EFFECT OF QUENCHING AND TEMPERING TEMPERATURES ON THE STRUCTURE FORMATION OF 4XMFC, 4X5MF1C STEELS DURING LOW-TEMPERATURE NITRO-CEMENTATION

Begatov Jakhongir Mukhammadjonovich[1], Ergashev Meirzhan Sattar ugli[1], Platoshina Malika Mirkamal kizi[2]
[1]Mechanical Engineering Technologies Department, Belarusian-Uzbek Intersectoral Institute of Applied Technical Qualifications, Tashkent, Uzbekistan
[2]Kimyo International University in Tashkent, Uzbekistan
E-mail: begali.bektemirov@tdtu.uz

Abstract - Currently, a large number of studies are being conducted in the field of application of methods of thermal and chemical-thermal treatment, and there are some unresolved technological problems. In particular, the issues of the formation of wear-resistant structures have not been sufficiently studied in the study of combined methods of chemical and thermal treatment, the development of which makes it possible to reduce the technological cycles of the hardening process of the surface layer of metals. This paper examines the issues of the formation of wear-resistant structures during technological operations that allow combining the tempering process with the saturation of the steel surface with nitrogen and carbon atoms, as well as issues of reducing the technological cycles of hardening using methods of chemical-thermal treatment, the possibility of using standard equipment, preparation of the saturation medium based on local raw materials.

Keywords: Nitrocementation, Methane, Quenching, Austenite, Wear resistance, Heat resistance, Martensite, Carbon.

1. Introduction

Tool steels are divided by purpose into two main classes – cutting and die steels [1]. In terms of heat resistance, they differ into non-heat-resistant, semi-heat-resistant, and heat-resistant [2]. A large range of cutting tools is made from cutting tool steels, from various cutters, cutters, drills, broaches, taps, etc. to complex combined tools. At the same time, the main class of tool steel for the manufacture of cutting tools is high-speed steel. Both cutting and stamping tools are subjected to high contact and thermal loads, so they require high hardness, strength, wear resistance, and heat resistance [3]. All these properties of steel are obtained in the process of heat treatment and chemical-thermal treatment [4]. In the process of various heat treatment modes, steels have a martensitic structure, and when using alloy steels, finely dispersed carbides are formed, providing hardness and heat resistance. According to [5], the indicator of the level of heat resistance is the temperature of a two-hour tempering of hardened steel to the required hardness. The wear resistance of the tool is determined by the wear rate of the tool, which is affected by the temperature and contact pressure experienced by the surface layer of the tool. However, it should be noted that in some cases the use of standard modes of heat treatment and chemical-thermal treatment cannot provide the necessary level of hardness and heat resistance. No less important, in addition, to wear resistance, are technological properties, in particular, such properties include: hardenability, hardenability, lack of decarburization during heating for quenching, and machinability by cutting.

2. Theoretical Research

The technological feature of the processes of saturation of steel with nitrogen and carbon is the possibility of using a wide range of saturation temperatures [6]. Depending on the temperature intervals of saturation, high-temperature, medium-temperature, and low-temperature nitro-cementation processes are distinguished. Cyanidation of steel is carried out in molten salts consisting of sodium cyanide or potassium cyanide. The saturation temperature range is 900-950°C for high-temperature cyanidation, 820–870°C for medium-temperature cyanidation. Both of these processes are quite toxic and violate environmental safety. In addition, it is difficult to regulate the saturation depth and the concentration of nitrogen and carbon atoms in the saturated layers in the processes. Therefore, the main application in the industry was the process of gas nitro-cementation

[7]. Gas nitro-cementation is carried out by feeding a mixture of natural gas and ammonia or a mixture of methane and ammonia (endogas) into the furnace space [8]. However, this process also has disadvantages:

- lack of accurate data on the addition of ammonia, i.e. in each case, a preliminary calculation is carried out, including data on the size of the furnace, the temperature of the process, the amount of gas passed through;
- changes in the content of undissociated ammonia in various furnace designs, affecting the results of nitro-cementation.

Sufficiently good and stable results of nitro-cementation processes are obtained with unchanged set process parameters, which is possible only in conditions of mass production, using continuous units for a specific part design [9]. Under these conditions, it is possible to automate the technological process of nitro-cementation, which is the main economic advantage of this process.

However, the use of gas nitro-cementation in conditions of single and small-scale production leads to an increase in the cost of the process due to the above disadvantages. In this case, the nitro-cementation process in a solid medium has an advantage. For the low-temperature nitro-cementation process, a solid medium consisting of 60-80% solid carbon black carburetor and 30-40% $CO(NH_2)_2$ – urea carbamide was used. A container with samples and a saturating medium was placed in a furnace heated to different temperatures (550-600 °C) and the saturation time was calculated from the moment the temperature was set after loading the container.

3. Experimental Research

The modes of combined chemical and thermal treatment were as follows. Quenching was carried out with different heating temperatures. For 4XMFC, and 4X5MF1C steels, standard heating temperatures were taken: 920-930°C for 4XMFC steel, and 1020°C for 4X5MF1 steel. As a second heating mode for quenching for 4XMFC steels. For high-speed steel, the heating modes for quenching were 1200-1230 °C.

The steels were heated for quenching in NaCl and $BaCl_2$ salt baths. Samples of 20x20x10 plates of steel were heated, and the heating time was 0.3-0.5 minutes per 1 mm of the cross-section. After heating, quenching in oil was carried out. Then the vacation was carried out with the combination of the nitro-cementation process.

Metallographic studies were carried out on microscopes MIM-8, and NEOFOT-21 using magnification from 100 to 800 times. Preparation and etching of the grinds were carried out following standard methods [10]. The value of austenitic grain was determined according to GOST 5639-82.

The state of the fine structure was determined radiographically. X-ray images were taken in auto-recording mode using a standard made of the same steels on X-ray diffractometers: Drone 3.0-Drone-3M. The radiation of an iron anode was used. The physical width of the X-ray line was determined by the approximation method using reference graphs [11]. To obtain each result, at least four radiographs were used and the arithmetic mean of the physical width of the line was determined. The results of the state of the fine structure were determined directly by the physical width of the X-ray interference line. The dislocation density was calculated using the formula:

$$\rho = \frac{\beta^2}{2b^2} \cdot ctg^2\theta \tag{1}$$

where

θ – reflection angle,

β is the physical width of the X-ray line,

b is the Burgers vector,

The period of the crystal lattice was found by the position of the center of gravity of the interference line distribution:

$$\alpha = \frac{\lambda}{2\sin\theta}\sqrt{h^2+k^2+l^2} \tag{2}$$

where

λ is the wavelength of X-ray radiation,

θ is the angle of reflection,

h,k,l is the crystallographic indices.

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

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

4. Research Results

Hardening of steel from different heating temperatures usually involves the growth of austenitic grains and when the steel is cooled, large-needle martensite is obtained. The presence of alloying elements somewhat reduces the increase in the intensity of austenitic grains. It is known [12] that the main barrier preventing the growth of austenitic grains is insoluble refractory phases.

These are mainly carbides and nitrides. The main obstacle to the growth of austenitic grain for the steels under consideration are carbides of alloying elements Cr, Mo, V.

When the studied steels are heated for quenching, the austenitic grain also grows (Fig.1), but it is not as pronounced as for simple carbon steels. The structure of these steels after quenching at temperatures of 1100-1150 °C is martensite, residual austenite, and carbides of alloying elements.

In previous studies, it was found that when quenching tool steels from extreme temperatures of 1100-1200°C, a structure with an increased dislocation density is formed. It was found that at these temperatures the initial stage of dissolution of refractory impurity phases occurs. All this in general leads to the appearance of chemical heterogeneity of austenite and the growth of austenitic grains (Fig.1). During quenching, an increased level of defectiveness of the crystalline structure of the α-phase is formed during abrupt cooling. In the process of tempering in these steels, the reverse process occurs – the release of carbides and impurity phases in the form of fine particles. This affects the percentage of residual austenite formed. Therefore, studies were conducted to determine the effect of the quenching temperature on the percentage of residual austenite (Fig. 2, 3).

The quenching temperatures for each steel grade were applied according to the experimental method and ranged from standard temperatures to temperatures up to 1200°C.

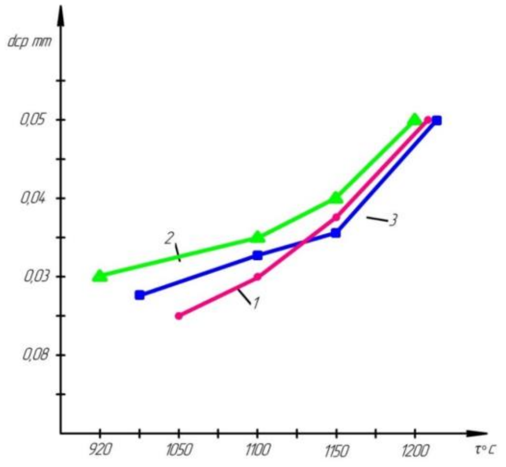

Figure 1: The value of austenitic steel grain depending on the tempering temperature:
1 – Steel X12F1, 2 – Steel 4XMFC, 3 – Steel 4X5MF1C

Thus, it should be noted that the effect of high-temperature hardening on the growth of austenitic grains and the amount of residual austenite is ambiguous. With an increase in the temperature of heating for quenching, the grain grows, but the increase at temperatures of 1100-1150°C is not so significant, the grain grows sharply only when heated at 1200°C.

The content of residual austenite after quenching with tempering for 4XMFC, 4X5MF1C steels varies slightly from standard temperatures to 1150°C and again increases sharply after quenching from 1200°C, regardless of the tempering temperature. Moreover, in all cases, the minimum values of residual austenite reach 600 °C with the use of tempering.

Since the width of the X-ray line (220) and (211) is an integral characteristic of the defect of the crystal structure, studies were conducted at the beginning on the effect of quenching and tempering modes on the width of line (220) (Fig.4)

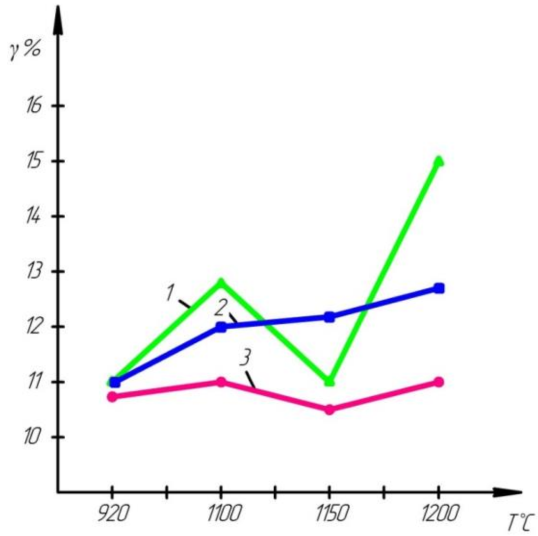

Figure 2: The content of residual austenite after quenching from various temperatures of steel 4XMFC: 1 – without vacation, 2 – 550 °C vacation, 3 – 600 °C vacation

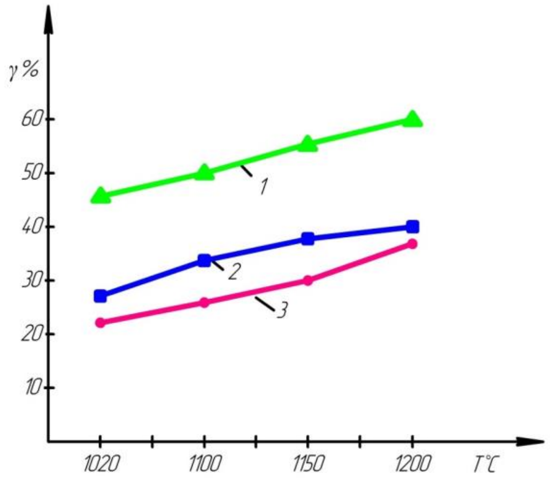

Figure 3: The content of residual austenite after quenching from various temperatures of steel 4X5MF1C: 1-without vacation, 2-vacation 550 °C, 3-vacation 600 °C

The crystal lattice period was determined according to the position of the center of gravity of the interference line distribution (section 2). Both of these structural parameters have a great influence on the wear resistance of steel [13].

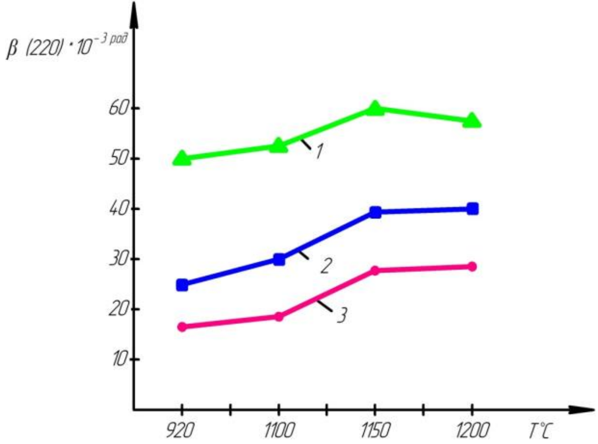

Figure 4: Influence of the tempering and tempering temperature of 4XMFC steel on the width of the X-ray line (220):
1-without vacation, 2-vacation 550 °C, 3-vacation 600 °C

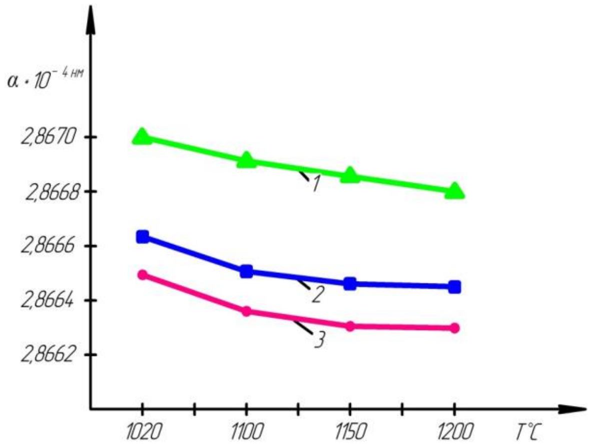

Figure 5: Influence of quenching and tempering temperature on the crystal lattice parameter of 4XMFC steel:
1-without vacation, 2-vacation 550 °C, 3-vacation 600 °C

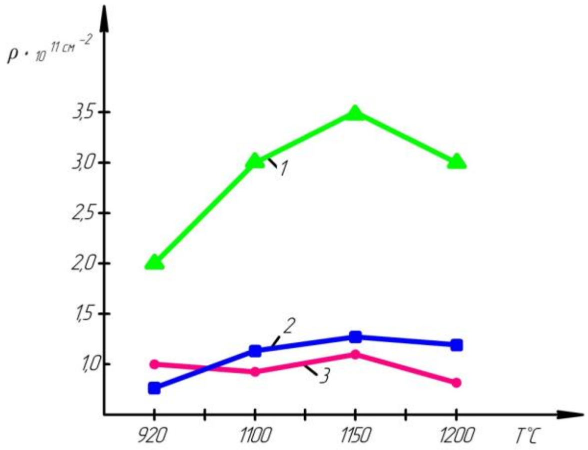

Figure 6: Influence of quenching and tempering temperature on the dislocation density of 4XMFC steel:
1-without vacation, 2-vacation 550 °C, 3-vacation 600 °C

The results of studies of the state of the fine structure of steels indicate that the maximum level of defects in the crystal structure of the studied steels is formed during quenching from temperatures of 1150-1200°C, which is confirmed by data on the physical broadening of the lines (220) and (211) and an increase in the density of dislocations (Fig. 5) [14]. The parameter of the crystal lattice decreases in this case (Fig.6) because an increase in the density of crystal lattice defects leads to a significant decrease in the amount of carbon in the tetragonal lattice of martensite, that is, part of the carbon atoms goes to lattice defects [15-21].

Figure 7: Cyanated layer after saturation 4 hours, x500

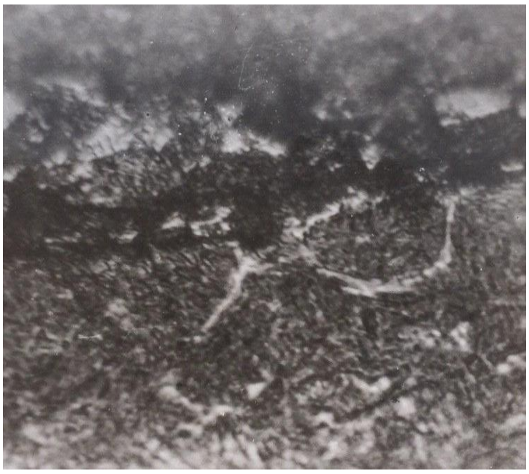

Figure 8: ε – phase along the grain boundaries in the cyanidated layer, x500

On the surface of the cyanidated layer, a thin non-etched layer of a light-colored carbide crust is formed. After the crust layer, there is a thick dark-etched zone that does not have a sharp boundary with the main structure. The hardness of the dark-etched zone is HV 10000 MPa, the hardness of the light skin is HV 8600 MPa.

The structure of the dark-etched zone is a mixture of martensite, carbides and carbonitrides of the $M_3(C, N)$ type. The change in the microhardness of the surface layer of samples of steels 4XMFC, 4X5MF1C and X12F1 subjected to low-temperature nitrocarburizing is shown in (Fig. 7-8) [22-26].

5. Conclusions

The use of high-temperature hardening for all the steel grades under consideration leads to an increase in the defect of the crystal structure, as well as an increase in the percentage of residual austenite.

The possibility of combining the processes of tempering and low-temperature cyanidation at temperatures of 550-600 °C has been established.

The most effective saturation composition shows composition 2 (60% soot + 40% carbamide).

The saturation depth depends on the temperature and holding time for all the steel grades under consideration.

The greatest value in the depth of the diffusion layer is achieved at a temperature of 600 °C and exposure in the region of 3-4 hours.

The greatest value of the microhardness of the surface layer reaches in the process of nitro-cementation with an exposure of 3-4 hours for all grades of steel.

Microhardness for 4XMFC and 4X5MF1C steels reaches values from HV 8000 to HV 10000. The hardness of samples and tools was determined on a TK-2 hardness tester, microhardness on a PMT – 3.

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