

Influence of binder composition on structure and properties of carbide alloyed composite manufactured with the PIM method

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ABSTRACT

Purpose: Influence of binder composition, debinding and sintering conditions on structure and some selected properties of the M2 type injected high speed-steel with 10% of carbides are demonstrated in the paper.

Design/methodology/approach: Torque-load test, rheological tests, microstructure examination, density examination.

Findings: Examination of the effect of the binder type and portion on structure and properties of the experimental tool materials with the cermets structure revealed that using the stearic acid not only reduces viscosity, thus improving technological properties of the feedstock, but also extend range of the optimum sintering temperature of the investigated cermets ensuring high density and homogeneous structure. Increasing of sintering temperature increase the density of sintering samples.

Practical implications: It is expected that further investigations of these materials will make possible their injection moulding, as well as their heat treatment increasing hardness and strength of matrix and thereby of the whole tool material. The Powder Injection Moulding gives the possibility to manufacturing tools materials on the basis of high speed-steel which characterised very good properties with their final or near net shape.

Originality/value: In the paper the using injection of the polymer-powder mix gives the possibility to fabricate carbide alloyed composite which, with their structure and mechanical properties, fill the gap in tool materials between the high-speed steels and cemented carbides.

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

1. Introduction

The state-of-the-art manufacturing technologies focused on the sintered tool materials aim to make the best use of the input materials and to fabricate the near-net-shape tools, eliminating in this way the costly plastic working and machining, using the

finish grinding and heat treatment only. Forming the plastic polymer-powder mix with the injection moulding method seems to be especially interesting. This method originated from the injection moulding of polymer materials used widely for fabrication of thermoplastic materials. This method makes it possible to exploit advantages of the polymers injection moulding for fabrication of metal-, ceramic-, and metal-ceramic materials

[1, 2, 13]. Extensive possibilities of fabricating elements with the complex form in their final shape or so called “near-net-shape” by the contemporary powder forming technology mentioned above have found many application areas. Powder injection moulding is also enjoying attention nowadays in tool materials processing, especially the high-speed steels and sintered carbides [1, 3-5, 9, 11, 12]. High-speed steels, in spite of their over a century long tradition of use hold still the strong position in the relatively small group of tool materials because of their desirable ductility thanks to which they are, among tools, the irreplaceable or complementary material in many cases. The high service properties of tools from the high-speed steels are obtained by their additional heat treatment after sintering. The unfavourable band segregation of carbides occurs often in the high-speed steels fabricated by the conventional casting with the subsequent plastic working. This drawback has an effect on the inhomogeneous distribution of mechanical properties that cannot be eliminated by the heat treatment. This phenomenon occurs especially in steels with the high contents of alloying elements originating the primary carbides. This shortcoming does not occur in the sintered materials and distribution of carbides is homogeneous in the entire tool volume. Employment of powder metallurgy methods is needed when portion of carbides in tool materials has to be increased. In this way the widely known cermets and sintered carbides were developed [2, 6-8, 10].

The goal of the project is employing the injection moulding of the mix to be used for fabrication of the contemporary tool materials with the cermet structure. The high-speed steel was used as the matrix material, which due to the secondary hardness effect occurring in it after tempering should be characteristic of higher mechanical properties compared to the constructional heat-treatable steel used in the classic cermets.

2. Material and method

Polypropylene and paraffin in the 50/50 volume portions were used as the main binder components in further investigations. Feedstock samples with the 60, 65, 70 and 75% of M2 steel powder contents were investigated to select its optimum portion. The metal powder used for this study was a prealloyed, gas atomised M2 high speed steel with spherical shape. The 90% of the particles were less than $16\ \mu\text{m}$ (Fig. 1). The density of the M2 HSS was $8.16\ \text{g/cm}^3$, as measured with a pycnometer Micrometrics AccuPyc 1330. Torque-load and rheological tests were carried out to this end. The same tests were carried out for feedstock based on carbides to determine their maximum portion. In case of carbides the effect of the stearic acid (SA) on feedstock viscosity was also tested.

Knowing the optimum feedstock components percentages with the M2 powder and of the feedstock with carbides their portions were selected so that the final mix contained 58% M2 and 10% carbides. Next, two types of the A feedstock without SA and B with the stearic acid covering the carbides were injected and sintered. The carbides powder mix is composed of WC, TiC, TaC, and NbC (33.3/33.3/26.6/6.8% in vol.) are shown in Figure 2. Percentages of these powders and binder are shown in Table 1. To cover the carbides surface with the stearic acid they were poured into the acid dissolved in methanol, and were churned for 30 min, so that the SA could cover uniformly carbides coatings. Next the mix was heated

to temperature of 60°C to evaporate the methanol. Mixes of carbide powders with SA coating prepared in this way were initially mixed with the high-speed steel powder and paraffin in a Turbula Mixer at room temperature during 1 hour.

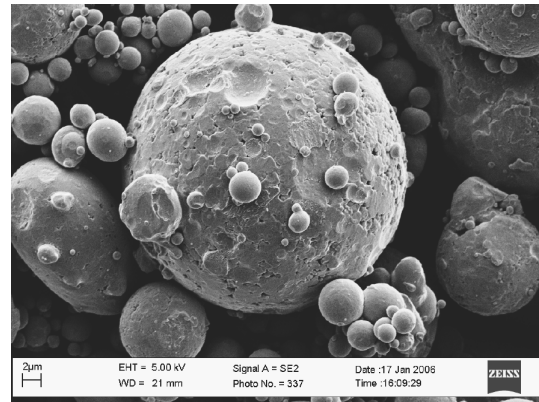


Fig. 1. The powder of high speed-steel M2 type

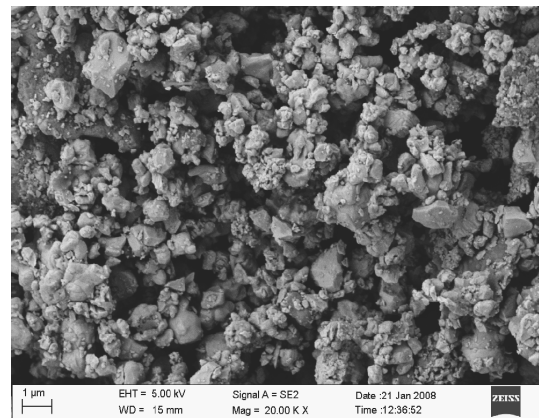


Fig. 2. The powder of WC, TiC, TaC, and NbC mixed carbides

The maximum metal and carbides powder percentage was determined by torque measurements. Next the feedstock formulation was granulated and extruded twice to guarantee a good homogeneity in a twin screw extruder Rheomex CTW100p. Feedstock of A and B types with the optimum binder contents were next injected in an Arburg injection machine 220-S using the mould for 3-point bending test.

Table 1.
Compositions of injected feedstock

Type of Feedstock	Portion of components in volume, %				
	M2	Carbides	PP	PW	SA
A	58	10	14	14	4
B	58	10	16	16	0

The binder has been partially driven off by solvent and thermal debinding. The moulded samples were debound in heptane at 60°C for 2 h and next in a cylindrical chamber furnace Goceram AB GCDV-50 under N₂-10%H₂ flowing gas at 440°C. These atmospheres prevent from the oxidation of metallic powders. Test pieces were sintered in the atmosphere of the flowing N₂-10%H₂ mixture of gases at the temperature between 1300 and 1380°C in steps of 20°C for 1h. Densities of the sintered parts were measured using Archimedes' method. Structure examination and the qualitative X-ray microanalysis were carried out on the SUPRA 25 scanning electron microscope (SEM) equipped with X-ray energy dispersive spectrometer (EDS).

3. Results and discussion

Investigation of the technological properties of mixes containing only powder of the M2 steel or carbides apart from the binder made it possible to select the optimum composition of the feedstock containing both powders, which, after forming, debinding and sintering should have a structure of a cermet. Both the feedstock A without SA and feedstock B with the stearic acid coating the surface of carbides demonstrated good technological properties during the extruding operation. The results of torque-load and rheological tests were in detail demonstrated in preceding article [3].

Solvent degradation was used to remove paraffin quickly from the injected prepregs. Therefore, the next operation of thermal degradation proceeds faster due to open pores and increase of the reaction surface with the flowing gas mixture. The effect of time on solvent degradation is presented in Fig. 3.

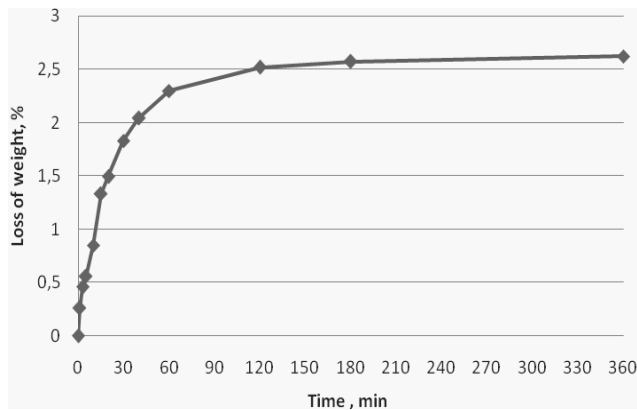


Fig. 3. Influence of solvent degradation time on loss on weight of debinding samples

Based on measurement of the test piece mass loss versus immersion time in heptanol one can state the most of the paraffin gets dissolved after about 30 min already, which was assumed to be the optimum solvent degradation time. Solvent degradation made applying 3.5°C/min test piece heating rate possible during thermal degradation and applying the short holding time of 30 min. Test pieces inspected with the unaided eye after degradation did not reveal cracks or gas

cavities on their surface. Increase of carbon portion in materials after degradation in respect to the powder used was only 0.3% mass.

It was found out based on examinations of cermet structure in the sintered state that regardless from the sintering temperature one can observe the primary grains of the high-speed steel powder surrounded by numerous light carbide precipitations. These carbides grow along with the rising sintering temperature but do not create the eutectic structure typical for the high-speed steel sintered at the excessively high temperature. No occurrences of eutectics were revealed also based on examinations of cermet sintered at the highest temperature, i.e., 1380°C, albeit the test piece deformation may indicate partial melting. Based on that one can assume that the introduced carbides with the regular structure stable at high temperatures do not cause origination of eutectics but only grow and coagulate at high sintering temperatures. This offers the possibility to use the heating units with the higher temperature tolerance during holding. Employment of the (N₂-10%H₂) nitrogen rich atmosphere during sintering causes development of the fine spherical carbonitrides of the MX type stable at high sintering temperatures. However, this effect is more explicit in the high-speed steels without the additionally introduced carbides.

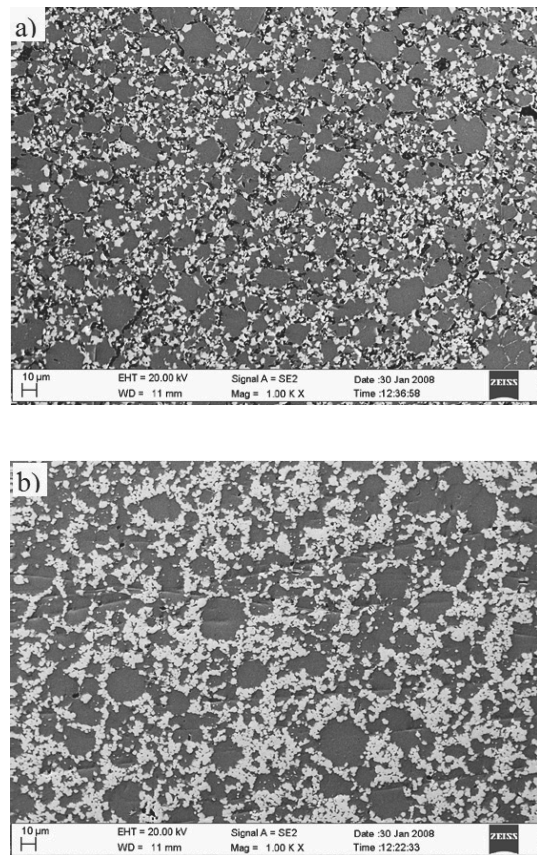


Fig. 4. Structure of carbide alloyed composite manufactured by PIM and sintered at 1320°C; a) without stearic acid, b) with stearic acid

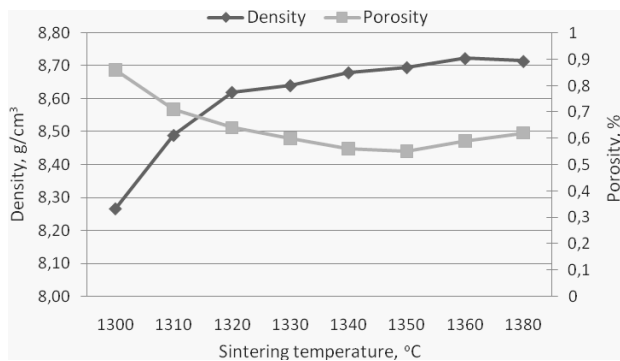


Fig. 5. Influence of sintering temperature on density and porosity evaluation

Structure examination of cermet sintered at temperature of 1320°C with the addition of the stearic acid (SA) and without the acid revealed that cermets in which carbides are coated with SA get compacted faster, have lower porosity, and can be sintered at lower temperatures (Fig. 4). Based on that it was decided to carry out the further investigations only for the A type material. Based on density test results it was found out that the maximum density is 8.72 g/cm³ and corresponds to the test pieces sintered at temperature of 1360°C, and the minimum density of 8.26g/cm³ is revealed by prepreps sintered at temperature of 1300°C. It was found out based on porosity examinations of test pieces from the HS6-5-2 steel reinforced with carbides that the portion of pores is relatively low, typical for the sintered tool materials, i.e., below 1%. Regrettably, big spherical pores occurring locally, having developed most probably because of the non-uniform distribution of powder in the binder matrix.

The pores are not dependant on sintering temperature and occur in all sintering conditions. Generally, porosity gets lower with the sintering temperature increase. The density and porosity plot versus sintering temperature is presented in Fig. 5.

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