Influence of production technology on the structure and properties of powder high-speed steels

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Abstract. Powder high-speed steels are a new group of tool materials with enhanced operational capabilities compared to steels made using traditional technology. The paper presents an assessment of the impact of powder steel production technology on their structure and properties. The studies are carried out on the example of two grades of experimental powder steels - R7M5F2-MP and M5F5-MP in three directions: the influence of compacting technology on the structure and properties of powder steels; development of optimum modes of hardening heat treatment; improving the quality control methodology of powder high-speed steels at the main stages of their production technology. A comparative analysis on the quality of the experimental powder high-speed steels depending on the chemical composition and various schemes for compacting workpieces from atomized powder is carried out. As a result of studies, the influence of hardening heat treatment modes on the phase composition, structure and basic properties of powder high-speed steels is established. Recommendations on hardening thermal working regimes to ensure the optimum ratio between hardness and heat resistance of steels are developed. Studies have shown feasibility of using powder high-speed steels (including domestic ones).

1. Introduction
A group of powder high-speed steels has been developed in Russia, and some of them are included in GOST 28393 [1]. These steels differ significantly in chemical composition from traditional high-speed steels according to GOST 19265 [2] by a high content of carbon (up to 2%) and carbide-forming elements, including inexpensive vanadium, as well as production technology [3]. This technology significantly improves the structure of steels, providing dispersed, uniformly distributed carbide particles [4]. This has a positive effect on the basic properties of powder tool steels - heat resistance, hardness and strength. To an even greater extent, this method of steel production allows to increase the level of their technological properties - hot ductility (up to 30%) and, especially, grindability (2–3 times) [5].

In this work, studies were conducted on experimental powder high-speed steels of domestic production (M5F5-MP) and imported ones (R7M5F2-MP) to establish the influence of production technology on quality of these steels. In accordance with the task, research was conducted in the following areas:

- improvement of the quality control method for powder high-speed steels in the delivery state, as well as heat treatment quality in the manufacture of tools;
- analysis of the impact of powder high-speed steels compacting technology on their microstructure and properties, identification of typical defects;
• development of hardening heat treatment modes for experimental powder high-speed steels to ensure the optimum ratio between secondary hardness and heat resistance.

As a result of the studies, a comparative analysis on the quality of experimental powder high-speed steels depending on the chemical composition and various schemes for compacting workpieces from atomized powder is carried out. Recommendations on hardening heat treatment modes are developed. Recommendations are given on the use of prototype steel grades.

2. Research methodology
It included quality control of powder high-speed steels of an experimental chemical composition at various technological stages of production. Namely, in the delivery state after compaction according to various schemes and after hardening heat treatment [6, 7].

One of the research objectives was development of hardening heat treatment modes. An analysis of the influence of the heat treatment mode (hardening temperature, tempering temperature, tempering rate) on the basic properties and microstructure of powder steels was conducted. Hardness and heat resistance were chosen as optimization parameters of hardening heat treatment modes. Impact strength was indirectly evaluated based on the results of microstructure control, namely, the grain score and the microstructure of heat-treated powder high-speed steel.

The studies covered determination of chemical composition, control of basic properties, as well as metallographic studies of the macro- and microstructure of steels in accordance with the Russian standard for powder high-speed steels (GOST 28393).

The list of tests included:
• determination of chemical composition;
• study of macrostructure - oxygen segregation;
• study of microstructure according to the parameters: structural banding, presence of foreign particles, presence of micropores, carbide heterogeneity, austenite grain size (GOST 5639);
• control of the main properties according to the following indicators: hardness in the state of delivery (GOST 9012), hardness after hardening, hardening and tempering (GOST 9013), main mechanical properties - impact strength (GOST 9454), bending strength (GOST 14019).

The phase composition was investigated using x-ray diffraction analysis and using magnetic measurements on an anisometer.

3. Research results
Two grades of powder high-speed steels of experimental chemical composition, differing in chemical composition from standard steels according to GOST 28393, were studied in the work. This is a tungsten-containing grade of the type R7M5F2-MP (0.9% C; 7.4% W, 5.7% Mo, 2.4% V) of foreign production and tungsten-free steel of the type M5F5-MP (1.75% C; 5.5% Mo, 6.0% V%, 4.8% Cr) of domestic production. These grades differ not only in chemical composition, but also in the technology features to produce workpieces [8, 9, 10].

The technology for manufacturing powder high-speed steels consists in spraying liquid steel in an inert medium and subsequent hot compaction of the powder into dense workpieces.

The powders of the studied steels were made by gas dispersion in a controlled atmosphere — in a nitrogen atmosphere. This technology enables to provide for the cooling rate of liquid metal droplets at the level of 103-105 deg/s. They had a size of up to 300 microns. At this stage, due to the high cooling rate during crystallization, a dispersed uniform microstructure with a particle size of 1-2 microns is formed in them.

The next step in the manufacture of powder high-speed steels is to compact the powder into dense workpieces. The compaction method should provide:
• obtaining dense workpieces, and this task is complicated by the high hardness of the powder;
• maintaining advantages of the structure and phase composition of powders obtained under conditions of high cooling rates;
• minimum costs for workpiece production.
Dense workpieces by traditional methods of pressing and sintering from high-speed steels cannot be obtained.

Currently, various technologies are used for manufacturing high-speed steel workpieces from dispersed powder. These include:

- hot isostatic pressing of vacuum-processed capsules with powder under pressure of 100-200 MPa, followed by forging;
- hot extrusion of capsules with powder;
- compacting the capsules with powder by rolling.

Pioneers are compacted in the temperature range of 1100–1200°C. Powder capsules are vacuum-processed or filled with an inert gas.

The tungsten-free powder high-speed steel M5F5-MP studied in this work was made of a powder atomized by nitrogen. Two workpiece production methods were used:

1) method of isostatic pressing followed by forging
2) hot extrusion of the workpiece with an extrusion rate of more than ten.

Despite high content of carbon and vanadium, there was no difficulty in compacting the experimental steels.

After compacting, the workpieces made of powder steels had an increased hardness of 56–58 HRC; therefore, they were necessarily annealed according to the standard regime for high-speed steels (at a temperature of 840–860 °C with an isothermal exposure of 720–750 °C for 1–2 hours depending on the mass of the workpiece) [11, 12].

After annealing, steel of M5F5-MP type had a hardness of no more than 240 ÷ 260 HB. The phase composition of steel consists of 25–30% of carbides of the type $\text{MC}$, $\text{M}_6\text{C}$ and $\text{M}_2\text{C}_6$, and the carbide $\text{MC}$ was the main one.

The compacting method was to ensure the production of dense workpieces and, at the same time, preserve the advantages that were laid down at the stage of powder production. Studies of microstructure and phase composition showed that this problem was solved. After one- and two-stage compaction of M5F5-MP steel workpieces, the microstructure was fine-grained, homogeneous with a uniform distribution of dispersed carbides with a size of not more than 1-3 microns (1000-1500X). Porosity (200X), structural banding (100X), oxygen segregation (200X), and sulfur segregation were not detected, i.e. steel met the requirements of standards.

Thus, powder high-speed steels of M5F5-MP type can be compacted both in two stages (by isostatic pressing followed by forging) and in one stage (by extrusion with a nominal deformation of at least 90%, for workpieces up to 50 mm in diameter).

Steel of R7M5F2-MP type, as shown by metallographic studies, was manufactured using simplified technology, i.e. single-stage compaction. She had a less favorable structure. Porosity was found here, but not higher than the allowable score of 1. The main drawback of this steel was presence of structural banding of 1 ÷ 2 points and less dispersed, fused carbides with sizes up to 5 ÷ 6 micron.

Deterioration of the microstructure can be explained by an insufficient degree of deformation and violation of the temperature regime of compaction. The overheating of steel during compaction led to a partial melting of the carbide phase and caused a deterioration in the microstructure, and an insufficient degree of deformation led to structural banding. In addition, sulfur segregation (point 1) and oxygen segregation (point 1) were present in the steel.

Thus, imported high-speed powder steel after compacting is significantly inferior in metallurgical quality to domestic-made tungsten-free powder steel.

The optimum heat treatment mode for experimental steels is developed on basis of ensuring the optimum ratio between hardness and heat resistance. Due to the special microstructure and phase composition, the hardening and tempering of powder high-speed steels is carried out at lower temperatures compared to traditional high-speed steels.

In order to optimize the hardening regimes of heat treatment of experimental powder high-speed steels, a generally accepted technique was used, namely, the hardening was carried out in the range
from 1160 to 1220 °С, and the tempering temperature was in the range of 540-560 °С. The rate of tempering varied from one to four, each of which was carried out for 1 hour.

The M5F5-MP steel study results are shown in Fig. 1 and Fig. 2.

**Figure 1.** Effect of hardening temperature on the primary (1) and secondary hardness of M5F5-MP steel after one (2), two (3) and three (4) temperings at 540 °С on the amount of residual $A_{res}$ austenite (5).

**Figure 2.** Effect of double tempering temperature on the secondary hardness of M5F5-MP steel depending on the hardening temperature: 1, 2, 3, 4, respectively, 1160 °С, 1180 °С, 1200 °С, 1220 °С.

**Figure 3.** The microstructure of powder high-speed steels M5F5-MP and R7M5F2-MP, respectively:
- a and c – grain size; b and d – microstructure after hardening heat treatment

Dependence of hardness of powder high-speed steel M5F5-MP on tempering temperature ($T_3$) is shown in Fig. 1. It can be seen from it that the maximum primary and secondary hardness is ensured in the temperature range of 1180 ± 5 °С. The tempering temperature should not exceed 1200 °С,
since otherwise there is a significant dissolution of the carbide phase and formation of an increased amount of residual austenite that is resistant to tempering, as well as an increase in austenite grain. On the other hand, at a tempering temperature less than 1160 °C, austenite alloying is not ensured, and consequently, the secondary hardness and heat resistance of steel (at 620 °C HRC 57).

In the heat-treated state, according to the optimum mode, M5F5-MP steel had a fine grain of 12 points according to GOST 5639-82 [13] and a uniform distribution of carbides by 1 point according to GOST 19265 with carbide sizes of 1 ÷ 2 μm, which corresponds to the optimum microstructure of heat-treated powder high-speed steel according to GOST 28393 (Fig. 3 a, b). The phase composition after heat treatment consisted of martensite, 19–20% of carbide phase, and 5–7% of residual austenite.

In powder steel, as in ordinary forged steel, dispersion hardening develops during tempering (see Fig. 2), which additionally increases hardness by 2.5–3 HRC. The dependency analysis in Fig. 2 shows that the most optimum tempering temperature is 540–550 °C (T_{tmp}) from the tempering temperature of 1180 ± 5 °C, and Fig. 1 indicates feasibility of a double tempering. This mode provides high secondary hardness (HRC 66–67). The heat resistance after exposure for 4 hours at 620 °C was HRC 60.

Regardless of compaction method, the steels have satisfactory toughness at the level of heat-resistant high-speed steels, which is 0.2 - 0.3 MJ/M². The bending strength corresponds to 2800 - 3200 MPa [12].

As a result of studies, for R7M5F2-MP steel, similar dependences of the effect of tempering temperature on primary and secondary hardness were obtained. This steel is distinguished by its chemical composition with a high content of tungsten, molybdenum and vanadium, which is reflected in the heat treatment conditions, primarily in the hardening temperature. It is higher by about 20–30ºC in comparison to M5F5-MP and comes up to 1210 ± 5 °C. Moreover, the maximum hardness is also achieved after a double tempering from a temperature of 540-550 °C. This mode provides maximum heat resistance, which is confirmed by the results of the heat resistance test presented in Fig. 4.

![Figure 4. Effect of tempering mode (T_{tmp} = 1210 ± 5 °C) of R7M5F2-MP steel on heat resistance after four hours at 630°C](image)

As you can see, frequency of tempering significantly affects heat resistance of steel. An increase in the frequency of tempering above two leads to a loss of heat resistance and is especially significant after tempering from 560 °C.

Studies of the microstructure of R7M5F2-MP steel showed that it is inferior in quality to M5F5-MP steel (Fig. 3 a and r), since carbide size in it reaches 5–6 μm. It corresponds to the grain size and microstructure in Fig. 3 g and r according to GOST 28393. However, despite the foregoing, the structure of the investigated steel is much better in comparison to traditional high-speed steels. The grain size of R7M5F2-MP steel corresponds to 11 points according to GOST 5639, and the carbide inhomogeneity point corresponds to 1 point according to GOST 19265.
4. Discussion of the results

The studies on the selection of a hardening heat treatment mode for powder high-speed steels using the examples of M5F5-MP and R7M5F2-MP have several features. This treatment is characterized by a low tempering temperature (from 1180 to 1210 °C) compared to traditional steels and depends on the chemical composition of steel. Underheating of steel during hardening does not provide maximum heat resistance, and overheating is dangerous by melting the grain, moreover, at sufficiently small sizes.

To ensure maximum secondary hardness and heat resistance for powder steels, it is advisable to conduct a double tempering at low temperatures in the range of 540–550 °C for 1 hour each. The increase in temperature and the frequency of tempering leads to a loss of heat resistance.

An analysis of the influence of the chemical composition on the basic properties of powder high-speed steels showed that tungsten-free steels, in this case, are not inferior to tungsten-containing powder high-speed steels in their basic properties. A negative effect on the phase composition and properties of R7M5F2-MP steel is caused by an imbalance in the chemical composition, in this case, a carbon deficit. An insufficient amount of carbon leads to a decrease in the volume of the carbide phase, and, therefore, negatively affects the secondary hardness and heat resistance of this steel [14].

Defects in metallurgical production (stitching, segregation, porosity, carbide melting) that occur at the compacting stage are inherited in the final structure of R7M5F2-MP steel and adversely affect basic mechanical properties, i.e., therefore, the tool life. This is another drawback of imported steel compared to domestic steel M5F5-MP.

5. Conclusions

1. Quality control of two grades of powder high-speed steels - M5F5-MP steel of domestic production and R7M5F2-MP steel of imported production is carried out in accordance with the Russian standard for these steels according to GOST 28393 at various stages of production. It is determined that
   – both steels comply with the standard in structure and properties (carbide inhomogeneity point 1, grain size 11–12 points);
   – R7M5F2-MP steel is inferior to M5F5-MP steel in macro- and microstructure due to the presence of porosity (point 1), structural banding (1-2 points), less dispersed, fused carbides with sizes up to 5-6 microns, sulfur segregation (point 1), oxygen segregation (point 1).

2. The effect of compacting technology on the structure and properties of powder high-speed steels is investigated. On the example of domestic steel M5F5-MP, the possibility of single-stage compaction by hot extrusion of workpieces with a nominal deformation of at least 90% for workpieces with a diameter of up to 50 mm is shown. R7M5F2-MP steel in accordance with the domestic standard has a deviation in structure in the form of fused carbides and structural banding.

3. Recommendations on the regime of hardening thermal testing for powder steels have been developed in order to ensure the optimum ratio between hardness and heat resistance. Namely:
   – the hardening temperature depends on the chemical composition and is about 1180 ± 5 °C for M5F5-MP steel; 1210 ± 5 °C for R7M5F2-MP steel;
   – double tempering at low temperatures in the range of 540–550 °C for 1 hour each;

4. Overheating of powder high-speed steels leads to the melting of carbides in the presence of fine grain, and underheating is undesirable due to a decrease in heat resistance. The increase in temperature and the frequency of tempering lead to a significant decrease in heat resistance.

5. The conducted studies have confirmed that the constant displacement of domestic tool materials by more expensive imported materials is not justified [15], despite the cost-effectiveness of using powder high-speed steels [16].
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