



Application of polymer-powder slurry for fabrication of abrasion resistant coatings on tool materials

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ABSTRACT

Purpose: Development of a new generation tool materials on the basis of M2 high speed-steel or 41Cr4 steel covered with the carbides. Application of pressureless forming of powder as a manufacturing method of anti-wear coatings gives the possibility to produce this materials with relative low cost of production.

Design/methodology/approach: Powder metallurgy, pressureless forming of powder, sintering, microstructure examination, X-ray dispersive energy examination, hardness examination.

Findings: Putting down coatings with this method does not call for using the costly equipment for the physical or chemical deposition of coatings from the gaseous phase. Coating thickness may be easily regulated by applying the powder-binder slurry layer once or several times on the prepared substrate surface. Hardness of coatings in the sintered state is higher compared to the HS6-5-2 and 41Cr4 steels by about 400 and 700 HV respectively. It is expected that hardness of the coatings and substrate will grow after their heat treatment.

Practical implications: Application of powder metallurgy and especially pressureless forming of powder to manufacturing of steel covered with anti-wear coatings gives the possibility to obtain tool materials with the relative high ductility characteristic of steel and high hardness and wear resistance typical for cemented carbides.

Originality/value: One can state, based on the investigations carried out, that the pressureless forming of the powder may be used for depositing the anti-wear coatings onto the tool materials and other elements in the abrasion wear service conditions.

Keywords: Powder Metallurgy; Powder Injection Moulding; Feedstock; Tool materials

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MATERIALS MANUFACTURING AND PROCESSING

1. Introduction

Tribological processes occur both in the tool - machined material system, as in any other system composed of two mating

surfaces. The wear rate of the mating surfaces decides oftentimes the life of the entire assembly or its particular elements that should be regenerated or replaced with the new ones. Economic costs connected with it compel to search for solutions increasing the abrasion wear resistance of the mating elements.

Undoubtedly, tool materials for machining and plastic working are subject to the most intensive wear processes resulting from the specifics of their service. High-speed steels used for cutting tools from which the high abrasion wear resistance and relatively high ductility are required are the particular example. These steels have mostly been replaced with sintered carbides, cermets, and hard tool ceramics which is decidedly more abrasion wear resistant [1,2]. Regrettably, these materials are characteristic of high brittleness and they cannot be used everywhere, especially in case of the worn out machine tools. High-speed steels and sintered carbides with the relatively high cobalt content seem to be still irreplaceable in this area. Properties of the high-speed steels may be significantly improved fabricating them with the powder metallurgy methods instead of the conventional casting, which makes elimination possible of the typical flaw in the conventional steel structure like the band-like segregation of the primary carbides. Employment of powder metallurgy, especially of the contemporary methods of forming the powders improves the service properties of the high-speed steels [3-17]. However, application potential of this material is still limited. This can be changed forming the tool surface with the relevant technology, which significantly extends their life and resistance to their degradation processes. Properties can be also changed by putting down the durable coating material on the surface. Coatings used most often are developed in the processes of physical or chemical deposition from the gaseous phase [18-23]. In this case, the substrate material has to be relatively hard, so that no deformation of the tool and decohesion of the thin coating would occur. Another decohesion reason are stresses occurring between the substrate material and coating developing in its deposition process and also adhesion of the coating to the substrate. These problems can be eliminated using methods of pad welding or remelting the tool surface with the welding techniques [24-26]. Surface hardness in this case is lower than in the PVD and CVD case, yet there are no so high stresses on the boundary between the coating and the substrate, and structure in this zone is often of the gradient character.

Searching for the new solutions of forming the surface layers or depositing the protective coatings to improve the abrasion wear resistance of tools the proposal is put forth in the paper for the pressureless forming of powder on surface of the ready tools and sintering to obtain the compact coating surface and high adhesion to the substrate. It seems that this technology may be also used for regeneration of tools in future, which is undoubtedly interesting from the economic point of view.

2. Materials and research methodology

Various binder types, presented in Table 1, were experimentally selected for fabrication of the anti-wear coatings. The main task of the binder is making it possible to form the powder and facilitating the powder sintering process. The binder-powder slurry was mixed by hand until the uniform mixture was obtained. The slurry was sprayed onto the prepared surfaces, put down by brush or spatula depending on the binder viscosity and its portion. The 316L, M2, T15 steels powders and a commercial carbides powder mix (tetra carbides) composed of WC, TiC, TaC, and NbC (33.3/33.3/26.6/6.8% in vol.) were used as the

main coating constituent. Table 1 presents the coatings used in detail - binder type, substrate material, and designation method for the investigated materials. In case of the test piece in which resin with T15 and carbides powder were used as a coating put down onto the high-speed steel the test piece designation will be as follows - 3/5/2 (Fig.1). The powders used are presented in Figures from 2a to 2d. Powders designations are according to ASTM standard because of their frequent use in foreign literature and commerce. Moreover, it helps to differentiate designations of the coating and substrate, as the M2 steel is the equivalent of the HS6-5-2 steel according to the pertinent standard. The 316L corrosion-resisting steel powder was added due to the high content of chromium which improves wettability at the high sintering temperature. The HS6-5-2 high-speed steel and the 41Cr4 heat-treatable steel were used as substrate material. Substrate material was acquired as square bars 40x20 (HS6-5-2 steel) and 30x16 (41Cr4 steel). The bars were cut into 5 mm thick test pieces and next they were ground and their surfaces were rinsed with acetone.

In case of materials for which acryl resin was used as binder the test pieces were put into the furnace for polymerization of the resin at the temperature of about 115°C. The mixtures in which the liquid glass and lacquer were used were dried at the ambient temperature, whereas, the polymer-powder slurry, fabricated using paraffin, was heated at temperature of 70°C and put down on to the previously prepared surface of the test pieces. Test pieces were also heated to temperature of about 70°C in the chamber furnace to make spreading the coating easier. In case of the binder like paraffin and polypropylene the powder painting technique was used for coating deposition and the substrate material was heated up to temperature of 250°C. Test pieces prepared in this way with the coatings put down were put into the pipe furnace and sintered in the atmosphere of the flowing N₂-10%H₂ mixture of gases at the temperatures of 1150, 1240, 1260, and 1280°C. The binder thermal debinding process proceeded during heating up to the sintering temperature because of the coatings thickness that did not exceed 1.5 mm. The longer heating up time and the isothermal hold at the temperature of 400°C for 1 h were used only in case of the thermosetting binder because of the threat of developing cracks in the coating.

Microsections were prepared on the transverse sections of the test pieces for assessment of structures of the coating and zone between the matrix material and coating, and also of the matrix structure. Moreover, porosity was examined on the Axiowert 405M Light microscope equipped with the LeicaQwin image analysis system. Coating hardness was tested on the FM-700 microhardness tester. Structure of the developed coatings were examined with SUPRA 25 scanning electron microscope (SEM) equipped with X-ray energy dispersive spectrometer (EDS).

3. Results and discussion

It was found out based on the preliminary examinations with the unaided eye of coatings deposited with the pressureless forming method onto the substrate that the different binders employed fulfil their main task and the coatings in the sintered state cover the substrate material. Local cracks and delaminations

occur in coatings in which the thermosetting resin was used, which is connected with the thermal debinding of this binder. Preparation of coatings in which paraffin, paraffin with polypropylene, or acrylic resin were used is more complicated from the technological point of view compared to coatings in which lacquer or liquid glass were used. This is because using the acrylic resin requires the preliminary holding of the test pieces at the temperature of about 115°C for polymerization of the binder. Using paraffin one has to heat up the polymer-powder mixture to put it down onto the substrate material and if paraffin (PW) with polypropylene (PP) are used then the substrate material should be heated up to 250°C and next the mixture of the powders with the binder should be put down onto the substrate.

Substrate temperature of about 250°C is higher than the softening temperature of the polymers used which results in softening the binder and covering the test piece substrate surface forming the compact and tight coating. Only the coating surface is characteristic of a high roughness which is connected with the low portion of the binder in respect to the powder being 30% by volume.

Examinations of structure on the scanning microscope revealed that using the liquid glass as a binder results in its presence in the coating after sintering. Structure of such coating is shown in Figure 3. Big areas rich in Si indicate that liquid glass is not subject to debinding during heating up to the temperature of

sintering and holding at this temperature in the N₂-10%H₂ atmosphere which renders it impossible to use it as a binder for fabrication of coatings with the pressureless forming method. In case if lacquer used as a binder many pores may be observed in the coating structure. This is caused, most probably, by evaporation of the solvent and by the fast and total thermal debinding of the lacquer, which does not leave on the surface the carbon powder initiating the process of sintering and compacting powder in the coating.

In spite of many pores no decohesion was revealed between the substrate and coatings for which lacquer was used as a constituent making their forming possible (Fig. 4). In case of using the thermosetting resin the carbon concentration occurs by about 0.5% in respect to the carbon concentration in the mixture of the input powders. The incomplete binder degradation causes that carbon remaining on the high-speed steel powder surface initiates the sintering process by diffusion to the powder grain surface layer and reduces the sintering temperature which initiates the sintering process. Analysing the effect of binders like lacquer and resin on the coating structure mixture of both binder constituents was used in the succeeding investigations.

Therefore, the solvent from the lacquer evaporates and opens pores which make fast degradation of lacquer and resin possible. Increase of carbon concentration in coating in the sintered state is only 0.3% in respect to carbon concentration in the input powder.

Table 1. Employed materials and method of designation of the test pieces

Mark position	Binder type			Binder type		
	Liquid glass	Lacquer	Thermosetting resin	Paraffin wax	Paraffin wax + Polypropylene	Lacquer + Thermosetting resin
First	1	2	3	4	5	6
	Single layer			Multilayer		
Second	1	2	3	4	5	
	316L	M2	T15	316L	T15	
				M2	T15+5% tetra carbides in volume T15+10% tetra carbides in volume T15+15% tetra carbides in volume	
Third	Substrate type			Substrate type		
		1			2	
		41Cr4			HS 6-5-2	

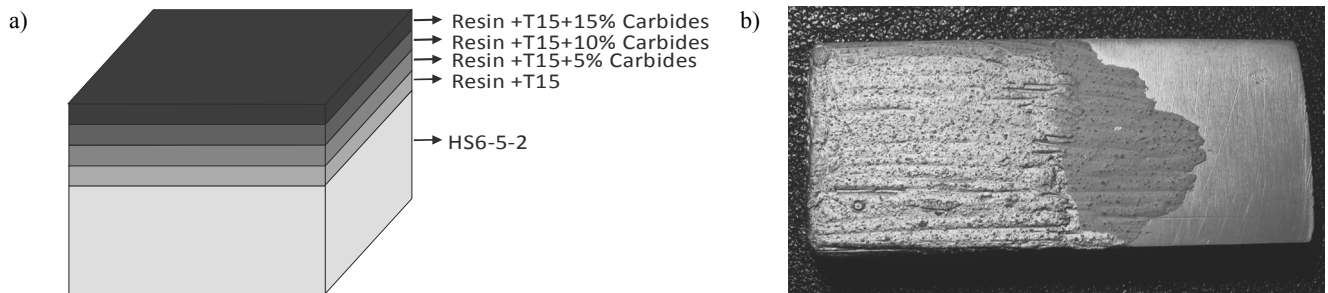


Fig. 1. a) Scheme of samples marked as 3/5/2 - conventional HSS covered with four different layers, b) view of the samples after sintering

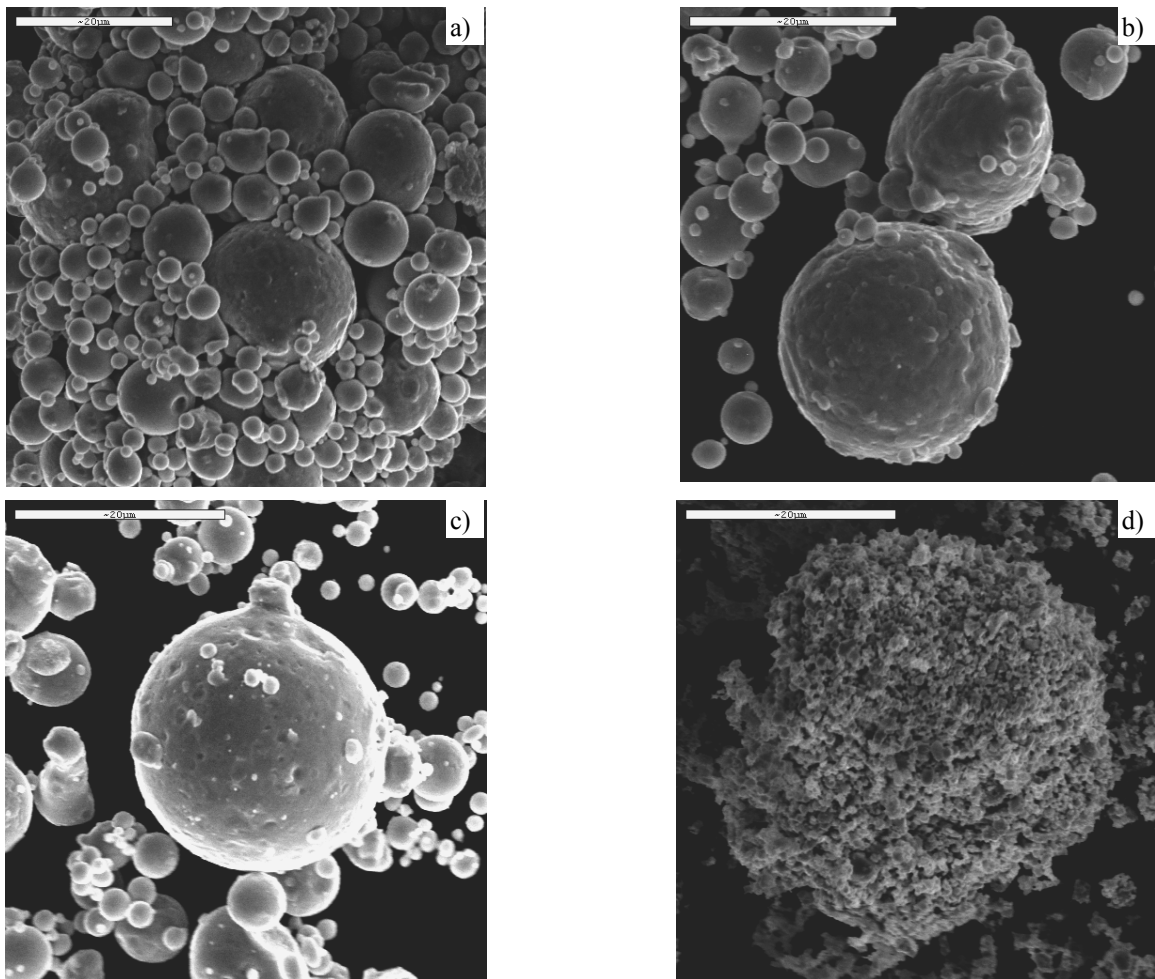


Fig. 2. Powder of a) M2, b) 316L, c) T15 and d) tetra carbides agglomerate

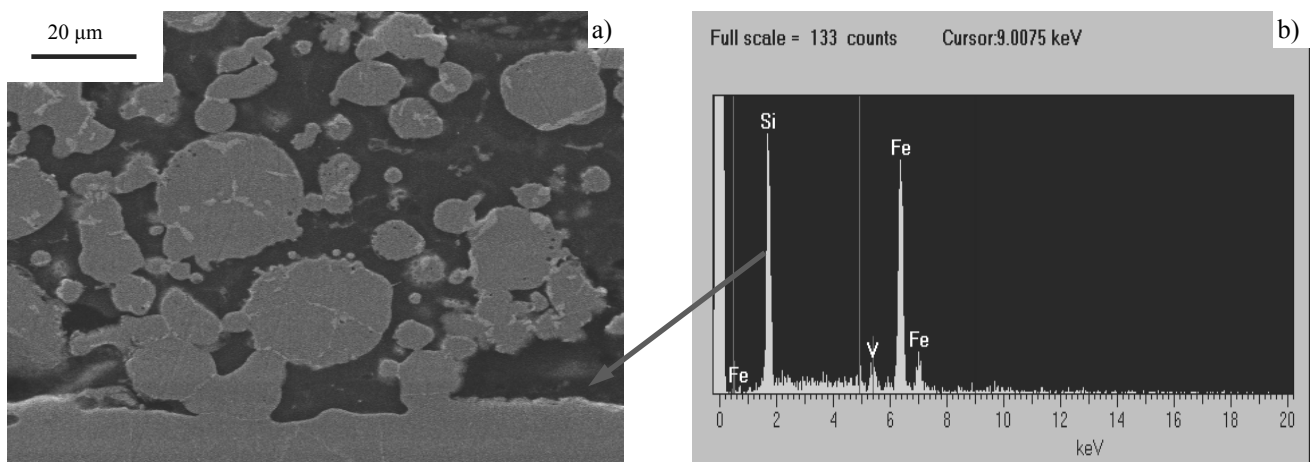


Fig. 3. a) Structure of 41Cr4 type steel covered with powder of M2 mixed with liquid glass sintered at 1240°C, b) plot of the X-ray dispersive energy phases of marked area from Figure 3a

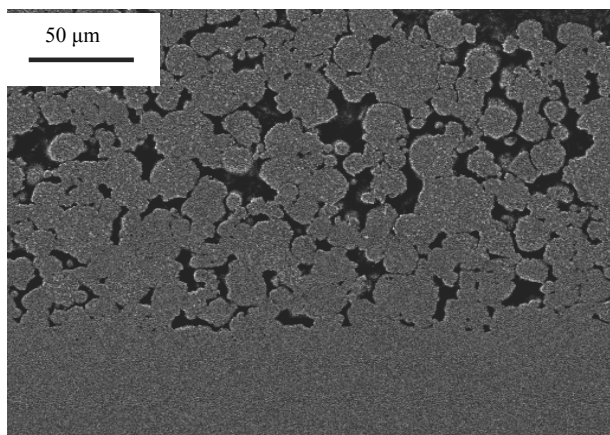


Fig. 4. Structure of 41Cr4 type steel covered with powder of 316L mixed with lacquer sintered at 1150°C

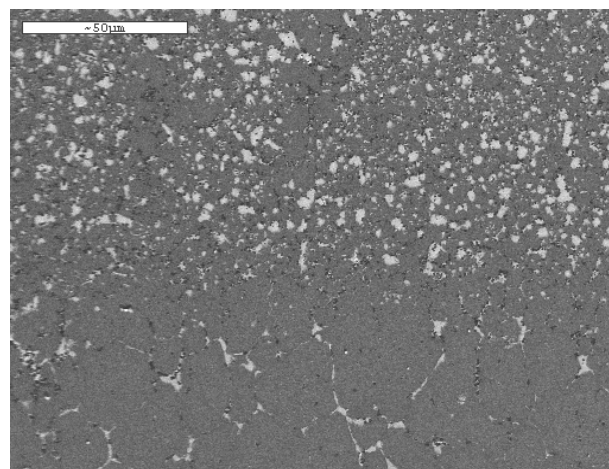


Fig. 6. Structure of HS6-5-2 type steel covered with powder of T15 mixed with PW sintered at 1280°C

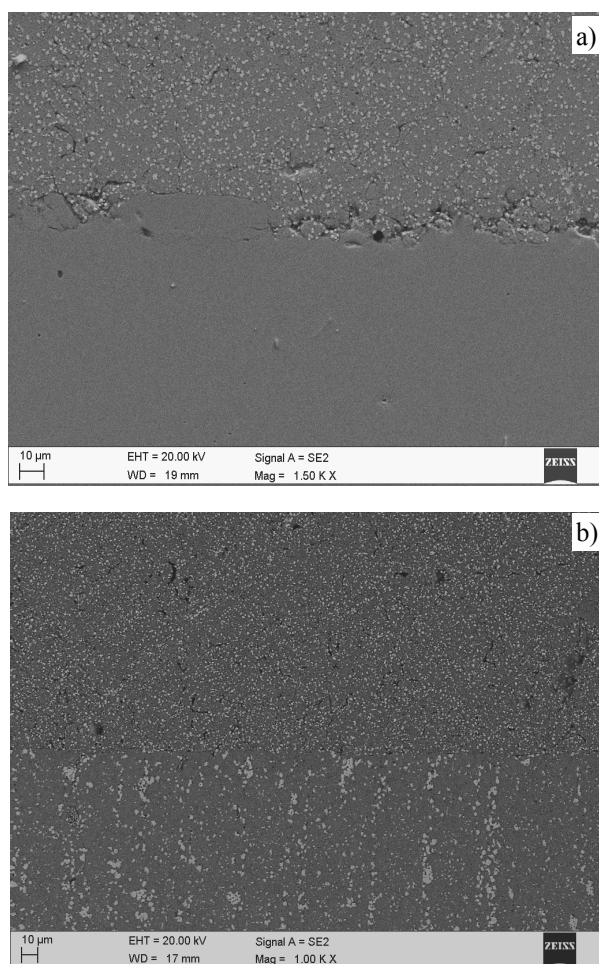


Fig. 5. Structure of samples sintered at 1240°C, a) 41Cr4 type steel covered with powder of T15 mixed with resin b) HS6-5-2 high speed steel with powder of T15 mixed with resin

The 316L corrosion resistant steel powder was used as the interlayer due to high chromium content in this steel. Due to the high chromium wettability during sintering, this element should increase the adhesion of the coating to the substrate. It was found out, based on examinations of coatings from the 316L and M2 steels sintered at the temperature of 1150°C on the scanning microscope, that both coatings tightly adhere to the substrate, therefore the 316L steel powder was not used in the succeeding investigations. The boundary is shown in Figure 5 between the substrate material and coating from materials sintered at temperature of 1240°C. No decohesion was found both in case of the 41Cr4 steel covered with M2 coating and for the HS6-5-2 steel covered with T15 coating. The surface portion of pores is above 3% due to the low sintering temperature.

Moreover, the coating structure is characteristic of the uniform distribution of carbides in the alloy iron matrix. The substrate structure in Fig. 5b is characteristic of the conventional high-speed steel and represents the band segregation of the primary carbides. Because of the coatings thickness that did not exceed 1.5 mm thermal debinding was carried out during heating up to the sintering temperature. No delamination were observed in spite of the high heating rate of about 10°C/min to the sintering temperature.

The exceptionally uniform structure with a low porosity of about 0.6% of the surface portion of the coating pores was obtained after using paraffin as a binder and sintering at temperature of 1280°C. The T15 high-speed steel powder used constitutes after sintering a homogeneous and uniform coating tightly adhering to the surface from the HS-6-5-2 type high-speed steel substrate (Fig. 6). The relatively high sintering temperature causes a slight growth of carbides in the coating, however, does not cause their partial melting. Also in the substrate material no partial melting of carbides was revealed and their band segregation is a typical structure defect occurring in the cast high-speed steels.

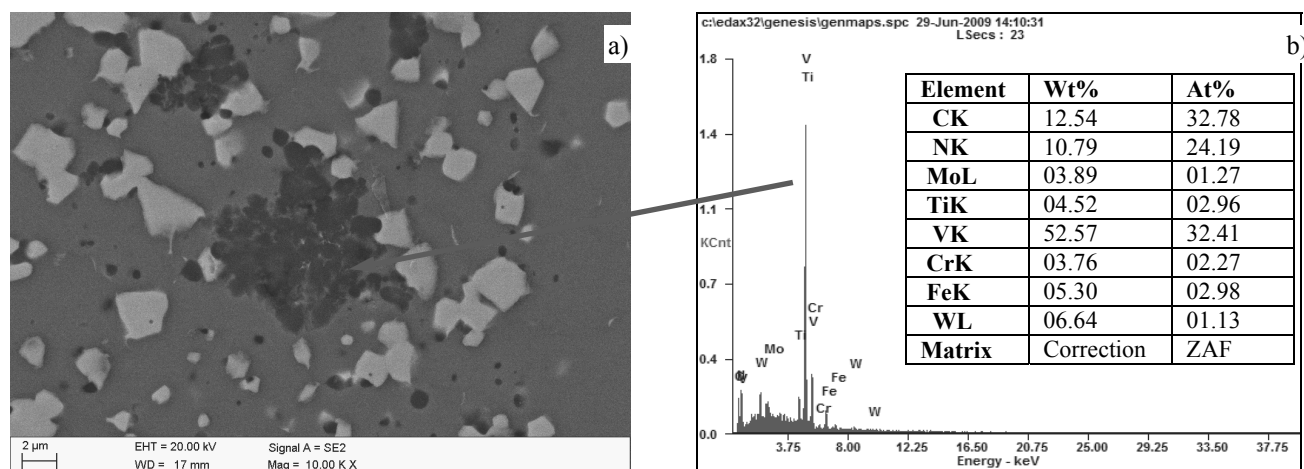


Fig. 7. a) Structure of surface layers of HS6-5-2 covered with T15 mixed with PW and sintered at 1280°C, b) plot of the X-ray dispersive energy phases of marked area from figure 7a

On the other hand the interface zone between coating and material substrate seems to be interesting. This zone is the surface layer of the substrate in which, most probably, the significant diffusion occurred of carbon from the coating, reducing the solidus temperature. Its effects are the local partial melting of carbides and numerous carbides precipitating on the primary austenite grains boundaries rich in W, Mo, and Fe. Also in the coating phases rich in W, Mo, and Fe occur originating, most probably, the M_6C carbides, however, their shape is close to spherical and size does not exceed 10 μm . Numerous occurrences of carbonitrides rich in V originating during sintering in the N_2 -10% H_2 atmosphere can be also observed in the coating structure. These phases, visible as dark spherical precipitations whose size does not exceed 2 μm , occur especially in the coating structure which is porous at lower sintering temperatures, which facilitates nitrogen diffusion. It was found out based on the substrate structure observations, i.e., of the HS6-5-2 conventional high-speed steel that in the surface layers the dark precipitations occur rich in V and N, most probably of the MX type, which attests the nitrogen diffusion into the substrate material and precipitation of carbonitrides in it (Fig. 7). Hardness testing of coatings from the T15 high-speed steel deposited onto the HS6-5-2 conventional steel revealed the coating hardness increase by about 2 HRC in respect to the substrate hardness after sintering at the temperature of 1280°C.

It is expected that after heat treatment the coating hardness will be also higher in respect to the substrate by at least 2HRC. It was found out based on the microhardness testing of the gradient coating in which the T15 steel was reinforced additionally with carbides and of the substrate, i.e., the HS6-5-2 steel that hardness grows from about 600HV to about 1000HV. That high hardness growth is caused by the significant volume portion of carbides in the coating surface layer amounting to 20%. Coatings put down onto the 41Cr4 heat-treatable steel are characteristic of the highest hardness growth in respect to the substrate due to the low hardness of the substrate itself. Substrate hardness is about 300HV, whereas, hardness of the gradient coating is about 1000HV like in the previous case. Figure 8 shown the distribution of hardness from surface to substrate of HS6-5-2 covered with T15 mixed with PW and sintered at 1260°C.

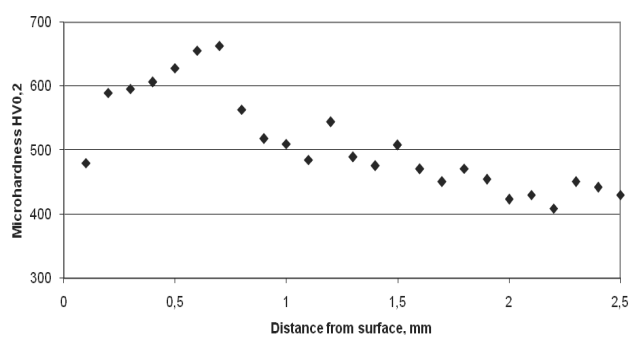


Fig. 8. Microhardness of HS6-5-2 high speed steel covered with T15 mixed with PW and sintered at 1260°C

4. Conclusions

One can state, based on the investigations carried out, that the pressureless forming of the powder may be used for depositing the anti-wear coatings onto the tool materials and other elements in the abrasion wear service conditions. Putting down coatings with this method does not call for using the costly equipment for the physical or chemical deposition of coatings from the gaseous phase. Coating thickness may be easily regulated by applying the powder-binder slurry layer once or several times on the prepared substrate surface. Moreover, the substrate surface does not require finishing grinding and polishing as in case of the PVD coatings deposition. Employment of this powder metallurgy method may be in future of a significant importance thanks to the possibility of regeneration of the worn out tools. Regrettably, the technological limitations resulting from the small chamber volume of the heating equipment do not let to use this method for regeneration of the large-size tools, which is desirable just in case of these tools due to their high manufacturing cost. The detailed investigation results revealed that lacquer and paraffin are the optimum binding agents used for

the pressureless forming due to the simple coating forming technological process, their structure after sintering, no delaminations and cracks and the low pores portion in the sintered state, which does not exceed 1%. Hardness of coatings in the sintered state is higher compared to the HS6-5-2 and 41Cr4 steels by about 400 and 700HV respectively. It is expected that hardness of the coatings and substrate will grow after their heat treatment.

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