MECHANICAL PROPERTIES AND SURFACE TREATMENT PM COBALT HIGH SPEED STEELS

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Abstrakt

The study investigated the relationship between microstructure and selected mechanical properties of high-speed steels (HSS) of P/M AISI M2 type alloyed with 5 and 8% Co, produced by rapid solidification and subjected to HIP compaction   and thermal treatment. Cobalt, as an alloying addition, refines and stabilises the structure and mechanic properties of the material increasing serviceability of steel of this type in comparison with P/M AISI M2 HSS without cobalt. Carbidic phases, uniformly distributed in the matrix, affect positively the hardness and cutting, strength and fracture characteristics. Comparison of selected mechanical properties and evaluation of structure by means of image analysis allowed us to establish important relationships and obtain more detailed knowledge about industrial properties of powder high-speed steel. Pulse plasma nitriding, used as an experimental surface treatment of the respective steel, represents a progressive approach aimed at improvement of surface properties of this material and increased service life of the produced cutting tools. Optimum thickness of the nitride layer was determined by changing thermal and temporal parameters of the process. The preliminary thickness of the nitride layer ranged between 17 and 50 μm at hardness HV 0.025 ranging from approx. 1100 to 2200.
Keywords: powder metallurgy, carbidic phases, mechanical properties, cutting edge, plasma nitriding

INTRODUCTION

Progressive powder metallurgy (P/M) technologies allow us to produce tools and parts under economically advantageous conditions, because of the extraordinary production-technological features specific to this branch. An important place among P/M materials is occupied by HSS, the industrial properties of which can be improved by suitable modification of chemical composition [1, 2, 3] and optimum thermal treatment [4, 5, 6]. The targeted production of HSS results in the development of fine-grained isotropic microctructure with uniformly distributed carbidic phases of size up to approx. 1 μm. The required structure of P/M tool steels is obtained by adding alloying elements such as: chromium (increases resistance to oxidation and corrosion), molybdenum, vanadium and tungsten (form carbides and increase hardness and resistance to red heat tempering). Cobalt is added to steel to improve their hardness at elevated temperatures. It increases secondary hardness but, on the other hand, decreases toughness and bending strength. It increases α to γ transition temperature, which results in increased resistance of α solid

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solution hardness reduction on heating simultaneously. It increases the transition rate of W and Mo from martensite to carbides at tempering by decreasing their solubility in the α solid solution [6, 7, 8]. Through this mechanism, cobalt supports the process of precipitation strengthening, increases secondary hardness, and improves thermal conductivity but decreases toughness. It produces no carbides but can penetrate into some of them and is present in α and γ solid. The influence of cobalt towards more uniform distribution of carbidic phases, which become smaller and more numerous and which give rise to a higher wear resistance is especially important [6-10].

The properties of HSS depend on the type and hardness of carbidic phases – primarily formed during solidification or secondarily eliminated from the solid solution during tempering. It is well known that the majority of primary carbides remain fixed in the final product, which can result in low fracture toughness and thermal-fatigue service life [7, 10].

The industrial properties of tools produced by P/M are higher by an order of magnitude [7, 11, 13] than the respective materials produced by conventional melt metallurgy (M/M).

EXPERIMENTAL PROCEDURE

Experiments were carried out on P/M HSS of AISI M2 type rapid solidification (RS), made via nitrogen atomozation of molten into nitrogen, and modified by 5% [3] and 8% of cobalt. Chemical composition of materials is summarised in Table 1.

Tab.1. Chemical composition of investigated materials in wt. %.

Mark	Material	C	W	Cr	V	Mo	Co
B	P/M AISI M2 + 5% Co	0.9	6.5	4.3	1.9	5.0	5.0
C	P/M AISI M2 + 8% Co	1.13	6.4	4.5	2.6	4.4	8.0

RS material was compacted by hot isostatic pressing (HIP) and subjected to the following heat treatment: AT - austenitizing temperature 1200°C / 20 min, oil quenching, tempering at 550°C for 1h / 3x water quenching [4, 5, 9]. The obtained samples were subjected to the following:
1. Microstructural analysis to evaluate the size and distribution of carbides phases,
2. Hardness,
3. Mechanical tests: notch toughness, resistance to abrasive wear and edge cutting life, the so-called frontal workability.

RESULTS AND DISCUSSION

The aim of production of high-speed cutting steels by P/M technology is to obtain uniform distribution of carbidic phases in the final structure of the material, which is the basic precondition for production of high-quality tools and parts. Special thermal processing of the compacted material provides microstructure consisting of primary, so-called solidification carbidic phases of an average size up to 0.5 μm, and secondary precipitates distributed throughout the martensitic matrix. The primary solidification phases grow from the melt during its rapid solidification and do not change their chemical composition during heat treatment, while secondary precipitates are formed in the solid solution during tempering [7, 13]. The basic material of high-speed steels should resist high temperature tempering, and hard carbidic phases in its matrix should be distributed in such a way so they increase the wear resistance to the highest possible degree. A high level of chemical homogeneity and isotropic structure predict a high quality of cutting tools [14].

Microstructural analysis

The microstructural analysis intended for evaluation of size and distribution of carbidic phases visualized by selective etching was carried out by image analysis of the final product using DIPS 5.0 equipment. The obtained and statistically processed data are presented in Tab.2. The data about quantity, size and area fraction of primary carbidic phases together with the hardness characterise the state of the final P/M product. The Table shows approx. 30% decrease in the number and 45% decrease in the mean of carbide particles in the material modified with 8% of cobalt. Graphic illustration is presented in Fig.1. Despite a decrease in the area fraction of carbides by 61%, an increase in HRC hardness of material C was observed. This was caused by the existing primary carbidic phases that had formed during solidification and by the secondary or precipitation hardening during tempering, as it was reported in some studies [7, 15]. The state of material C determined the shape of its frequency curve, i.e. its sharpness and dispersion variance (Fig.1.).

Tab.2. Size and area distribution of carbidic phases and hardness of the final materials.

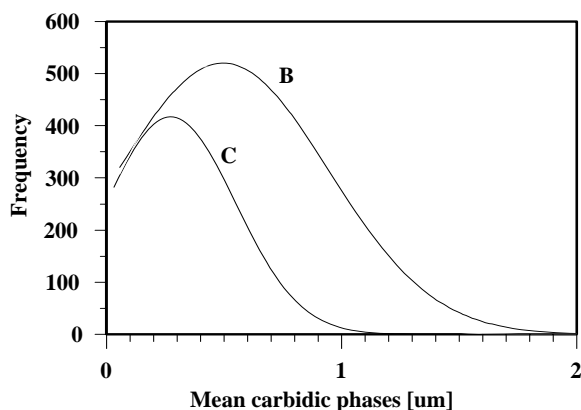| Specimen	Number of carbidic phases	Mean of carbidic phases [μm]	Area fraction of carbidic phases [%]	HRC	HV30 pri T[°C]	
B	2116	0.4964	8.8	64	839	20
					856	100
					793	200
					766	300
					668	400
C	1465	0.2730	3.4	65	915	20
					902	100
					879	200
					713	300
	- 30%	- 45%	- 61%		735	400

Fig.1. Frequency curves of average diameter (mean) of carbidic phases.

Hardness of material

HRC at room temperature and HV 30 at elevated temperatures was evaluated on specimens in the final state prepared for mechanical testing. The values in Tab.2 and Fig.2 show an increase in HV 30 hardness of the material modified with 8% Co (C) and smaller differences in HV 30 of material B with increasing temperature. The hardness decreased with increasing temperatures due to structural changes in the material related to transformation of the residual austenite, carbide-forming reactions, and volumetric changes.

Mechanical tests

Notch toughness

Notch toughness should be related to hardness and microstructural characteristics of the material tested. It is a property of special importance for cutting characteristics at intermittent cutting and can be used in correlation with resistance to wear. With increasing hardness of materials of this type their brittleness increases due to the presence of carbidic precipitates that initiate failure in micro-volumes.
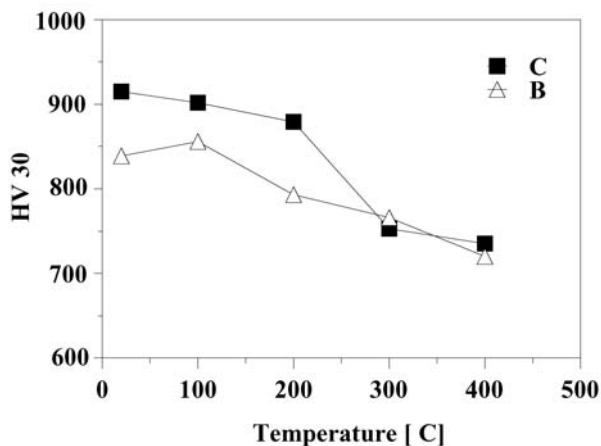

Fig.2. HV 30 hardness of final materials at elevated temperatures.
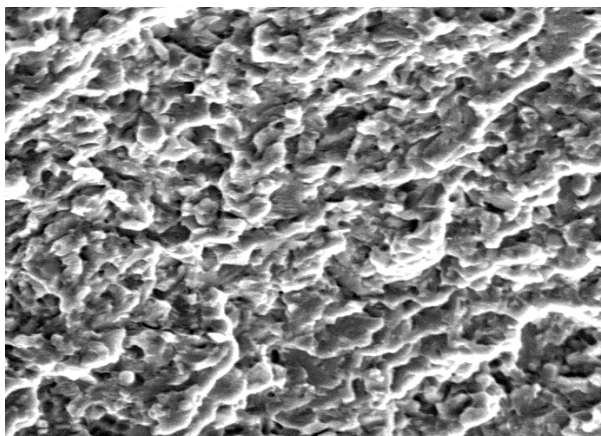

Fig.3. Typical fracture surface of P/M material after the impact test.

Standardized specimens with R 2.5 notch were subjected to impact test using a 15 kg load. The values of toughness KC_v presented in Tab.3 demonstrate its significant increase with increasing cobalt content. Fig.3 illustrates typical fracture surface of P/M material after the impact test. The fracture surface exhibited quasi-cleavage fracture with transition to plastic failure of a pit-like character. The failure occurred in the zone of potential microstructural heterogeneities with interdendritic segregation during solidification, and in locations containing hard primary MC-type carbides as it was also reported in [6, 7].

Resistance to abrasive wear

High-speed tool steel (SN 19 830, or P/M AISI M2 HSS) should comply with some criteria that are used to evaluate its industrial properties. One of them is the wear resistance. It is the important material property of considerable technical and economical impact affected by the character of the material surface [8, 15]. The specimen was tested for wear by putting it into contact with abrasive cloth over the pre-set abrasive path on a laboratory instrument APGi (VEB Leipzig) in order to determine the relative resistance of the respective materials to abrasive wear according to SN 01 5084. Equivalent steel produced by conventional melt metallurgy M/M was used as a reference material.

K_{AO} - coefficient of resistance to abrasive wear was calculated as follows:

$$K_{AO} = \frac{(m_0 - m_1)_{RS}}{(m_0 - m_1)_{TS}} \cdot \frac{\rho_{TS}}{\rho_{RS}} = \frac{\Delta m_{TS}}{\Delta m_{RS}} \cdot \frac{\rho_{TS}}{\rho_{RS}} \qquad (1)$$

where:
TS - tested specimen
RS - reference specimen
m_0 - initial weight of the specimen [g]
m_1 - weight of the specimen subjected to wear [g]

Table 3 shows the obtained values of relative wear K_{AO} calculated from mean weight losses of the materials tested. The most suitable was the material B which exhibited the lowest weight loss Δ m and the values of relative wear K_{AO} after the test.

Tab.3. Notch toughness KC_v, specific weight ρ, weight loss Δm and coefficient of relative abrasive wear K_{AO} of investigated materials.

Material	KCv [J.cm^{-2}]	ρ [g.cm^{-3}]	Δm [g]	K_{AO}
B	22.7	7.95	0.0435	1.109
C	24.4	7.77	0.0456	1.034

reference material M/M:**:** ρ -7.58 g.cm^{-3}, Δm – 0.0460 g

Cutting edge durability

The respective materials were subjected to long-run test of durability of the cutting edge. The so-called frontal workability test according to ISO 3685-1977 for the machining of specimens of dimensions 12x12x6 mm, uses complete loss of cutting ability as a criterion of wear. A test of this type appears effective when one has to compare several cutting tools. Steel 12 050.1 that, according to SN 41 2050, corresponded to ISO 683/1-87 requirements [16], was used as material intended for machining. To compare the excellent

cutting properties of P/M tools, Fig.4 also shows the values for tools produced by conventional melt metallurgy (M/M). Experimental validation showed that the tip of the tool produced by M/M technology was losing its shape during machining, which resulted in decreasing cross-section of chips and load on the cutting material tested. The graphic illustration shows that the tools produced by P/M technology were unambiguously better, almost by 100%, with regard to preservation of shape of the cutting edge.

The final fine-grained isotropic structure of the P/M materials with carbidic phases ranging from 0.3 to 0.5 μm, results from their production route using cobalt as a modifier of the chemical composition. Their excellent cutting properties correspond well with their toughness and wear resistance.
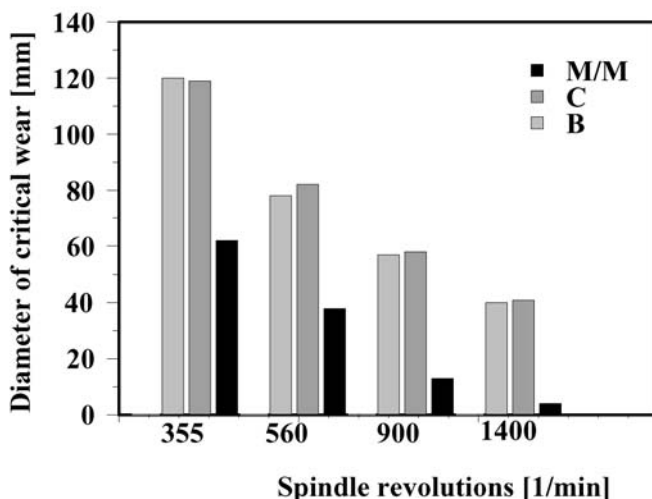

Fig.4. Comparison of investigated materials with regard to the maintenance of the required shape of the cutting wedge.

Increasingly, higher stress has been laid on the quality improvement of cutting tools. It is almost impossible to develop a material that would comply with all the requirements, high hardness and strength, high toughness at low and elevated temperatures, high resistance to wear, and high chemical stability. By suitable selection of surface treatment of the material - cutting tool, e.g. chemical-thermal processing, implantation of ions beneath the cutting surface, stellite welding-on, coating, etc., the cutting properties can be improved considerably [17, 18]. The most frequent up-to-date world-wide technical approach is the ionic saturation of steel surface with nitrogen [17]. The investigated P/M tools were subjected to experimental pulse plasma nitriding by means of a Rűbig GmbH equipment. The nitriding parameters are presented in Tab.4.

Tab.4. Parameters of nitridation of individual specimens.

Temperature [°C]	Composition of atmosphere $N_2:H_2$	Time of nitridation [min]
470		
500	1 : 3	120
530		

The thickness of the nitride layer was measured under a light microscope Neophot 21, and the hardness profile from the nitridated surface toward the specimen core was measured by a LECO micro-hardness tester. The thickness of nitride layers of specimens subjected to pulse plasma nitriding ranged between 17-50 μm, depending on nitridation parameters and chemical composition of the material. Figure 5 shows the microstructure of the nitride layer of specimen C produced at 530°C over 120 min.
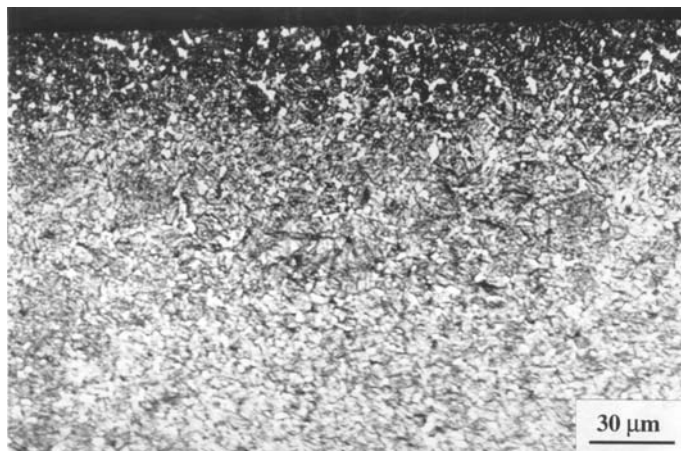

Fig.5. Microstructure of the nitride layer of P/M material.

On the basis of X-ray diffraction phase analysis [19] the following was determined:
- the dominant matrix phase was γ-Fe enriched with a phase containing a certain proportion of alloying additions,
- the matrix contained most likely a carbide of M_6C type or an intermetallic phase,
- along with the dominant γ-Fe also α-Fe was found in the nitride layer,
- none of the specimens contained any nitride phases. We assumed that nitrogen did not form separate nitrides in the nitride layer but was present in the form of a solid solution.

It appeared necessary to confirm or define more accurately the results of X-ray diffraction phase analysis using an additional analytical method, e.g. auger electron microscopy (AEM), secondary ion mass spectroscopy (SIMS) and scanning electron microscopy (SEM).

Figures 6a, b show the microhardness profile in a direction from the nitridated surface to the specimen core. It is evident that the presence of cobalt in specimens of high-speed P/M steel affected microhardness values. Comparison of the depth of nitrogen diffusion and the thickness of surface layer with increased microhardness indicates a more uniform distribution of microhardness at 530°C.

In future studies the specimens can be subjected to two-stage ion nitriding to obtain nitride layers of higher thickness and toughness, but the same hardness.
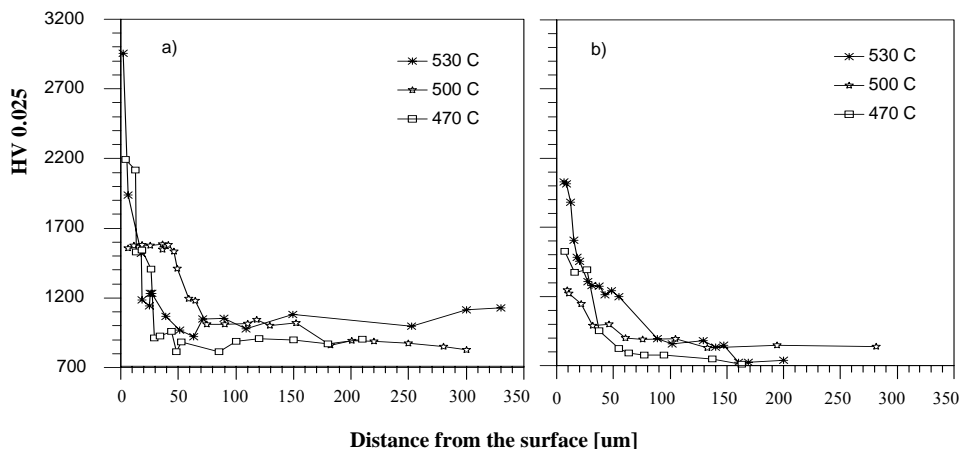
Fig.6. The microhardness profile starting from the nitridated surface of specimen B (a) and specimen C (b)

CONCLUSION

On the basis of microstructural analyses and the tests performed on materials produced by P/M AISI M2, modified with 5 and 8% of cobalt and subjected to hot isostatic pressing and special thermal processing, the following conclusions can be drawn:

1. The data obtained by image analysis and processed by statistical methods characterise the state of the final P/M product. The increase in addition of cobalt to 8% resulted in approx. 30% decrease in the quantity, 45% decrease in diameter and 61% decrease in area fraction of primary carbidic phases in favour of material C.

2. By increasing the temperature, the hardness of material decreases due to structural changes in the material related to transformation of residual austenite, carbide-forming reactions and parallel volumetric changes. Smaller differences in HV 30 hardness with increasing temperature were observed in material B.

3. The addition of 8% Co increased notch toughness by about 7% compared to the material with 5% Co. The fracture surface indicated quasi-cleavage fracture with transition to plastic failure of a pit-like character. The failure occurs in the zone of potential microstructural heterogeneities and interdendritic segregation, and in locations with hard primary carbides.

4. The tests of resistance to abrasive wear performed on respective P/M materials shown at material B was more suitable as both weight loss Δm and relative wear K_{AO} (Tab.3) were lower for this material.

5. Experimental validation of cutting properties of the produced tools showed that tools produced by P/M technology (particularly material B) were unambiguously better (almost by 100%), with respect to preservation of shape of the cutting wedge, than the tools produced by M/M. The tip of the reference tool produced by M/M technology was losing its shape during machining, which resulted in decreased cross section of chips and decreased loading on the investigated cutting material.

6. The surface treatment, plasma nitriding, causes a considerable increase in hardness of layer of thickness up to 17 to 50 μm with dependence on nitridation parameters and chemical composition of the material.

7. No nitride phase was observed in specimens. We assume that nitrogen does not form separate nitrides in the nitridated, layer but it is present in the form of a solid solution. Additional analytical method, e.g. microanalysis and scanning electron microscopy should be used for a better explanation of results.

8. Comparison of P/M materials shows higher values of microhardness (25 g loading) of material B compared to those of material C. This was due to the decrease of quantity, size and area fraction of primary carbidic phases in material C. Comparison of the depth of nitrogen diffusion and the thickness of surface layer with increased microhardness indicates a more uniform distribution of microhardness at 530°C.

9. The material subjected to nitridation under selected conditions complied with the requirements on hardness, and thus we can recommend additional experiments with the selected products and subsequent testing by recommended tests.

In conclusion we can summarise that P/M materials, with regard to their excellent industrial properties, particularly resistance to wear and cutting characteristics, can be recommended as a replacement of materials produced by melt metallurgy for use as tools for machining of refined steel with $R_m > 1400$ MPa and annealed steel with $R_m < 900$ MPa [16]. Some mechanical properties evaluated in this study were higher in material B [3] compared to material C, known as vanadis 30 (or ASP 30), i.e. material with a lower addition of cobalt. We plan to investigate further the respective P/M materials within the projects VEGA 2/2115/04 and international project EUREKA 2708 UPLETOOLS in the period 2002-2004, focusing on optimisation of parameters of nitridation of the surface layers, and on their testing under operational conditions.

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