Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 42
ISSUES 1-2
September-October
2010

Carbide alloyed composite manufactured with the Powder Injection Moulding method and sinterhardened

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Received 01.06.2010; published in revised form 01.09.2010

Manufacturing and processing

ABSTRACT

Purpose: Development of a new generation tool materials on the basis of M2 high speed-steel reinforced with the mixture of carbides and with their structure and mechanical properties, fill the gap in tool materials between the high-speed steels and cemented carbides.
Design/methodology/approach: Powder metallurgy, powder injection moulding, sintering, sinter hardening, heat treatment, microstructure and porosity examination, X-ray analysis, TEM, bending test, hardness test.
Findings: Powder injection moulding processes were used to fabricate the proposed carbide alloyed composite materials. The addition of hard particles increase hardness after heat treatment and slightly reduces the ductility of these materials. Compared with M2 high-speed steel the bending strength of carbide alloyed composite decrease. The main advantage of the presented experimental tool materials is application of powder injection moulding to produce tool materials in a mass scale with relative low cost of production. Moreover the cost of production reduce application of sinterhardening.
Practical implications: Application of heat treatment and especially sinterhardening to improve the mechanical properties of presented experimental tool materials gives the possibility to obtain tool materials with the relative high ductility and high hardness typical for cemented carbides.
Originality/value: The essential advantage of the investigated injection moulded material and sintered is the broad range of the optimum sintering temperatures and the relatively small effect of the sintering temperature growth on the carbides growth makes using the industrial heating equipment possible.
Keywords: Powder metallurgy; Powder Injection Moulding; Feedstock; Tool materials

Reference to this paper should be given in the following way:
G. Matula, Carbide alloyed composite manufactured with the Powder Injection Moulding method and sinterhardened, Journal of Achievements in Materials and Manufacturing Engineering 42/1-2 (2010) 164-171.

1. Introduction

Powder Injection Molding (PIM), which encompasses Metal Injection Molding (MIM) and Ceramic Injection Molding (CIM) is a net-shape process for the manufacturing of small, complex and high precision components for use in a diverse range of industries. Products obtained by PIM processes can be found in many areas of life, from automotive vehicle to watches, dental braces or mobile phones. Metal injection moulding application are growing at a rapid rate with an increase of over 80% in the tonnage of metal injection moulded parts shipped in the period 2003 to 2006. The comparison of powder injection moulding with other methods of manufacturing of metal or ceramic parts from powder is shown in Figure 1. The process of powder injection moulding consists of four key steps: compounding of powder and polymer components to produce the "feedstock", moulding with conventional injection moulding equipment to produce the green parts, debinding to remove the binder in order to obtain binder-free brown parts and sintering [1-8].

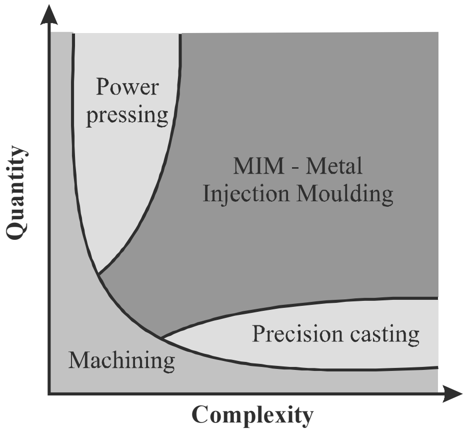

Fig. 1 Powder injection moulding in comparison with other manufacturing method [1]

Powder injection moulding is a development of the traditional powder metallurgy (PM) process and is rightly regarded as a branch of that technology. The standard PM process consists on compaction of a lubricated powder mix in a rigid die by uniaxial pressure, ejection of the compact from the die, and sintering of it. Quite complicated shapes are regularly produced in huge quantity. There is also significant limitation of the shape of compacts. After compaction in the die, the part must be ejected, i.e. pushed out of the die cavity. It will be obvious, therefore, that parts with undercuts or projections at right angles to the pressing direction cannot be made directly. That limitation is substantially removed by the metal injection moulding process. Moreover PIM process have many other advantages. Residual carbon originated from binder causes decreasing sintering temperature and initiates the sintering process, thus giving the possibility to obtain parts with very high density [9-11]. Manufacture of tools like HSS, cemented carbides or cermets can be achieved by PIM and is already in use in the market as valuable process for obtaining low porosity tool materials where porosity is not higher than 2% [12, 13].

The goal of the work is development of a new generation of the composite tool materials with high-speed steel as the matrix material, reinforced with mixture of hard carbides inducing increase of wear resistance saving relatively high ductility.

2. Materials and research methodology

The feedstock preparation and powder injection moulding conditions of investigated specimens are presented in previous papers [13-15]. The binder has been partially driven off by solvent and thermal debinding. Test pieces were sintered in the atmosphere of the flowing N_2-10%H_2 mixture of gases at the temperature between 1200 and 1300°C in steps of 10°C for 0.5h. Heat treatment of the newly developed materials consisted in cooling from their sintering temperature [16,17]. Such treatment, defined in the literature as sinterhardening, was carried out to skip the succeeding austenitizing operation which is necessary for material hardening. To determine the sinterhardening treatment effect on microstructure and cermet properties, the classic heat treatment was carried out also, i.e., hardening from the austenitizing temperature with the succeeding tempering. Taking care to maintain the comparable experiment conditions, the austenitizing temperature was set equal to the sintering temperature and was 1260°C, whereas its time was 30 min in both cases. The sinterhardened test pieces and those heat treated in a classic way were next tempered at temperatures of 510, 540, 570, 600 and 630°C for 1 hour. Density and pore portion examinations were made of the sinterhardened samples to determine the effect of cooling rate after sintering on the sintered material density and porosity, and their results were compared with those for the samples cooled at the 10°/min rate, i.e., typical cooling rate after sintering. Density examination in the as sintered or hardened states from the sintering temperature was made with the densimetric method. Lacquered coating was not used due to the low porosity of the investigated materials. The measurements were made on the WAS 220/C/2 analytical and precision balance. The pore portion assessment was made on the non-etched microsections with the image analysis method on the OPTON Axiovert 405 M light microscope equipped with the Leica QWin computer image analysis system. The average pore portion was calculated based on ten measurements for each investigated sample. Morphology observations of the powder grains and structure of the fabricated materials were carried out on the Zeiss Supra 25 electron scanning microscope at the accelerating voltage of 20 kV using detection of the scattered and secondary electrons. The X-ray qualitative phase analysis of the investigated materials was made using the X'Pert PANalytical diffractometer in the Bragg-Brentano setup (Kα, Co, step 0.05, impulse counting time 10 sec, angle range 2° from 35 to 110°). Hardness tests of the investigated materials were carried out on the ZWICK ZHR hardness tester with Rockwell method in scale C.

3. Results and discussion

The results of feedstock preparation process, conditions of injection, solvent and thermal degradation, as well as selection of the optimum binder degradation cycle are presented in papers [13-15]. Further activities included investigation of microstructure and mechanical properties of the developed materials. It was found out based on the investigation of the sintering temperature effect on size of carbides and their surface portion that the growth of carbides is insignificant in comparison with carbides in the high speed steel, and their portion and size are presented in Figure 2. The maximum size of carbides is about 40 μm, whereas its average value is about 2.8 μm.

The size of carbides occurring in cermet sintered at the temperature of 1300°C is comparable to size of carbides occurring in cermets sintered at a lower temperature.

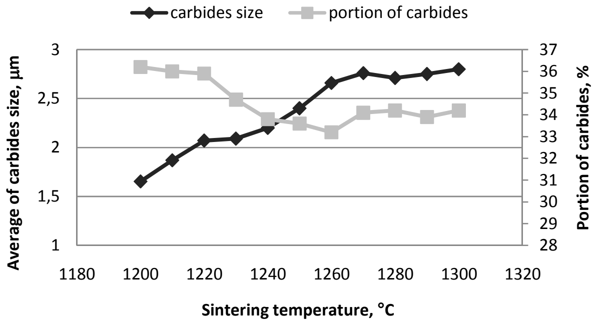

Fig. 2. Influence of sintering temperature on Carbides size and surface portion of carbides

The surface portion of carbides occurring in cermet sintered at the temperature of 1200°C is largest and is about 36%. The portion of carbides drops to the value of about 33% along with the temperature rise, after sintering at the temperature of 1260°C, and grows next to the value of 34.2% after sintering at the temperature of 1300°C. One may also note that the average carbide size grows from the value of 1.7 to about 2.8 µm along with the temperature growth from 1200 to 1300°C.

It was found out based on the microstructure examinations on the scanning microscope equipped with EDS that growth of the bright carbides appearing in the secondary electrons image, rich in W, Mo, and Fe is limited by the surrounding grey phases rich in Ti and V, which is presented in Figure 3.

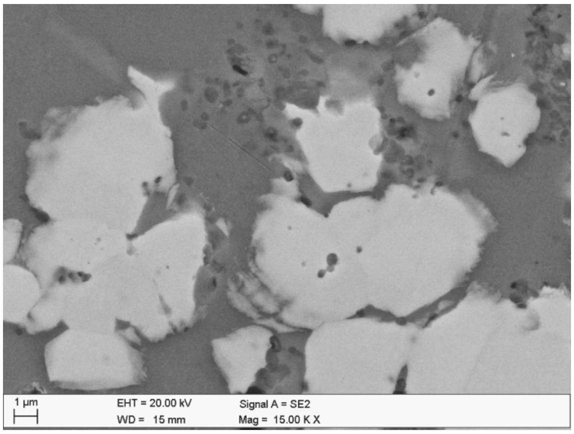

Fig. 3. Microstructure of investigated materials sintered at 1280°C

Types of these carbides were identified with the diffraction methods and presented in Figures 4 a, b and c. These are the typical carbides of the MC and M_6C types occurring in the matrix used, i.e., M2 high speed steel. Moreover, it was found that carbon atoms in phases with the MC regular lattice are partially replaced with nitrogen during sintering in the nitrogen rich atmosphere, creating in this way carbonitrides rich in V. Results of examination in TEM revealed that the carbides MC_3 and M_7C_3 also occurring in the carbide alloyed composite (Figs. 5 to 7).

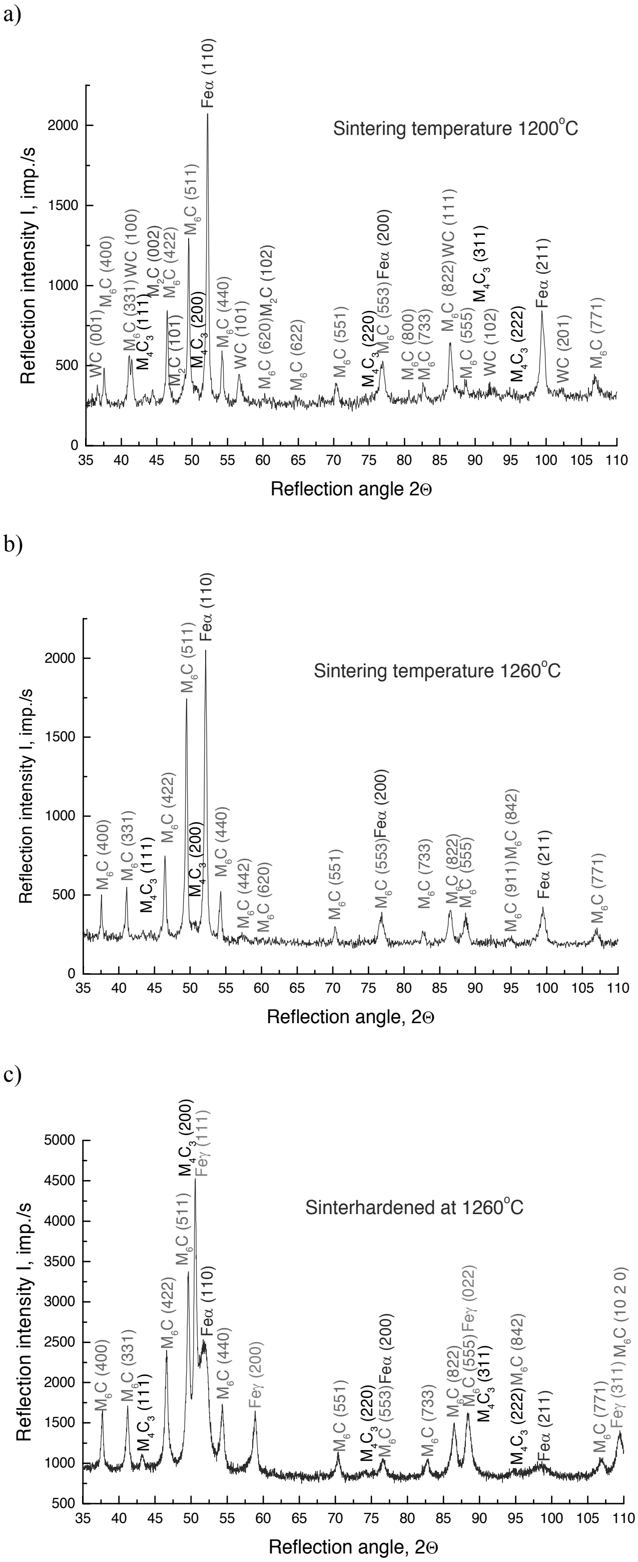

Fig. 4. X-Ray diffraction pattern for sample a) sintered at 1200°C, b) sintered at 1260°C and c) sinterhardened at 1260°C

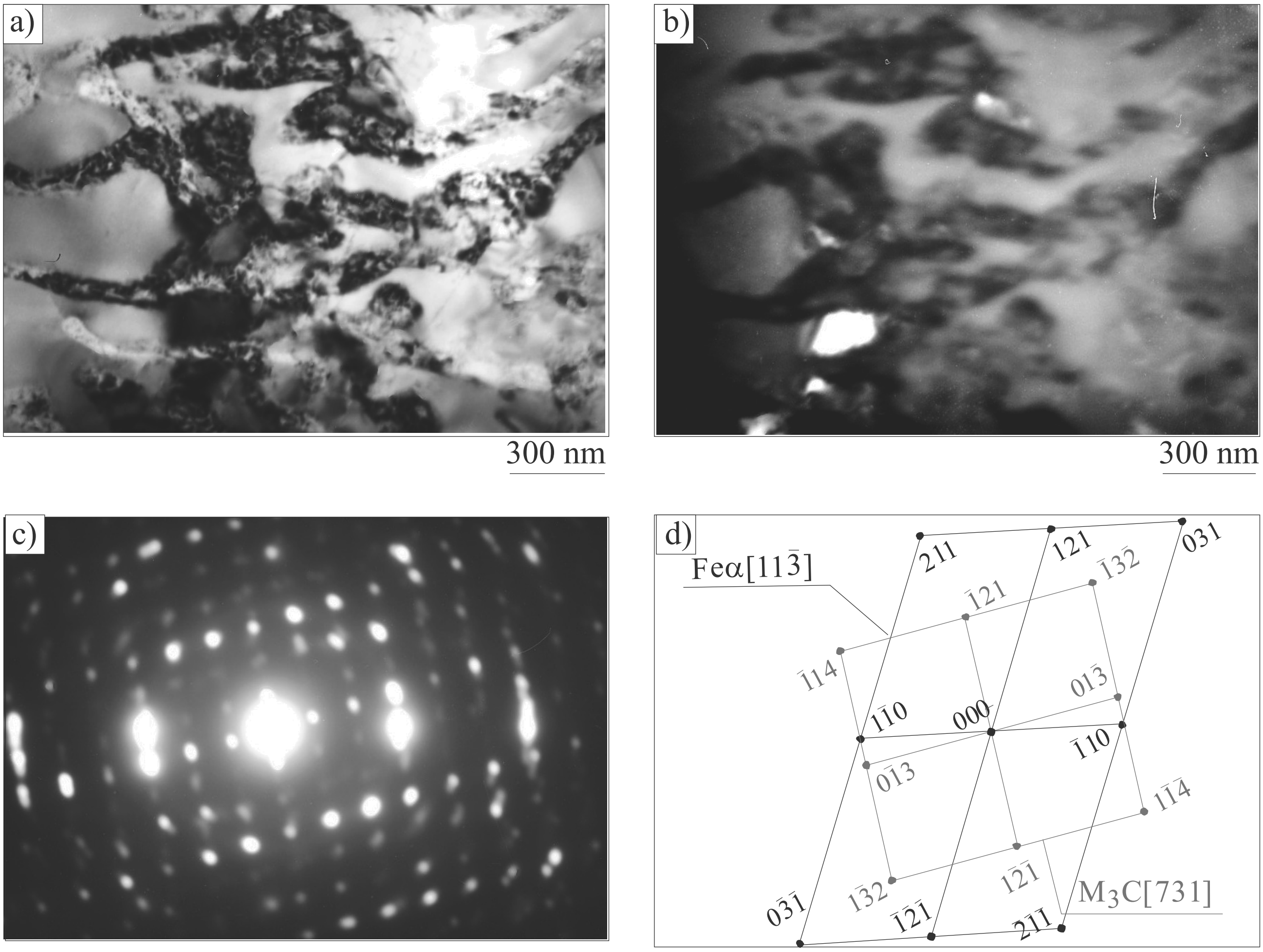

Fig. 5. a) microstructure of carbide alloyed composite manufactured by PIM method austenitized at 1260°C temperature and quenched, b) dark field from reflex (114) M_3C, c) diffraction pattern of area shown in a), d) part of solution for diffraction pattern shown in c)

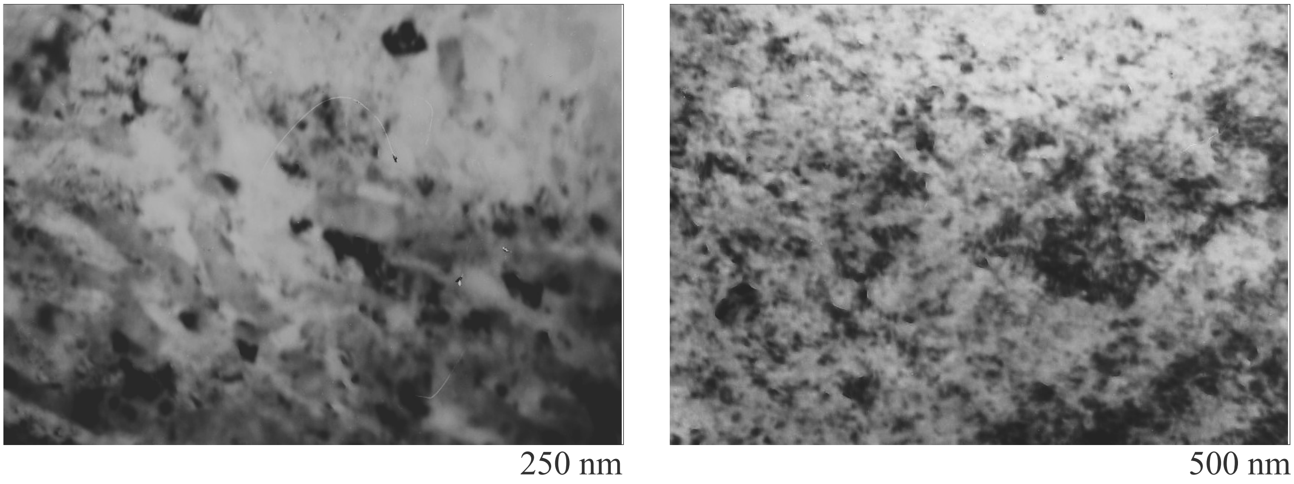

Fig. 6. a), b) microstructure of carbide alloyed composite manufactured by PIM method austenitized at 1260°C temperature, quenched and tempered at 600°C (thin foil)

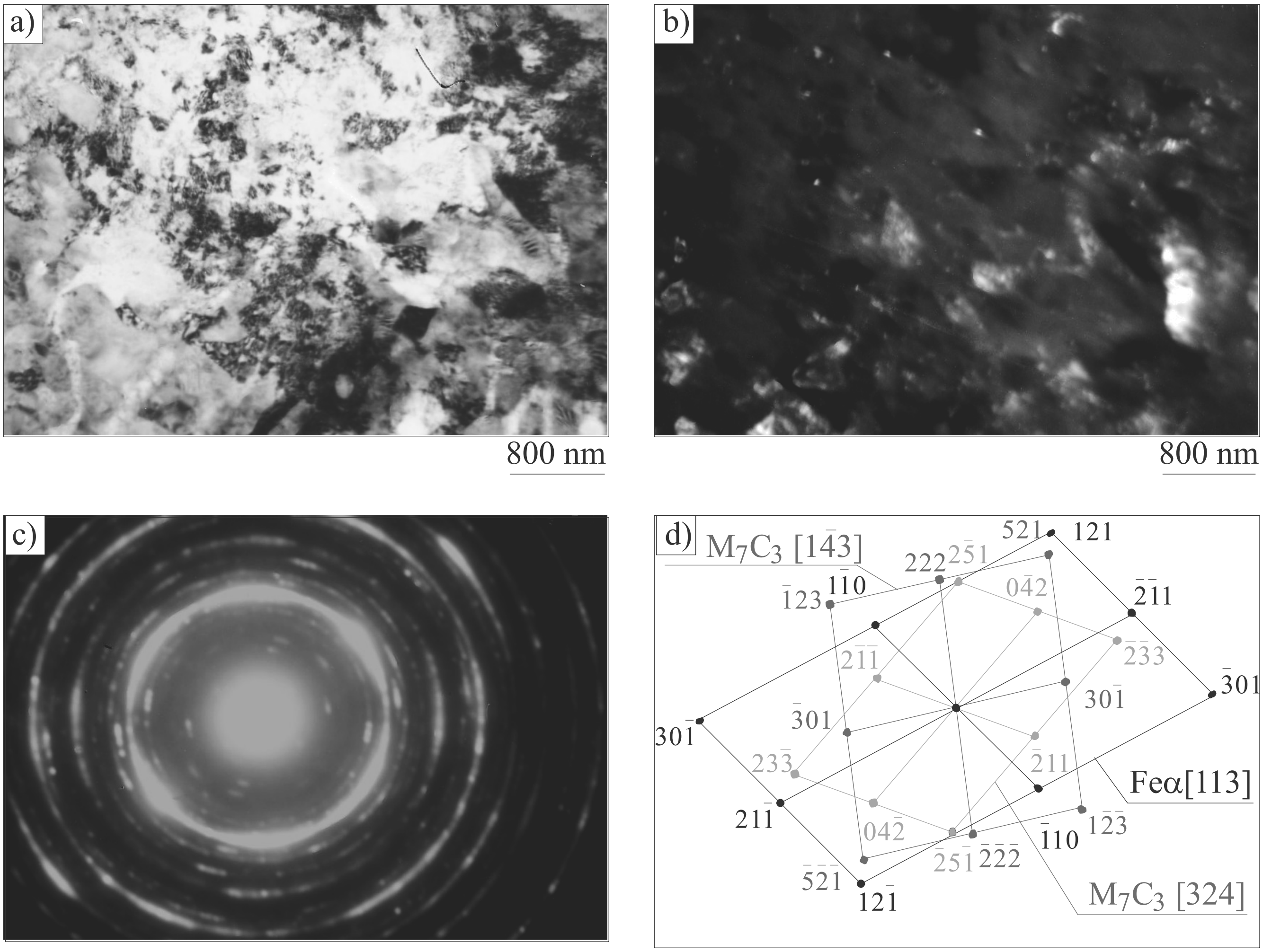

Fig. 7. a) microstructure of carbide alloyed composite manufactured by PIM method austenitized at 1210°C temperature, quenched and tempered at 600°C (thin foil), b) dark field, c) diffraction pattern of area shown in a), d) part of solution for diffraction pattern shown in c)

The mechanism of the carbonitrides and nitrides generation during sintering in the N_2-H_2 protective atmosphere was investigated and described before by Palma et al [18] and Giménez et al [19]. The investigation results of the newly developed cermet showed that this material has the best mechanical properties and the homogeneous and fine microstructure after sintering at the temperature of 1260°C. Density tests and examinations of pores portion in samples hardened from the sintering temperature, i.e., subjected the sinterhardening process, have revealed that the cooling rate does not affect change of the density, and ipso facto neither its porosity. Results of these tests are presented in Figures 8 and 9. Results of examination with the X-ray phase analysis revealed that the diffraction patterns differ from each other.

Diffraction pattern of the sample cooled slowly after sintering does not display any occurrence of austenite. One may observe clear peaks from the retained austenite in the diffraction pattern of the sample hardened from the sintering temperature. Examinations carried out with the X-ray quantitative analysis revealed that the retained austenite portion is about 60%, which significantly exceeds the amount of the retained austenite in the classic high speed steels after hardening. The high portion of the retained austenite is caused by the content increase of carbon remaining after the binder temperature degradation and resulting from dissolving the carbides introduced as additives. Moreover, carbon is released also during generation of carbonitrides according to the MC+N→M(C,N)+C mechanism [11, 12]. Increase of carbon content lowers the martensitic transformation temperature, therefore the retained austenite portion after hardening is so high in these materials. Triple tempering of cermet at the temperature optimal for the high-speed steels. i.e., 540°C, results in reducing the austenite portion to 22% only. Only tempering of cermet at the temperature of 540°C causes reduction of the austenite portion to about 4%. This value, however, is encumbered with the big error resulting from the examination method. Further increase of the tempering temperature to 630°C causes complete disappearance of the Feγ peak from the (200) plane which does not coincide with any other peak. Based on that, one may expect that the retained austenite does not occur in this material or its portion is close to 0%.

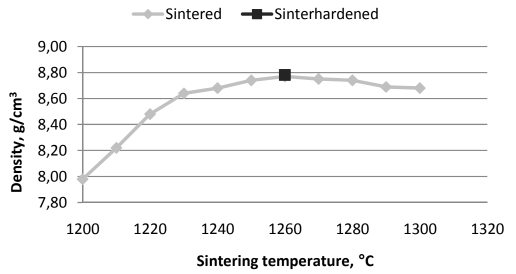

Fig. 8. Influence of sintering temperature on density sintered or sinterhardened carbide alloyed composite

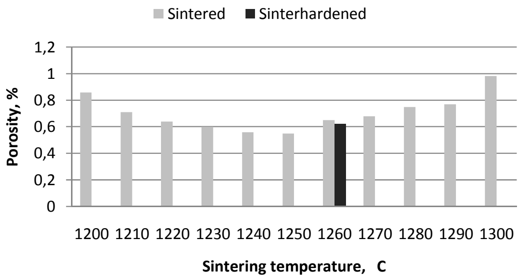

Fig. 9. Influence of sintering temperature on porosity sintered or sinterhardened carbide alloyed composite

The cermet hardness grows along with the lowering portion of the retained austenite, reaching its maximum value after tempering at the temperature of 600°C. This is connected with the transformation of the austenite into martensite during cooling from the tempering temperature and with precipitation of carbides during holding at the tempering temperature.

Hardness tests results of samples hardened from the sintering- or austenitizing temperatures, as well as of samples after tempering are presented in Figure 10.

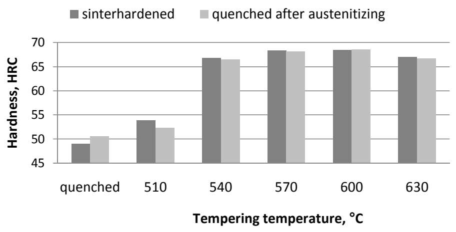

Fig. 10. Influence of heat treatment on hardness of investigated carbide alloyed composite

Structure and properties of the investigated carbide alloyed composite undergo significant changes during tempering. It was found out, basing on the analysis of retained austenite (Fig. 13), hardness tests and bending test (Figs. 11 and 12) that in the presented materials the secondary hardness effect have place. Moreover the effect is higher compared to hardness after quenching and hardness after tempering of the commercial high-speed steels.

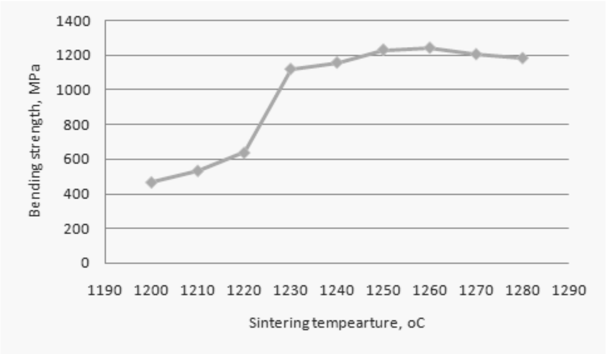

Fig. 11. Influence of sintering temperature on bending strength of carbide alloyed composite

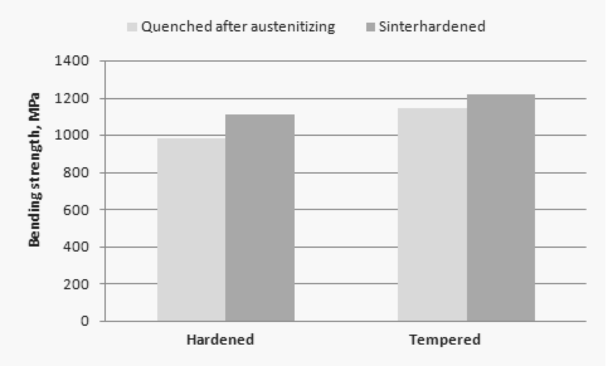

Fig. 12. Influence of heat treatment on bending strength of carbide alloyed composite

4. Conclusions

One can state, based on the presented results, that the chemical composition of the newly developed materials - cermet - was selected properly and the finally obtained material is characteristic in the sintered state of the homogeneous structure with the fine precipitations of carbides in the alloy ferrite matrix. The essential advantage of the investigated injection moulded material is its mass production possibility, which is just offered by the injection moulding of the powder. Moreover, the broad range of the optimum sintering temperatures and the relatively small effect of the sintering temperature growth on the carbides growth makes using the industrial heating equipment possible, especially the flow ones, for which temperature regulation accuracy and keeping the stable temperature during sintering is difficult.

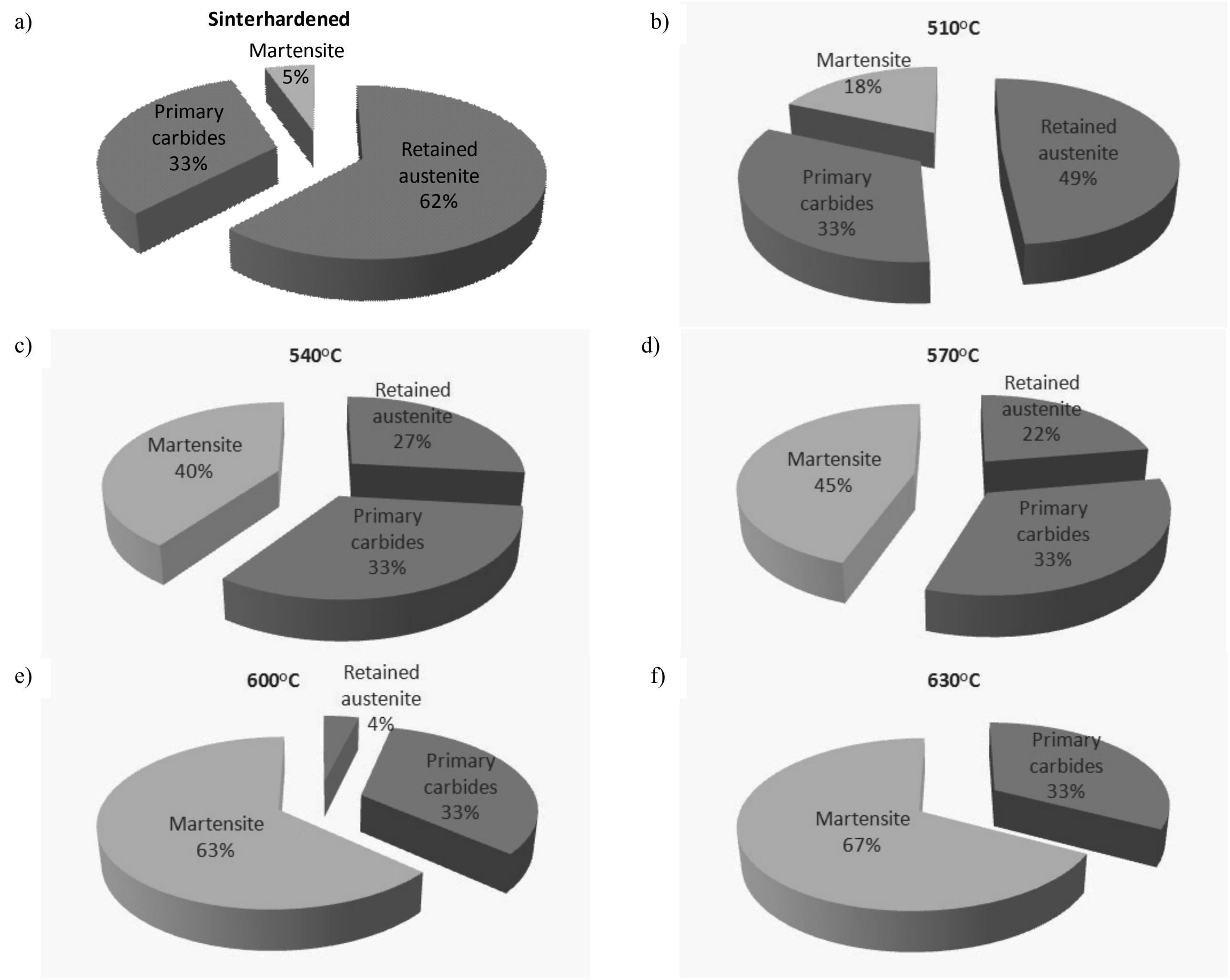

Fig. 13. Influence of tempering temperature on portion of retained austenite

Introducing the tetra carbides into the high speed-steel makes using higher sintering temperatures possible, therefore, a lower sinter porosity can be attained. Moreover, carbides with the regular lattice do not get dissolved in the matrix and do not create the eutectic structure at the high sintering temperature. In addition, sintering in the atmosphere of the protective gases N_2-10%H_2 causes diffusion of nitrogen into the sintered material and development of phases rich in this element.

Porosity examination revealed that the average portion of pores does not exceed 1% in the investigated materials; however, one may observe locally occurring big pores which may decidedly impair the mechanical properties of the investigated materials.

Additional information

Selected issues related to this paper are planned to be presented at the 16th International Scientific Conference on Contemporary Achievements in Mechanics, Manufacturing and Materials Science CAM3S'2010 celebrating 65 years of the tradition of Materials Engineering in Silesia, Poland and the 13th International Symposium Materials IMSP'2010, Denizli, Turkey.

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