystal lattice. A further increase in the heating temperature in the γ-region beyond the extreme temperatures leads to the homogenization of austenite and after cooling the lattice defectivity of the α-phase is lower.

During the process of hardening and tempering, interstitial atoms migrate to lattice defects and fix them.

The second phase recrystallization is carried out from heating temperatures Ac_1 +30-50°C depending on the steel grade, followed by hardening and tempering.

Thermal stability of dislocation structures fixed by impurity phases allows to obtain an increased level of defectiveness of the crystal lattice after new phase recrystallization from standard heating temperatures. This allowed to significantly increase the wear resistance of heat-treated tool steel at the same level of hardness [10].

Methodology. The heat treatment of the tool was as follows. The first group was heat treated using the usual modes for this steel: heating to 820°C, quenching in water-oil and immediate tempering at 180–240°C. The second group was heat treated using optimal modes, ensuring maximum dislocation density. A double quenching mode was used.

Metallographic studies were carried out on MIM-8 and NEOPHOT-21 microscopes using magnification from 100 to 800 times. Preparation and etching of sections were carried out in accordance with standard methods [1, 2]. The size of the austenite grain was determined according to GOST 5639-82.

The state of the fine structure was determined radiographically . The X-ray diffraction patterns were taken in the auto-recording mode using a standard made of the same steels on X-ray diffractometers: Dron 3.0-Dron-3M. Iron anode radiation was used. The physical width of the X-ray line was determined by the approximation method using correction graphs [3,4,5,6]. To obtain each result, at least four X-ray diffraction patterns were used and the arithmetic mean of the physical width of the line was determined.

The change in the crystal lattice period is due to the different dissolution of carbon and alloying elements, which depends on the heating temperature for quenching.

To determine the amount of residual austenite after heat treatment, X-ray lines (211) were recorded, where α is the (200) phase and γ is the phase. The calculation was carried out by determining the intensity ratios of these lines [9].

Results. Heating the considered tool steels to extreme temperatures of 1100-1200°C and subsequent hardening ensures the creation of a fine structure in the steel with a high dislocation density, but also with a large grain.

Repeated hardening ensures grain refinement if the heating during repeated hardening is carried out from temperatures Ac_3 +30+50°C. For U11 steel this is 820°C, for KhGS and 9KhVG – 840°C.

Dislocations obtained during quenching from heating temperatures of 1000-1100°C are thermally stable and can be inherited during repeated quenching from standard temperatures. These dislocations during repeated quenching act as ready Frank-Read sources and thus maintain a high dislocation density already with a fine grain of steel.

A number of studies have been conducted to study the mechanism of formation of grain and dislocation structure during cyclic quenching [11-13], however, all these studies do not provide a complete picture of structure formation. In the present study, possible mechanisms of structure formation in terms of manifestation of heredity of extreme heating during steel quenching are examined in more detail.

The increased dislocation density that occurs when steel is overheated to 1000-1100°C (with austenitization for 20-30 minutes) and cooled is a thermally stable structure and is retained during secondary quenching. The combined effect of these two factors can lead to the formation of a peak in dislocation density at 1200°C and tempering at 200°C.

The formation of new grains occurs inside the austenite grains obtained during the first heating. These newly formed grains acquire the same crystallographic orientation, which leads to the emergence of microstresses in the crystal lattice.

Thus, the dislocation density increases due to two factors. The first factor is the effect of the first hardening temperature of 1200°C on the microstructure of the steel. The second factor is the formation of new grains, which lead to the occurrence of microdistortions in the crystal lattice, which are preserved at the final tempering temperature of 200°C. With an increase in the final tempering temperature, the initial microstresses in the crystal lattice are gradually relieved [7,8].

These results fully correspond to the structure formation during phase transformations in steels described in the works of prof. Dyachenko S.S. the main conclusion of which is that as the new phase of the lamellar structure grows, the elastic energy of the interphase boundary increases and coherence is disrupted and new misfit dislocations are formed. Phase hardening takes place. In the process of α-γ transformation of the oxide, the structure of the interphase boundary is disrupted due to the development of relaxation processes as a result of the accumulation of misfit dislocations.

The difference between U11 steel and the results of U7 steel research is the presence of secondary carbides in the structure. During high-temperature heating, their complete dissolution in austenite occurs in the temperature range of 1000-1100°C. Dissolution of impurity phases – carbonitrides and oxygen-containing phases – occurs at approximately the same temperatures (Table 1).

The level of dislocation density in hardened steel depends very significantly on its thermal history. The highest level of dislocation density is formed if the first hardening is carried out with heating to 1000-1200°C without intermediate tempering and with intermediate tempering at 400°C. In this case, during the second hardening, carbon atoms move to dislocations (Table 1).

Table 1. Average carbon content in the tetragonal lattice of martensite, in residual austenite in U11 steel depending on the temperature of the first quenching

First hardening temperature °C	% carbon in tetragonal lattice	% residual austenite	% carbon in austenite
820	0.8	22	0.3
1000	0.75	21	0.25
1100	0.8	21	0.3
1200	0.8	21	0.3

Similar studies were conducted for 9KhVG steel (Table 2).

Table 2. Dislocation density $\rho \cdot 10^{11}$ 1/cm ^{2}, lattice period a_{nm}, amount of carbon in the tetragonal lattice of martensite (α-phase), average diameter of austenite grain d_{sr} mm, after double quenching (second at heating to 840°C) and final tempering at 200°C of 9KhVG steel

First hardening temperature °C	$\rho \cdot 10^{11}$ 1/cm ^{2}	Hardness HRC	$a_{nm} \cdot 10^{-1}$ α-phase	% carbon in tetragonal lattice	d_{cp} mm	GOST score number
840	2.8	60	2,8642	0.65	0.09	11
900	3.3	60	2,8652	0.6	0.07	11
1000	3.9	60	2,8660	0.55	0.05	12
1100	3.6	60	2,8660	0.65	0.05	12
1200	3.2	60	2,8658	0.8	0.04	13

The table shows that the highest dislocation density occurs when the first hardening is performed with a heating temperature of 1000°C, and subsequent heat treatments are hardening at 840°C and tempering at 200°C. In this case, the grain size of the steel reaches 12-13 points, i.e., very fine-grained. The table shows that the highest dislocation density occurs with a minimum lattice period and a minimum number of carbon atoms in the tetragonal lattice of martensite.

Conclusion. The present studies confirm that there is a heredity of the fine structure when heating steel to high temperatures, which is typical for many structural tool steels of industrial smelting. This heredity is expressed in the fact that when heating steel to temperatures of 1000-1100 ° C with subsequent cooling, a fine structure with a high level of dislocation density is formed. Regardless of the grain size from the first quenching, during repeated quenching with heating Ac_3 + 30-50 ° C, there is an inheritance of the elements of the original structure - a high level of dislocation density, an effect on the size of the austenite grain during a new α-γ-α transformation. There is a significant redistribution of carbon atoms between martensite and residual austenite. Ultimately, the manifestation of heredity at high heating of steel will affect the change in the size of steel products during heat treatment, as well as the wear resistance of finished products.

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