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Structure of sintered gradient tool materials

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Properties

ABSTRACT

Purpose: Investigation of influence of sintering conditions onto the structure of composite gradient tool materials. The chemical composition of the investigated materials' core was corresponding to the M2 high-speed steel and was reinforced with the WC and TiC type hard carbide phases with the growing portions of these phases in the outward direction from the core to the surface.

Design/methodology/approach: Powder Metallurgy, SEM, X-Ray Microanalysis.

Findings: Changing of technological properties and structure of composite gradient tool materials (depending on the compositions of used mixtures and sintering temperature) was analyzed and described.

Practical implications: Tool materials used especially for cutting tools and plastic forming tools.

Originality/value: Providing of high properties characteristic of cemented carbides keeping high ductility characteristic of steel was achieved.

Keywords: Sintering; Composite materials; Gradient materials; Tool materials

1. Introduction

Recently functionally graded materials (FGM) became one of most progressive branch of engineering materials. Gradually changing properties of engineering materials, including tool materials is possible due to changing of chemical or phase composition, or atomic configuration. Continuous or step by step changing of materials structure causes higher mechanical properties, abrasive and chemical wear resistance and resistance to elevated work temperature in comparison with traditional tool materials. Inhomogeneity of FGM structure occurs usually in one assumpted direction determined by further application of materials [1-4].

Working out and producing of sintered gradient tool materials was a major step forward in area of sintered tool materials development. Modern sintered gradient tool materials have to meet high functional requirements especially by keeping high properties at evaluated temperature combined with high pressure per unit area.

Powder of M2 (HS6-5-2) type high-speed steel used as matrix of experimental gradient materials, was chosen purposely owing to secondary hardening effect occurring in heat-treated state of this kind of material. This effect should be a reason of additional hardening of developed materials' matrix. M2 (HS6-5-2) type high-speed steel is characterized by relatively low content of alloy additives in comparison to most of other high-speed steels, decreasing material cost of production, so it is often used in researches, leaded in area of powder metallurgy [5-9]. Important disadvantage of chosen matrix steel is very small sintering window range of ~5ºC, causing that there is necessary to use very precise heating equipment characterized by high temperature stability. Sintering window and sintering point depends on the method of moulding, powder's grain size, sintering atmosphere as well as many other factors (kind of used sliding medium or activators of sintering process). Basing on preliminary own researches it was found that the most interesting results can be achieved by sintering developed materials in nitrogen-hydrogen gas atmosphere [10, 11].

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TiC and WC carbides, chosen as hard reinforcing phases, are often used commercially for reinforcing tool materials like carbide alloyed composites or cemented carbides. The obvious fact is, that in cemented carbides most used material for matrix is cobalt, because of its high wettability of WC carbide level during liquid phase sintering. However in case of using high-speed steels containing chromium, wettability in high temperature is also high [12].

2. Experimental procedure

The investigations were carried out on the samples made with use of unilateral uniaxial moulding, sintered in the pipe furnace in protective atmosphere. The Höganas' M2 type high-speed steel powder, atomized with water and annealed was used as matrix. Shape of used powder is shown in Figure 1.

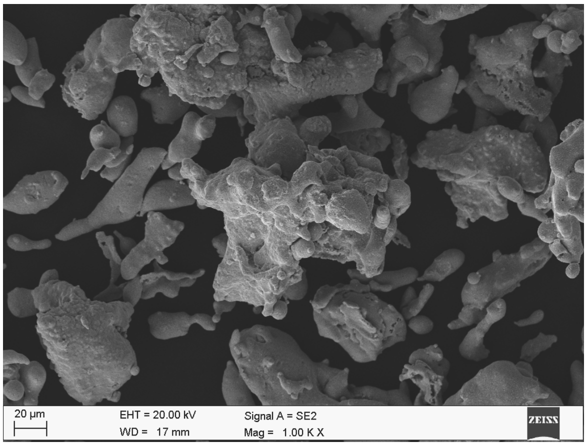

Fig. 1. Powder of high speed-steel M2 type atomized by water

The powder used for matrix is characterized by developed surface and high plasticity, what makes it very suitable for uniaxial compacting in locked die. Titanium and tungsten carbide powders was used as the reinforcing phases (Figures 2 and 3). Technological properties of used powders and its mixes witch maximal fraction of reinforcing phases amounted to 32% are introduced in Table 1.

Investigations of flow rate and bulk density of powders were carried out in accordance with standard with use of Hall flowmeter. Powders were mixed without addition of lubricant in Turbula mixer during 1h. The example of mixture M2-32%WC is shown in Figure 4.

The powder mixes were moulded in a die pouring it in 5 sequenced layers with the successive lots of mixes with the growing content of reinforcing phases, cut-and-fill balancing each time the surface of a layer about 0.5 mm thick. Thickness of the bottom layer, containing pure high-speed steel powder or in some variations high-speed steel powder with minimal addition of reinforcing phases featuring simultaneously the gradient material core, was about 2 mm. The aim of such composition was decreasing of distortion during sintering process. Layer with maximum fraction of reinforcing phases, as characterized by the least moulding capability, was poured as last one to obtain the maximum pressure in area just under the punch during uniaxial moulding process. Fractions of WC and TiC powders in particular layers of developed gradient materials are shown in Table 2.

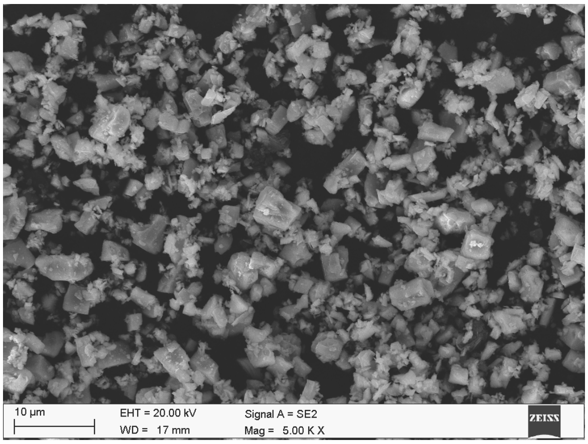

Fig. 2. Powder of TiC carbides

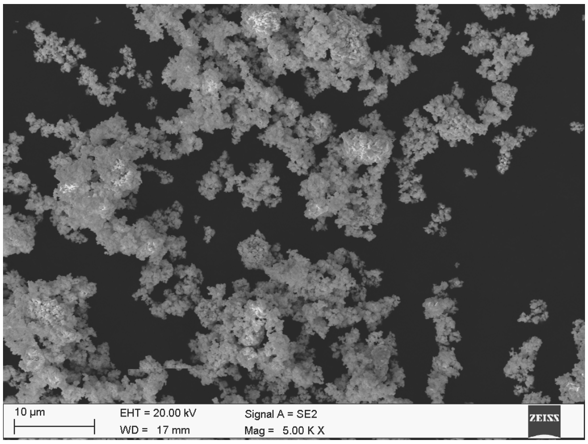

Fig. 3. Powder of WC carbides

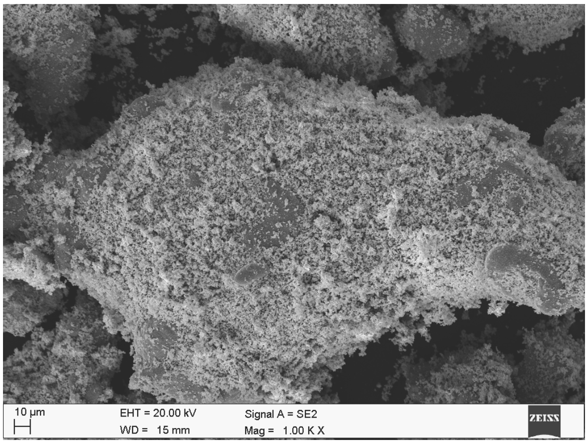

Fig. 4. Mixture of M2 and WC powders

Table 1.
Technological properties of used powders

Powder	Powder properties		
	Density g/cm^3	Bulk density g/cm^3	Tap density g/cm^3
M2	8.17	2.45	3.43
WC	15.51	2.70	4.47
TiC	4.89	1.36	1.68
M2/TiC 68/32 % in vol.	7.07 calculated	2.16	3.25
M2/WC 68/32 in vol.	10.50 calculated	2.55	3.98

Table 2.
Portion of the ceramic powder in the particular layers of the gradient material in the M2 steel matrix

Layer number	Mass concentration of the reinforcing powder, %			
	Material reinforced with WC		Material reinforced with TiC	
	variant 1	variant 2	variant 1	variant 2
5	12	32	12	32
3	9	29	9	29
2	6	26	6	26
1	3	23	3	23
substrate	0	20	0	20

All layers of the gradient material were compacted simultaneously under the pressure of 600 MPa. Next the prepregs were sintered at the temperature from 1210 to 1310°C with 10ºC gradation in N_2-10%H_2 flowing gas atmosphere for 1/2 h. Part of samples was vacuum sintered at the temperature of 1260°C.

Structure observations, X-ray qualitative and quantitative microanalysis of the investigated materials were made with use of ZEISS SUPRA 35 scanning electron microscope at the accelerating voltage of 20 kV.

3. Discussion of results

On the basis of the SEM observations of prepared powder mixes it was found, that employed process of mixing in Turbula mixer during 1h is enough to achieve a homogeneous mixture with uniform cover of high-speed steel powder by grains of carbides (Fig. 4). On the basis of the technological properties investigations of used powders and prepared mixes it was stated, that carbides powders decrease insignificantly flow rate of high-speed steel and its density, shape and grain size have an effect on bulk density and tap density of mixture.

It was found out, based on the structure observations of developed gradient tool materials, that there is no fractures and delamination between poured in matrix and moulded layers. It was also found that, in spite of step character of pouring process, gradient structure was obtained with the close to linear portion change of the used WC and TiC carbides starting from core to the surface layer with maximum addition of reinforcing phases. Structure of gradient material reinforced with TiC carbide with range of reinforcing fraction from 20% to 32%, sintered in N_2-10%H_2 flowing gas atmosphere at temperature of 1260°C for 1/2 h is shown in Figure 5.

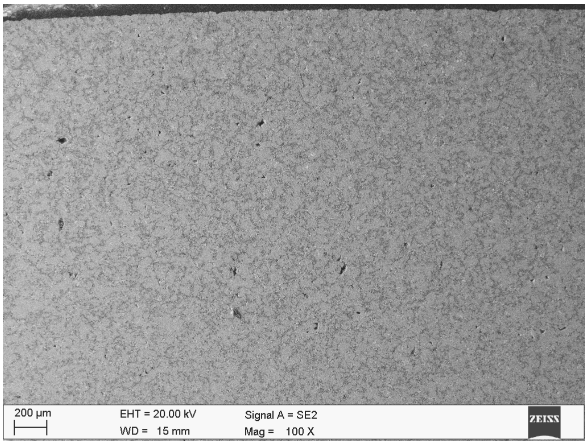

Fig. 5. Microstructure of M2/TiC graded material sintered, under N_2-10%H_2 at 1260ºC

The structures comparison of gradient materials reinforced with TiC carbide vacuum sintered and sintered in N_2-10%H_2 flowing gas atmosphere gave the basis to state, that sintering in nitrogen-hydrogen gas atmosphere gives better results (Figs. 6, 7). Especially material samples vacuum sintered at the temperature of 1260°C was highly porous at TiC carbide high concentration areas, that is in grain boundary of high-speed steel areas. Structure of high-speed steel in this case is characterized by large precipitations of bright carbides rich with vanadium and iron. Simultaneously, in case of above-mentioned process' conditions, locally occurring eutectic mixture was observed, that indicate the unsuitable temperature or too long sintering time (Fig. 6). Decreasing of porosity would have required increasing of the temperature which was already to high.

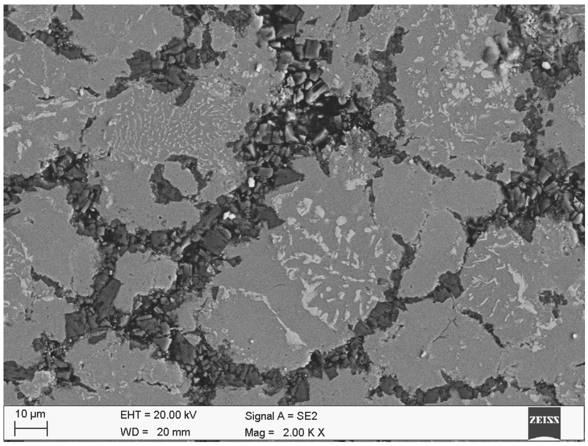

Fig. 6. Microstructure of M2 reinforced by TiC, sintered under vacuum at 1260ºC

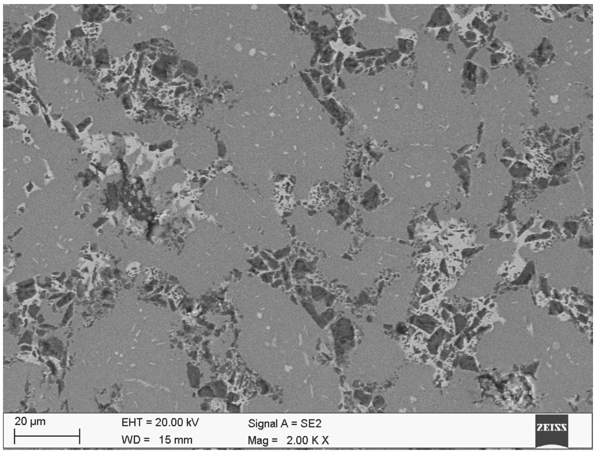

Fig. 7. Microstructure of M2 reinforced by TiC, sintered under N_2-10%H_2 at 1260°C

During investigations of gradient materials reinforced with WC carbide in case of material samples with low volume fraction of WC or sintered in temperature over 1260°C no grains of WC carbide was observed after sintering process. Simultaneously considerable increase of other carbide phases, especially M_6C was observed. It was caused by dissolution of WC carbide and precipitation of new carbide phase rich with tungsten from WC carbide and of iron from matrix.

In case of gradient material reinforced with WC carbide with maximum reinforcing fraction of 32% sintered in temperature under 1260°C, bright, fine-graded WC carbide surrounded with M_6C carbides were clearly apparent. Investigated microsections was characterized by protruding WC phase after long polishing time, what indicates its high hardness (Fig. 8). Amount of dissolved WC increases as an effect of decreasing the sintering temperature. In case of WC carbide reinforced samples sintered in nitrogen-hydrogen gas atmosphere, there was no eutectic mixture observed, however in this case occurrence of MX carbonitride (rich with vanadium) in matrix of carbide alloyed composites was stated.

It was found on the basis of detailed SEM investigations, that an optimal sintering point for carbide alloyed composites (with maximum reinforcing WC volume fraction of 32%) is 1260°C (Fig. 9). Significant increase of carbide phase fraction in this case is connected with entire dissolution of WC carbide and precipitation of new M_6C carbide phase. Such structure is similar to structure of cermet with low fraction of binding matrix. Increase of sintering process temperature to 1270°C causes deformation of surface, rich with WC carbide, as well as rounding of edge, what indicates partial melting process. Even then in areas rich with WC carbide typical eutectic structure was not observed. Sintering process temperature of 1250°C is too low for core area, where fine-graded carbides and much more porous structure than in case of material sintered at 1260°C (Fig. 10) was observed.

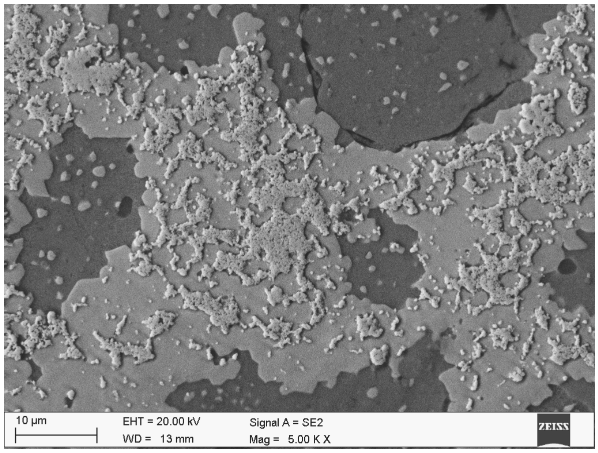

Fig. 8. Microstructure of M2 reinforced by WC, sintered under N_2-10%H_2 at 1210°C

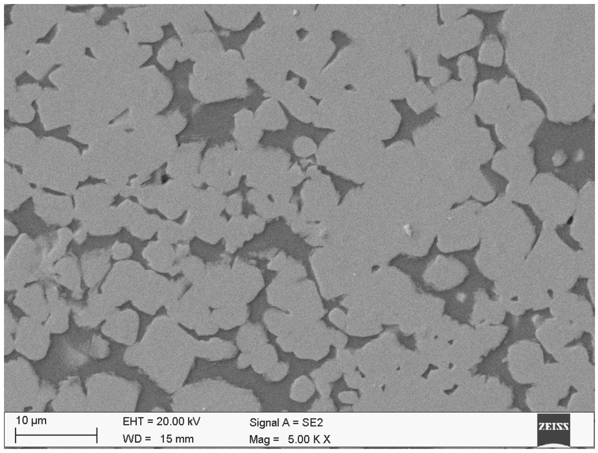

Fig. 9. Microstructure of M2 reinforced by WC, sintered under N_2-10%H_2 at 1260°C

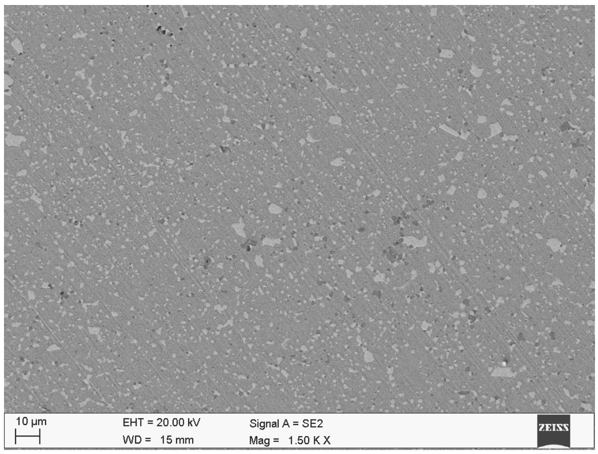

Fig. 10. Microstructure of M2, sintered under N_2-10%H_2 at 1260°C

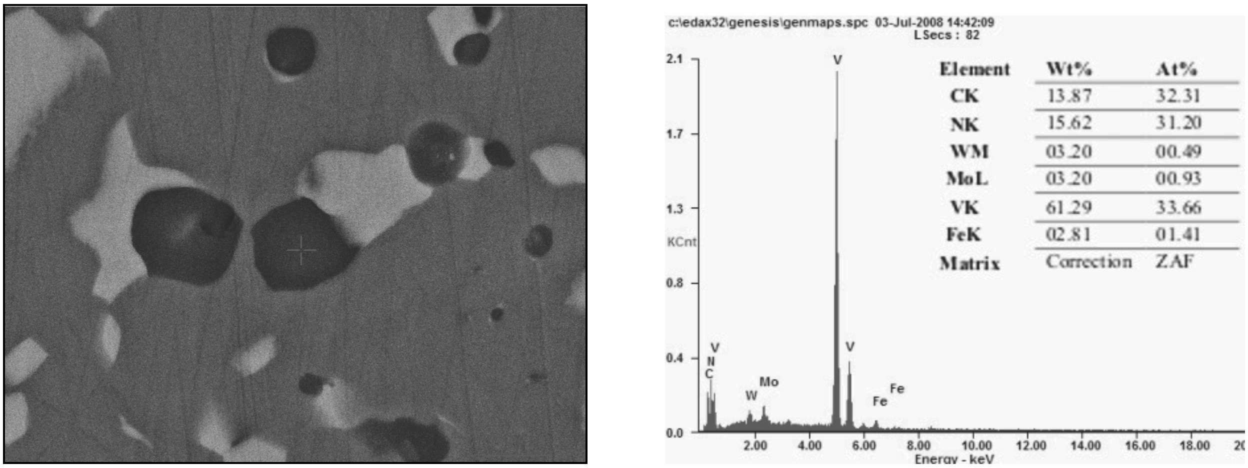

Fig. 11. Microstructure of M2 reinforced by TiC, sintered under N_2-10%H_2 at 1280^{o}C

The structures comparison of gradient materials reinforced with WC carbide vacuum sintered and sintered in N_2-10%H_2 flowing gas atmosphere gave the basis to state, that sintering in nitrogen-hydrogen gas atmosphere makes structure of investigated material more compact, with pores occurred only locally, without eutectic mixture of carbides in area of high-speed steel. Large conglomerates of dark and bright carbides rich with tungsten spaced at grain boundary of high-speed steel was observed. Detailed SEM investigations and X-ray microanalysis gave basis to state, that dark carbides rich with titanium reminds in respect of chemical composition the TiC carbide (alloyed to the composite) while bright carbides rich with tungsten and iron are most probably M_6C carbide, typical for high-speed steels. Moreover analysis carried out gives basis to assume the occurrence of MC carbide, rich with vanadium, carbon and nitrogen precipitations, with chemical composition similar to MX carbonitride (Fig. 11). Such phases was not observed in case of vacuum sintered samples.

It was demonstrated in previous research works (focused on injection moulded and sintered in nitrogen-hydrogen gas atmosphere high-speed steels), that MX carbonitride created during sintering process increases properties of steel, especially phases are stabile in high temperature of sintering and during austenitizing process, do not create the eutectic mixture, what indicates low solubility of MX carbonitrides [13, 14]. High stability of carbonitrides and the influence of nitrogen on decreasing a propensity for adhesive wear, during machining processes, were described also in previous works [15].

Flowing nitrogen-hydrogen gas atmosphere, make possible to increase the temperature of sintering process even up to 1310°C, making it possible to reach high density without the occurrence of eutectic mixture (Fig. 12). Alloying TiC carbide into high-speed steel and sintering in nitrogen atmosphere make it possible to extend the sintering window which originally in case of used high-speed steel equals only 5°C. It is especially important in case of using industrial furnaces much more difficult to control of temperature then in laboratory conditions.

In case of gradient materials reinforced with WC carbide with maximum reinforcing fraction of 12% it was stated, that in top layer rich with WC carbide, reinforcing phase was entire dissolved at temperature of 1210°C in high-speed steel matrix creating M_6C carbide. Locally occurred pores was observed in this area, but core area of pure high-speed steel is much more porous. After structure analysis of gradient materials reinforced with WC carbide with maximum reinforcing fraction of 12% sintered with different temperatures the optimal sintering temperature of 1260°C has been evaluated.

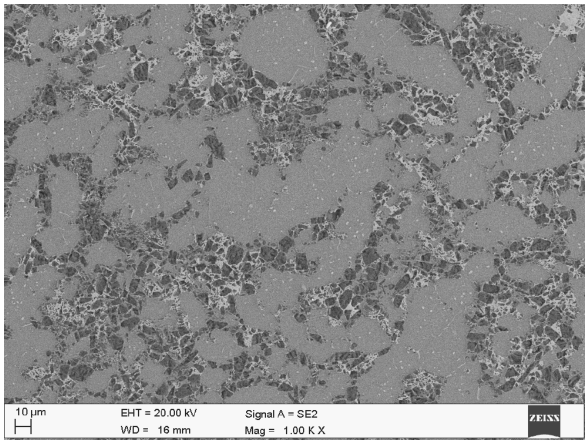

Fig. 12. Microstructure of M2 reinforced by TiC, sintered under N_2-10%H_2 at 1310^{o}C

4. Conclusions

It was demonstrated, that developed composite gradient tool materials, with the core sintered with the matrix obtained using the powder metallurgy of the chemical composition corresponding to the M2 high-speed steel, reinforced with the WC and TiC type hard carbide phases with the growing portions of these phases in the outward direction from the core to the surface there are characterized by no fractures and delamination between layers poured. Gradient structure with the close to linear portion change of the used WC and TiC carbides in spite of step character of pouring process was obtained. Choosing an optimal fraction of reinforced phases is very difficult because of sintering point changing during change of reinforcing phases friction . Partial solution of the problem can be using the N_2-10%H_2 flowing gas atmosphere which make possible to enlarge sintering window up to ~40°C and to increase of sintering point. It makes possible to sintering the matrix high-speed steel in range of 1260-1300°C.

Alloying TiC or WC carbides into high-speed steel appropriately increases or decreases the sintering point in comparison with pure powder of high-speed steel. Sintering point for materials reinforced with TiC carbide is higher then in case of reinforcing with use of WC carbide because of more stabile, regular lattice character of TiC carbide what make it less dissolved in high-speed steel matrix and causing the higher porosity of the material. In materials reinforced with WC carbide, increasing of reinforcing phase friction causes decrease of the porosity. Layers rich with high-speed steel are characterized by higher porosity, because WC carbide dissolved at the grain boundaries of high-speed steel, makes locally the carbon and tungsten concentration higher. Increasing of carbon concentration, decreases the solidus temperature and initiates the sintering process at lower temperature than in case of pure M2 type high-speed steel.

Acknowledgements

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