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Analysis of improvements in technological properties of WC-Co tool materials fabricated by spark plasma sintering

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ABSTRACT

Purpose: The technological properties of WC-Co tool materials fabricated by spark plasma sintering has been investigated.

Design/methodology/approach: The technological properties has been investigated using instrumented indentation and a Vickers tip indenter with an applied load of 300 mN. SEM and LM methods were used to characterize the microstructure.

Findings: The best results of nanohardness (2144 $HV_{0.031}$), elastic modulus (673 GPa), susceptibility to brittle cracking (0.60 μm/N) and work cracking (0.09 μJ) were obtained by compacts spark plasma sintered for a holding time of 3 min and at a heating rate of 300°C/min from a WC-6Co premixed powder with a particle size of 100-200 nm.

Practical implications: The nanohardness, elastic modulus, susceptibility to brittle cracking and work cracking have been studied.

Originality/value: Investigations of the role of holding time and heating rate in the spark plasma sintering process of WC-6Co tool materials revealed that nanohardness, elastic modulus and susceptibility to brittle cracking increase with a decreasing holding time and heating rate in contrast to work cracking which decreased.

Keywords: Spark plasma sintering; Cemented carbide; Tool material; Technological properties

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PROPERTIES

1. Introduction

WC-Co tool materials due to their favourable combination of soft and plastic Co, which combines with very hard and brittle WC, are the most promising materials for machining, mining, cutting and drilling tools because they offer a very good balance of hardness and fracture toughness [1-4]. Increasing the content of the WC fraction can increase hardness and wear resistance, while fracture toughness can be increased by increasing the Co content

[4-5]. That is why WC-Co cermets with 5-10 wt% Co are the most promising materials for industrial applications [5]. WC-Co tool materials, as well as other sintered carbides, are produced by powder metallurgy methods, and in addition to conventional sintering, in recent years, spark plasma sintering has been used for the production of WC-Co cermets [6-8]. This sintering technique is becoming increasingly more attractive for the fabrication of WC-Co tool materials in view of the rapid heating and cooling, short holding time and controllable compaction pressure over conventional powder metallurgy methods. That is why Garbiec and Siwak in a previous study [8], investigated the effect of the heating rate and holding time on the microstructure and mechanical properties of nanocrystalline WC-6Co cermets and achieved tool materials characterized by a hardness of 1842 HV_{30} and fracture toughness of 12.39 $MPa \cdot m^{1/2}$. Moreover, the obtained results clearly show the remarkable effect of the heating rate and holding time on the spark plasma sintering process and the results from this microstructure evolution and changes in the main mechanical properties. Therefore, in this paper we report the results of WC-6Co tool materials spark plasma sintered for holding times of 3 and 6 min and at heating rates of 300 and 600°C/min.

2. Materials and methods

Two types of WC-6Co premixed powders with a particle size of 100-200 nm in the first case and 30-50 µm for WC and 4 µm for Co in the second case were used. The powders were supplied by Kamb Import-Export, Poland. The powders were densified by spark plasma sintering using an HP D 25-3 furnace (FCT, Germany). The sintering temperature of 1450°C was reached at the heating rates of 300 and 600°C/min. The compaction pressure level on the specimens was kept constant at 50 MPa throughout the sintering process. The vacuum level of the sintering chamber was set at 5Pa. After a 3 or 6 min holding time, the sintered compacts were rapidly cooled to room temperature. From the spark plasma sintered compacts with dimensions of Ø20x3 mm, samples for testing were cut by wire electrical discharge machining. The studies on the nanohardness (H_{IT}), elastic modulus (E_{IT}), susceptibility to brittle cracking and work cracking (W_t) were carried out in accordance with the EN ISO 14577-1:2002 standard by means of a Picodentor HM500 (Fisher, Germany). A Vickers indenter was used and a load of 300 mN was applied for 5s. The microstructure of the spark plasma sintered materials was observed on polished cross-sectioned surfaces by scanning electron microscopy using an Inspect S (FEI, Netherlands) microscope.

3. Results and discussion

Figure 1 shows the SEM micrographs in backscattered electron contrast showing the microstructure of WC-6Co cermets prepared from a powder mixture having a particle size in the range of 100-200 nm. It revealed two phases: WC (bright field) and Co (dark field). The black areas represent pores. On the basis of analyses of the obtained microstructures, it was demonstrated that increasing the heating rate from 300 to 600°C/min for a holding time of 3 min increases the WC grain size, resulting in a less homogeneous microstructure. The phenomenon of grain growth was observed also in the case of the cermets sintered for 6 min, irrespective of the heating rate, whereby the microstructures of these composites are more homogeneous and the grain growth is a direct result of the longer holding time.

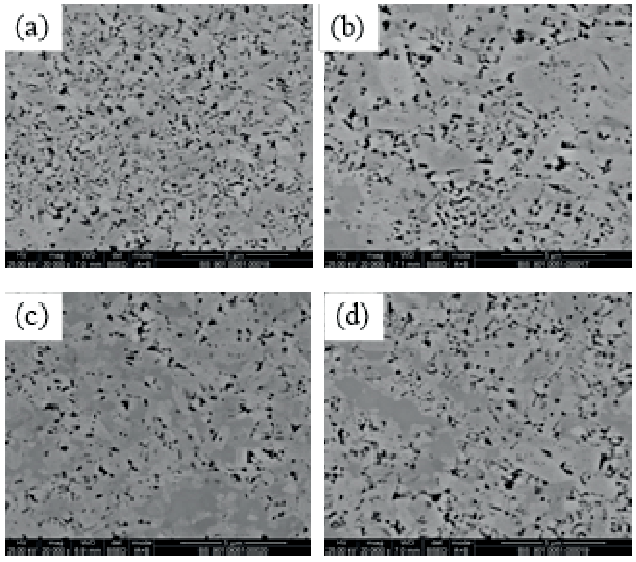

Fig. 1. Backscattered SEM micrographs of spark plasma sintered WC-6Co tool materials for (a) 3 min and at 300°C/min, (b) 3 min and at 600°C/min, (c) 6 min and at 300°C/min, (d) 6 min and at 600°C/min

In turn, Figure 2 show LM micrographs on which the microstructure of WC-6Co composites prepared from a mixture of WC powder having a particle size in the range of 30-50 µm and Co having a mean particle size of 4 µm are visible. Similarly, the dark phase is WC while the

bright phase is Co and the black areas represent pores. It has been shown that increasing the holding time from 3 to 6 min increases the density of the sintered materials, resulting in a smaller pore volume, their transformation to spherical shape and dissolution. Figure 3 shows the nano-indentation curves for spark plasma sintered WC-6Co tool materials. The nanoindentation test with a load of 300 mN indicates that the obtained cemented carbides are very hard.

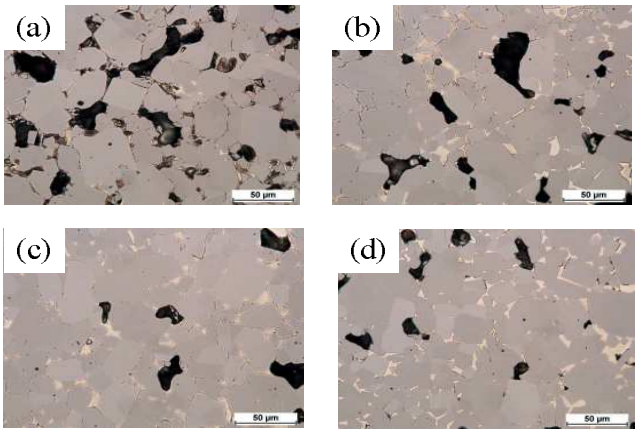

Fig. 2. LM micrographs of spark plasma sintered WC-6Co tool materials for (a) 3 min and at 300°C/min, (b) 3 min and at 600°C/min, (c) 6 min and at 300°C/min, (d) 6 min and at 600°C/min

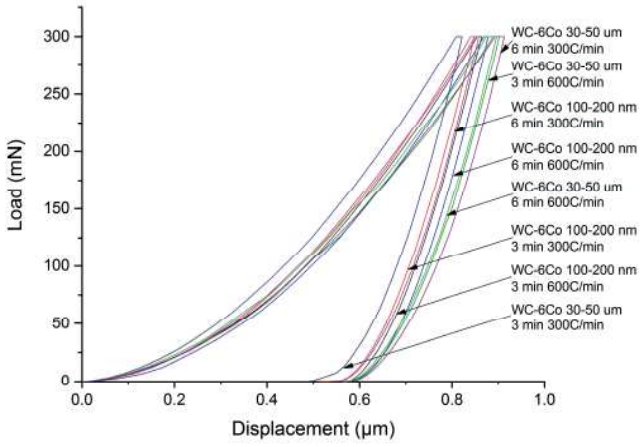

Fig. 3. Nanoindentation load-displacement curves of WC-6Co tool materials spark plasma sintered with various holding times and heating rates

Based on the results presented in Table 1, it was demonstrated that there is a correlation between the hardness and the holding time and heating rate in the spark plasma sintering of WC-6Co powders. The nanohardness of the spark plasma sintered WC-6Co compacts produced from powder of an ultrafine particle size is 2089 and 1912 $HV_{0.031}$ for the holding times amounting to 3 and 6 min and a heating rate of 600°C/min. By heating the powder at 300°C/min, sintered materials were obtained with a higher hardness equalling 2144 and 2096 $HV_{0.031}$ for the same holding time.

This means that the nanohardness of the spark plasma sintered compacts from ultrafine particle size powder increases with a reduction in the holding time and reduced heating rates. The same relationship was observed for the sintered materials produced from micrometre powder. It is necessary, however, to bear in mind that measurement of the nanohardness of these materials, because of the resulting microstructure was performed only on the WC grains. This means that our results do not take into account the Co phase and porosity, and thus do not give a complete view of the actual hardness of the cermets. Nevertheless, the measured nanohardness of the WC grains are 1739 and 1382 $HV_{0.031}$ for the holding times amounting to 3 and 6 min and the heating rate of 600°C/min. In the sintered compact produced using the twice lower heating rate equalling 300°C/min, the WC grains have a higher nanohardness of 1982 and 1487 $HV_{0.031}$ for the holding times amounting to 3 and 6 min. The obtained results are much lower than the data presented in [9], where a nanohardness of 40.4 GPa was revealed. Nonetheless, it should be kept in mind that Duszová et al. in their nanoindentation test used a much lower load ranging between 0.1 and 10 mN. Moreover, as is well known, the hardness increases with decreasing the applied load and thereby the penetration depth. Moscicki et al. [10] demonstrated that the microindentation hardness of a spark plasma sintered WB_x ceramic is 21.9 GPa for an applied load of 5 N. An increase in load up to 294 N causes a decrease in hardness to 18.5 GPa.

An analogous relationship, as in the case the nanohardness, was demonstrated for the elastic modulus. E_{IT} of the spark plasma sintered WC-6Co compacts produced from the ultrafine particle size powder are 654 and 649 GPa for the holding times amounting to 3 and 6 min and heating rate equalling 600°C/min. By heating the powder at 300°C/min, cermets with a higher elastic modulus amounting to 673 and 652 GPa for the the same holding time were obtained.

In the case of the sintered materials from the micrometer powder, EIT are 607 and 541 GPa for the holding time amounting to 3 and 6 min and heating rate equaling 600°C/min. The cermets prepared using the twice lower heating rate equaling 300°C/min display a larger elastic modulus amounting to 674 and 550 GPa for the the same holding time. The obtained results of elastic modulus are similar to the data presented in [8-9].

Table 1.
Results of nanohardness, elastic modulus, susceptibility to brittle cracking and work cracking of WC-6Co tool materials spark plasma sintered with various holding times and heating rates

	Indentation nanohardness (H_{IT}), $HV_{0.031}$	Elastic modulus (E_{IT}), GPa	Susceptibility to brittle cracking, µm/N	Work cracking (W_t), µJ
WC-6Co (100-200 nm)				
3 min / 300°C/min	2144 ± 55	673 ± 10	0.60 ± 0.01	0.09 ± 0.00
3 min / 600°C/min	2089 ± 45	654 ± 13	0.59 ± 0.01	0.09 ± 0.00
6 min / 300°C/min	2096 ± 30	652 ± 18	0.59 ± 0.01	0.09 ± 0.00
6 min / 600°C/min	1912 ± 150	649 ± 6	0.57 ± 0.02	0.09 ± 0.00
WC-6Co (30-50 µm)				
3 min / 300°C/min	1982 ± 245	674 ± 56	0.57 ± 0.01	0.09 ± 0.01
3 min / 600°C/min	1739 ± 139	607 ± 52	0.57 ± 0.03	0.10 ± 0.01
6 min / 300°C/min	1487 ± 240	550 ± 85	0.57 ± 0.04	0.11 ± 0.01
6 min / 600°C/min	1382 ± 360	541 ± 97	0.53 ± 0.03	0.13 ± 0.03

The abrasion resistance of the materials is generally greater, the higher the hardness thereof [11]. Hard materials used for cutting tools are generally subject to typical abrasion, whereby one should bear in mind that in operating conditions of shock, the wear process takes place primarily through cracking, and only to a limited extent by typical wear. Given the above, the susceptibility to brittle cracking of the obtained spark plasma sintered WC-6Co tool materials was determined. The susceptibility to brittle cracking of these cermets is at a similar level and ranged between 0.57 and 0.60 µm/N. Only in the case the cermet produced from the micrometer powder, characterized by the lowest nanohardness and elastic modulus and spark plasma sintered for 6 min at 600°C/min, is the susceptibility to brittle cracking equal to 0.53 µm/N. Of course the highest value of this parameter (0.59-0.60 µm/N) is noted for WC-6Co cermets obtained from the powder with ultrafine particles and characterized by the highest hardness (over 2000 $HV_{0.031}$). The work cracking of WC-6Co tool materials made from a powder having a particle size in the range of 100-200 nm, independent of the applied holding time and heating rate, is 0.09 µJ. Slightly higher values of W_t are noted for the WC-6Co tool materials obtained from the microparticle powder and ranged between 0.09 and 0.13 µJ. It means that the work cracking of these materials slightly increases with a significant decrease in nanohardness of the WC grains.

The work cracking of spark plasma sintered WC-6Co tool materials obtained from microparticle powder characterized by a nanohardness of 1982 $HV_{0.031}$ is 0.09 µJ, the same as the cermets obtained from the ultrafine particle powder. In contrast, the work cracking of WC-6Co tool materials characterized by the lowest nanohardness and spark plasma sintered for 6 min and at 600°C/min is 0.13 µJ.

4. Conclusions

The technological characteristics determined by means of a modern Picodentor HM500 device presented in the paper, are the main characteristics of the suitability of WC-Co cermets fabricated by spark plasma sintering of tool material. It has been shown that the nanohardness, elastic modulus and work cracking increase with shortening of the holding time and a reduction in the heating rate in contrast to work cracking which decreased. In the case of the cermets fabricated from the WC-6Co micrometer powder, because of the resulting microstructure, nanohardness measurements were carried out on the WC grains. This means that our results do not take into account the Co phase and porosity, and thus do not give a complete view of the actual hardness of the cermets. The best results of nanohardness (2144 $HV_{0.031}$), elastic modulus (673 GPa), susceptibility to brittle cracking (0.60 µm/N) and work

cracking (0.09 μJ) were obtained by the WC-6Co tool materials spark plasma sintered for the holding time of 3 min and at the heating rate of 300°C/min from powder with the particle size of 100-200 nm.

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