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EFFECT OF COARSE-TO-FINE WC GRAIN RATIO ON MECHANICAL PROPERTIES AND ABRASIVE WEAR OF WC-8Co CEMENTED CARBIDES

WPŁYW UDZIAŁU WĘGLIKA WC GRUBOZIARNISTEGO DO DROBNOZIARNISTEGO NA WŁAŚCIWOŚCI MECHANICZNE I ZUŻYCIE ŚCIERNE WĘGLIKÓW SPIEKANYCH WC-8Co

Key-words:

cemented carbides, hardmetals, grain size manipulation, WC-Co

Słowa kluczowe:

węgliki spiekane, sterowanie wielkością ziarna, WC-Co

Abstract

This study performs a comprehensive analysis concerning the amount of fine tungsten carbide (WC) grains needed for the appropriate reinforcement of the cobalt (Co) metallic binder in WC-8Co cemented carbides. The goal is to investigate the balance of coarse-to-fine grain distribution to achieve overall improvement of the material's mechanical and wear properties. All samples

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possessed the same WC-8Co binder content, therefore, allowing the role of grain size distribution to be tested. It was found that a ratio of 8:1 wt% of coarse to ultrafine grain WC yielded an appropriate balance between material hardness, fracture toughness, and rupture strength. Upon adding grain growth inhibitors vanadium carbide (VC) and chromium carbide (Cr_3C_2), the overall wear resistance is further improved compared to undoped composites when samples are tested under abrasive wear conditions.

INTRODUCTION

Cemented carbides have been well studied for the last century, especially the basic composite of tungsten carbide and cobalt (WC-Co). The classic WC-Co hardmetal offers a unique combination of refractory hardness and metallic toughness, while undergoing wear stresses in aggressive environments **[L. 1]**. Heavy industries, among them mining, tunnelling, and drilling have deployed WC-Co tool bits in a wide variations. The degree of industrial relevance for hardmetals in this case is reflected by their deployment in the tunnel boring machine (TBM) technology as drag bit inserts **[L. 2–7]**. When a drag bit breaks down due to broken inserts, the replacement operation is both time consuming and a potentially dangerous task since it involves hyperbaric working environment **[L. 7]**. Any endeavour to increase the lifespan of the drag bit against a wide multitude of wear or mechanical failure during operation leads to the decrease in TBM downtime and industrial cost.

On the microscopic level, the WC-Co system offers a versatile material produced from powder metallurgical methods. Its mechanical and wear properties can be controlled by manipulating the carbide grain’s size, distribution, binder content, and even production pathway **[L. 8-10]**. In terms of mechanical properties, the WC-Co system tends to balance its toughness and hardness by the amount of metallic binder. Various attempts to overcome this limitation have forced researchers to focus on using double cemented (DC) carbides, which contains granules of WC/Co cemented carbide in a matrix of cobalt. The composite exhibits a superior combination of fracture toughness and high-stress wear resistance compared to conventional cemented carbides **[L. 2]**. The early 2000s established a method using submicron and ultrafine WC grains in refractory tool development as an ideal approach, especially when combined with improved pressing behaviour, proper doping, and optimized processing **[L. 11]**. However, contemporary research has indicated that a major wear mechanism of near-nano grade WC is related to micro-cracking and micro-chipping leading to the detachment of the WC-Co agglomerates. Conversely, a major wear mechanism of coarse grade WC-Co tools is related to the wear of the binder interlayers among the large WC grains **[L. 5]**. Recent developments have shown a novel ultra-coarse WC-Co grade with Co-based binder reinforced with nano-particles termed MASTER GRADES **[L. 6]**. The composite is characterized by a combination of higher hardness and transverse rupture strength compared to conventional grades of WC-Co without binder reinforcement. A recent effort to improve the classic WC-Co composite has been related to redesigning the microstructure and the proportions of grains, particularly the interaction between the coarse and fine grains.

Two methods of enhancing cemented carbide WC-Co wear and mechanical properties are examined concurrently: (a) using ultrafine WC grains to improve the wear resistance by reinforcing the metallic binder **[L. 12]**, and (b) using micron WC grains to increase the toughness and resistance to crack propagation **[L. 13]**. These two concepts are well documented; however, little research has been conducted about combining the two principle. The goal of this study is to create a composite material with a gradient of grain sizes, where coarse WC grains are surrounded by fine WC grains cemented together by a metal binder. In theory, this microstructure should result in simultaneous hardening and toughening effects on the classic WC-8Co composite, as reported in **[L. 12]**. The finer WC provides the hardening effect, while the coarser WC grains allow for crack bridging. A series of WC-8Co samples were synthesised in order to test the ratio of coarse-to-fine grain mixture and the effects on mechanical properties and abrasive wear resistance. The effect of VC and/or Cr_3C_2 grain growth inhibitors **[L. 14]** is also studied concerning their effects on these composites. Scanning electron microscopy (SEM) images and grain size distribution graphs are shown along with mechanical properties and wear properties.

MATERIALS AND METHODS

Commercial powders $WC_{0.1\mu m}$ (99% WC DN-4.0) and $WC_{10\mu m}$ (99% WC HC-1000) made up the base of the refractory carbides. Other additive carbides were sourced as followed: $VC_{0.9\mu m}$ (99% VC-8002), and $Cr_3C_{2(2.0\mu m)}$ (99% CrC-7002). The binder material was $Co_{0.3\mu m}$ (99.3% 9721-79). To make the hardmetal composites, the ratios between coarse-to-fine WC grains are of foremost importance. The composite samples are detailed in **Table 1**, where samples B1, B2, and B3 show the increasing percentages of coarse $WC_{10\mu m}$ to ultrafine $WC_{0.1\mu m}$ grains and samples C1 and C2 are comparable control samples containing only $WC_{0.1\mu m}$ or $WC_{10\mu m}$ grains in cemented carbides, respectively. From the three B samples, one in particular stood out and inspired an additional experiment producing B2A containing grain growth inhibitors.

The process to create the samples was divided into two steps: The first step involves creating a binder material embedded with ultrafine WC grain, and the second step involves adding $WC_{10\mu m}$ grains into the milling. In the first step, $WC_{0.1\mu m}$ – 8Co was subjected to high-energy attrition milling: ball-powder (BP) ratio 8:1 under ethanol at 800 rpm for 4 h. The $WC_{0.1\mu m}$ – Co powder was dried, sieved, and then pre-sintered to 1200°C in a vacuum to yield a brittle bulk cemented carbide material. This bulk material was then high-energy attrition milled again with a BP ratio 12:1 wt. % under ethanol at 800 rpm for 1 h. During second step, the required content of $WC_{10µm}$ powder was added and the mixture was subjected to ball milling using WC-Co balls and containers at a BP ratio of 6:1 wt % with ethanol for 24 h before being dried and sieved. The samples were pressed at 15 MPa (at room temperature) and underwent SinterHIP at 1450°C (heating rate 10°C min^{-1}) in a vacuum for 15 min and then in argon gas for 15 min with a pressure of 30 bars.

Table 1. Composition of samples (wt.%)
Tabela 1. Skład próbek (wt.%)

Samples	Ratio $WC_{10µm}$: $WC_{0.1µm}$	VC / Cr_3C_2	Co
B1	4:1	-	8
B2	8:1	-	8
B3	14:1	-	8
B2A	8:1	0.4 / 0.4	8
C1	Pure $WC_{0.1µm}$	-	8
C2	Pure $WC_{10µm}$	-	8

The test samples were ground and then polished down to 1µm using diamond paste before undergoing tests including Vickers hardness, fracture toughness by Palmqvist via indentation under 147 N load **[L. 15]**, density by Archimedes, and transverse rupture strength by three-point bending. Abrasive wear testing was done in a way similar to ASTM-132 **[L. 16]**. A sharp corner of a test sample was pressed against a rotating disc covered by P180 (82 µm) SiC abrasive paper. A CETR/Bruker UMT-2 tribometer with servo-controlled loading was used for testing. The frequency of rotation was – 12 rpm; load – 0.5 N; sliding distance – 135.7 m; duration – 1 h. Fresh abrasive was continuously supplied into the wear zone. Volume lost was evaluated by measuring the dimensions of the wear scar remaining after testing and by applying the equation for the calculation of the volume of the pyramidal area lost. An optical microscope Discovery V20 SteREO equipped with AxioVision software from *Carl Zeiss* was used. Test results are reported as the volume loss in cubic millimetres per load and distance (mm^3 N^{-1} m^{-1}).

The mean free path (MFP) of Co was determined using SEM images and the Line Intercept Method **[L. 17]**. A set of parallel equidistant lines was applied onto the SEM image. The length of the lines crossing the grains indicated the size of the grain. Grain size distribution graphs are plotted with the indication of the relative frequency of the grains. The total length of lines between grains divided by the number of MFPs gives the average value of MFP **[L. 18, 19]**.

RESULTS AND DISCUSSIONS

Effect of coarse-to-fine WC grains ratio on mechanical properties

The morphology of the ultrafine $WC_{0.1\mu m}$-Co powder after attrition high-energy milling is shown in **Fig. 1**. The SEM shows the ultrafine grains of WC embedded into the larger Co particles. When mixed with the coarser $WC_{10\mu m}$ grains, the samples B1, B2, and B3 have varying proportions between coarse-to-ultrafine WC grains. The mechanical properties are displayed in **Table 1**, and the respective trends in the relative density and mean free path (MFP) are of particular interest. With increasing coarse $WC_{10\mu m}$ grain content, the composites' relative densities decrease. This trend is also reflected in research done by Liu et al **[L. 13]**, where the addition of micron WC grains to ultrafine WC showed similar results. However, contrary to their research, we detected a steady increase in composite hardness increasing from 1267 HV_{30} with sample B1 to 1315 HV_{30} with B3 with increasing $WC_{10\mu m}$ concentration. In **Fig. 2**, a plot showing all samples featuring hardness and fracture toughness indicated a peak fracture toughness value of 12.4 $MPa{*}m^{1/2}$ along with hardness 1282 HV_{30} for B2. Fracture toughness is typically prioritized over hardness for industrial tool bits, and especially for cemented carbides **[L. 5, 20]** to reduce brittle fracturing **[L. 6, 21–23]**.

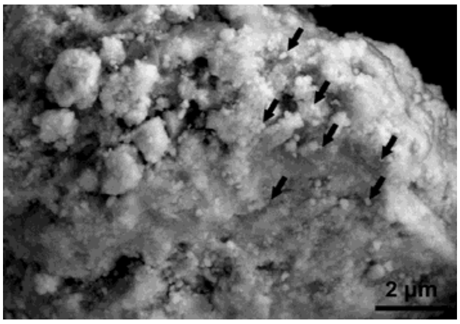

Fig. 1. $WC_{0.1\ \mu m}$–Co reinforced binder powder after milling. Black arrows indicate some of fine WC grains

Rys. 1. Spoiwo kobaltowe wzmocnione węglikiem wolframu (WC0,1 µm–Co) po mieleniu. Czarne strzałki wskazują drobne ziarna WC

Table 2. Density and mechanical properties of sintered hardmetals

Tabela 2. Gęstość i właściwości mechaniczne badanych kompozytów

Sample	Relative density [%]	MFP [µm]	Hardness [HV30]	K_{IC} [$MPa{*}m^{1/2}$]	TRS [MPa]
B1	98.8	0.47	1267 ± 12	11.3 ± 1.5	2842 ± 99
B2	98.4	0.49	1282 ± 16	12.4 ± 1.8	3012 ± 80
B3	97.7	0.55	1315 ± 14	12.1 ± 1.1	2809 ± 88
B2A	98.9	0.48	1287 ± 22	12.0 ± 1.4	3468 ± 92
C1	98.2	-	1315 ± 17	10.1 ± 1.5	2770 ± 51
C2	97.0	-	1350 ± 13	12.2 ± 1.5	2584 ± 98

Samples B1-B3 display an increasing MFP with an increasing coarse $WC_{10\mu m}$ concentration, as indicated in **Table 2**. It has been known that, as the mean size of WC grain size increases, so too does the mean free path **[L. 22]**. Typically, with WC cemented carbides, the fracture toughness is linearly correlated with mean free path of the binder phase regardless of the shape and contiguity of the WC crystals **[L. 24]**. However, in our case, the overall strength and toughness of the composites seems to be correlated to a particular proportion of coarse to ultrafine WC grains, since the optimal ratio is 8:1 wt.% in sample B2. However, this study has reversed the ratios of the previously published research on bimodal WC-Co production of $WC_{ultrafine}$ to WC_{micron} grain size mixtures. Yang et al. **[L. 12]** studied a $WC_{0.1\mu m}$ to $WC_{1\mu m}$ ratio of ~7.6:1 wt.%, while Liu et al. **[L. 13]** studied a $WC_{0.2\mu m}$ to $WC_{1\mu m}$ ratio of ~8:1 wt.%. In the latter case, a series of alloys were created with varying proportions of micron grain ratios; one of their alloys (~8:1 ultrafine to micron WC) showed an intercept point where hardness and fracture toughness properties were highest. Both these studies reflect the same principles of the research done in this study, where the ratio of 8:1 could be an optimal ratio when two proportions of WC grains (whether small to large, or large to small as in this study) work to improve the overall mechanical properties of the composites.

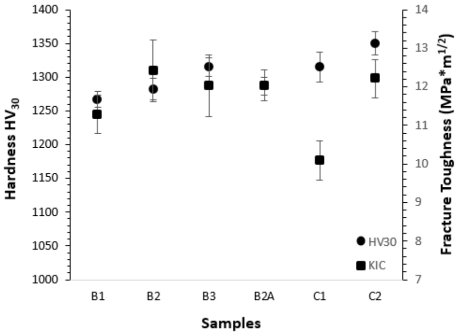

Fig. 2. Hardness and fracture toughness of cemented carbides

Rys. 2. Twardość i udarność próbek z węglików spiekanych

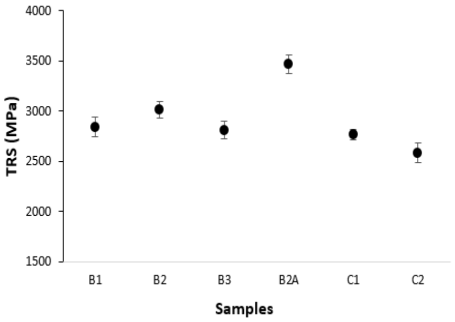

Fig. 3. Transverse rupture strength of cemented carbides

Rys. 3. Poprzeczna wytrzymałość na pękanie próbek z węglików spiekanych

Figure 3 shows the transverse rupture strength (TRS) of all samples based on three-point bending tests. Again, from the series of B1-B3, B2 is the sample that stands out as having improved strength due to its grain ratio of 8:1 wt. % (B2). Certainly, all experimental samples ranging from B1-B3 showed improved mechanical properties over the controls C1 & C2, indicating that the addition of an increasing concentration of $WC_{10\mu m}$ and, for that matter, the presence of a proportional gradient of large to small WC grains can affect and improve the mechanical properties of the overall composite. Looking at the SEM image in **Fig. 4** of the B2 cross-section after bending strength testing, plenty of indications of transgranular fractures amongst the coarse carbide grains could be seen. This is characterised by slip lines across the coarse WC-Co cemented carbides. It is believed that the plastic deformation and rupture of the Co binder phase ligaments contributes the major portion of the dissipative work during the rupture of WC–Co cemented carbides due to its high value of the critical strain energy release rate **[L. 22, 25, 26]**. This would explain the result with sample B2 having both high TRS and fracture toughness values as seen in **Fig. 2** and **Fig. 3** out of the three experiment samples. The microstructure cracks are forced to pass over the energy barrier when they propagate across WC grains, the binder, and their interface, which enhance the resistance against the crack propagation **[L. 22]**. Lastly, considering sample B2A, which is similar to sample B2 but doped with VC/Cr_3C_2 showing the highest TRS value among all samples. The presence of a grain growth inhibitor also makes the WC grains more faceted and the cobalt distribution more uniform, which prevent agglomeration **[L. 27]** leading to better densification as shown in **Table 2**.

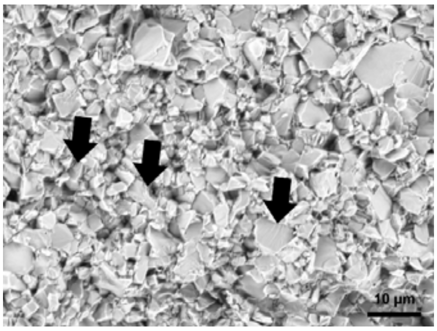

Fig. 4. SEM image of B2 sample after transverse rupture strength test, where black arrows indicate transgranular cracks and slip lines on the carbides

Rys. 4. Obraz SEM próbki B2 po teście poprzecznej wytrzymałości na pękanie, czarne strzałki wskazują płaszczyzny pęknięć ziaren i kierunki poślizgu

Figure 5 displays SEM images for all experimental composites along with their grain size distribution charts. Although the smallest WC grains used in this study were ~0.1 µm, the sintering of ultrafine WC leads to grain growth during sintering, especially at the early stages. The initial rapid grain growth is at least partially attributed to the process of coalescence of grains via the elimination of common grain boundaries **[L. 28]**. The grain growth of the ultrafine WC grains follows the classic change during the heating process from equiaxial to a faceted platelet shapes between 800°C to 1200°C **[L. 29]**. Continuous grain growth during liquid-phase sintering may be considered to be similar to Ostwald's ripening process. Smaller WC particles dissolve due to their higher dissolution potential and re-precipitate after diffusion through the binder at coarser WC grains **[L. 11]**. As such, given the sintering parameters and method of milling, there is no presence of ultrafine WC0.1 µm in the microstructure, since most of such grains have either been absorbed into the coarse grains or grown tenfold due to re-precipitation process.

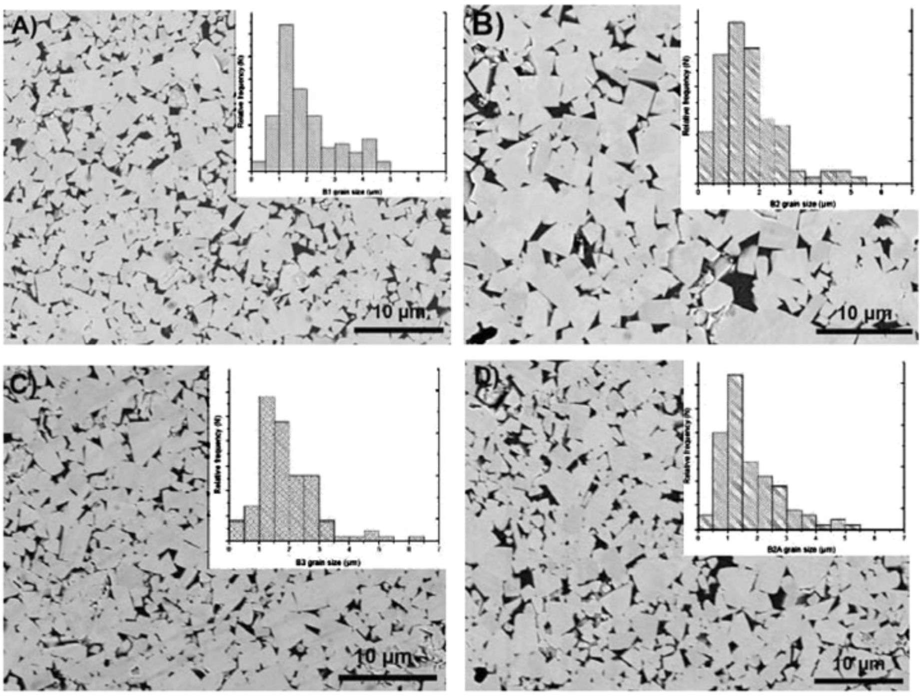

Fig. 5. SEM micrographs of composite microstructure with accompanying grain size distribution charts: A) B1, B) B2, C) B3, and D) B2A

Rys. 5. Obrazy SEM mikrostruktury próbek kompozytu z rozkładami wielkości ziaren: A) B1, B) B2, C) B3, D) B2A

Considering the grain distribution charts, sample B1 has a narrower distribution of WC grain sizes when compared to other materials. There is no indication of visible $WC_{10\mu m}$ grains in any of the samples suggesting the method of ball milling, using WC-Co balls and containers and milling for up to 24 h was too severe, effectively reducing the size of the coarse WC grains. Hence, there seems to be a 50% reduction in coarse WC grain size throughout all microstructures. Nevertheless, there is an uneven (with two local maxima) and wide distribution of WC grain sizes of samples B2, B2A, and B3. These results, along with their mechanical properties, seem to contradict the established research in WC-Co strength and grain size distribution. Typically, the low contiguity and narrower (more uniform) range of grains means that stresses and elastic energies from fractures are evenly distributed across the microstructure and leading to improved strength **[L. 22, 30]**. In our case, the wider distribution of grains seems to show improved mechanical properties in samples B2 and B2A.

Effect of coarse-to-fine WC grains ratio on abrasive wear rates

The control C1 and C2 materials both exhibit the characteristics of their respective grain sizes with decreasing density and reduced hardness, but with an increase in strength when going from fine to coarse WC grains in **Table 2**. Of course, the ultrafine $WC_{0.1 \mu m}$ would yield lower fracture toughness compared to the $WC_{10 \mu m}$ hardmetal. It has also been shown in field testing of tool bits for the mining industry that hardness is not always a determining factor in abrasive wear resistance **[L. 4–6, 31]**. As shown in **Fig. 6**, both the C1 and C2 perform worse under abrasive wear conditions than experimental products combining ultrafine and coarse grains of the same binder concentration. Ultrafine $WC_{0.1 \mu m}$-Co grains of C1 are so small that they could be removed together with the binder, while the soft cobalt binder between coarse grains of C2 ($WC_{10 \mu m}$-Co) is not sufficiently protected and is suffering from wear that finally leads to fracturing or removal of grains. There is a particular threshold in the ratio between coarse and ultrafine WC grains content in the case of sample B2, where rupture strength, hardness, fracture toughness, and wear resistance **(Figs. 2, 3, 6)** are optimised. Wear resistance is gained from the use of mixed grain sizes in the composite. Larger WC grains are stabilizing the tribolayer and minimizing removal due to scratching while smaller WC grains are protecting the binder that is holding the large grains and providing ductility.

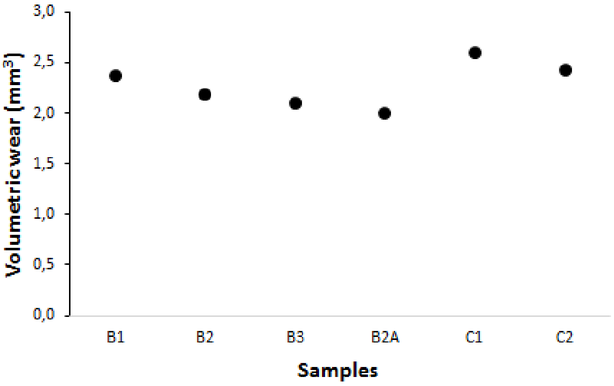

Fig. 6. Volumetric loss of samples during abrasive wear test
Rys. 6. Zużycie objętościowe próbek po badaniu zużywania ściernego

Samples B1-B3 show a general increase in wear resistance with an increasing concentration of $WC_{10\mu m}$ grains. The additions of grain growth inhibitors further amplify the properties of the composite B2 in both strength and wear resistance. In the sample B2A, the addition of grain growth inhibitors is supposed to supress the coalescence of coarse and ultrafine grains. The grain growth inhibitors are supposed to reduce the sliding friction of the composite leading to greater wear resistance **[L. 27, 32, 33]**. The mechanical properties of samples with suppressed WC grain should be emphasized, because both the TRS value and wear resistance of the composite are greatly enhanced. B2A displays 10 to 20% higher wear resistance as compared to other samples in **Table 2**, even though its hardness is lower compared to C1 or C2, and it is comparable to B2. These results certainly motivate continued research into mixed grain size composites. The major advantage with this approach is the fact that it does not change the basic chemical or elemental composition of the material, but it merely redesigns how they are synthesised. In this case, tungsten carbide and cobalt chemically remains the same, but the grain size and distribution are changed to offer new properties. The addition of grain growth inhibitors such as VC and Cr_3C_2 is a known technique. The pathway taken to produce the final product offers a potential that can minimise the need for drastically new approaches to manufacturing.

CONCLUSIONS

In this study, it has been shown that the mechanical properties and wear resistance of the classic WC-8Co can be enhanced by introducing a suitable distribution of different size grains in the system. Experiments reveal that, by choosing the optimum coarse-to-fine WC grain size ratio, it is possible to increase the toughness and bending strength of the WC-8Co cemented carbide. The sample B2 demonstrates that a ratio of 8:1 wt %. of coarse-to-fine grains seems to be the optimal proportion for the system with wide grain size distribution possessing two local maxima. It is possible to produce a WC-8Co with mechanical properties of $K_{IC} \approx 12.4$ MPa*m$^{1/2}$, and TRS ≈ 3010 MPa for sample B2, while the addition of grain growth inhibitors in B2A resulted in at least a 10 % better abrasive wear resistance and bending strength as compared to B2. B2A also has 20 % better wear resistance when compared to single grain size reference samples with either only $WC_{10\mu m}$ or $WC_{0.1\mu m}$ grains.

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Streszczenie

W artykule przedstawiono wyniki badań dotyczących wpływu ilości drobnoziarnistego węglika wolframu na wzmocnienie metalicznego spoiwa kobaltowego (Co) w węgliku spiekanym WC-8Co. Celem badań jest znalezienie optymalnego udziału węglika gruboziarnistego do drobnoziarnistego dla uzyskania poprawy właściwości mechanicznych i charakterystyk zużyciowych. Dla wszystkich próbek zastosowano jednakowy udział spoiwa, by zbadać jedynie wpływ udziału grubo- do drobnoziarnistego węglika w spieku. W wyniku przeprowadzonych badań stwierdzono, że dla proporcji 8:1 udziału węglika grubo- do drobnoziarnistego uzyskuje się najkorzystniejsze cechy użytkowe, tj.: twardość, udarność i odporność na pękanie. Ponadto poprzez dodanie do kompozytu inhibitorów wzrostu, np. węglika wanadu (VC) lub węglika chromu (Cr_3C_2), zwiększa się odporność na zużycie ścierne w stosunku do kompozytów bez dodatku inhibitorów.