Recycling of WC-Co hardmetals by oxidation and carbothermal reduction in combination with reactive sintering

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Abstract. A novel recycling technology for the production of bulk WC-Co hardmetals from a mixture of WO_3, $CoWO_4$ and graphite powders by way of carbothermal reduction in combination with reactive sintering has been developed. Waste hardmetals parts with 15 wt% Co from hardmetals production were fully oxidized into a mixture of WO_3 and $CoWO_4$ powder. To oxide powder mixtures, carbon was added in the form of nanocrystalline graphite, milled, pressed into compacts and sintered. During reactive sintering carbothermal reduction, tungsten monocarbide (WC) synthesis and structure formation occurs in one cycle. The influence of different graphite content in the initial powder mixtures on the phase composition, and linear shrinkage during solid and liquid state sintering is discussed. The microstructure of reactive sintered WC-Co composites is fine-grained and identical to that of the original WC-Co microstructure and has similar mechanical properties.

Key words: hardmetals, recycling, microstructure evolution, carbothermal reduction, reactive sintering.

1. INTRODUCTION

The increasing use of materials with special properties and the introduction of new cost-efficient manufacturing processes have had a decisive influence on the further development of hardmetals.

Various methods for recycling WC-Co hardmetals, such as chemical modification, thermal modification, zinc melt, cold stream and electrochemical methods have been investigated and some of them are actually being employed in industry [1–3]. However, these conventional methods present many unresolved problems and are not always based on well-established technologies. For

example, the chemical modification method requires large-scale equipment, and reaction times are relatively long. Recycling by the thermal modification method usually involves the decomposition of WC and leads to the formation of the η-phase, which degrades mechanical properties. Finally, the zinc melt and cold stream methods have problems with contamination by undesirable elements [4,5].

It is known that WC-Co hardmetals can be easily transformed into a sub-micron particle size oxide mixture of $CoWO_4$ and WO_3 by oxidation and mechanical milling processes [6,7].

Direct carburization of WO_3 is particularly desirable from a practical point of view, but a problem arises in controlling both the particle size and the carbon content. Generally, reduction of WO_3, carried out between 800–1000 °C, ensures adequate control of the particle size, whereas a minimum temperature of 1200 °C is required for carburization. This temperature difference makes it difficult to combine both processes in a single operation [8]. However, several authors have reported that the reduction and carburization of WO_3 with C under proper reactive conditions is possible [9–14]. Several researchers [15,16] have also found that the carburization of tungsten at around 900 °C is possible in the presence of cobalt, due to the catalytic effect. Finally, in [15] it is reported that WO_3 can be carburized in hydrogen at about 900 °C in the presence of Co_3O_4.

WC-Co composite powders with pure phases and homogeneous and ultrafine particles can be synthesized from tungsten oxide, cobalt oxide and carbon black at 1323 °K under vacuum conditions. Compared with conventional reaction methods, while intermediate products such as $CoWO_4$ play an important role in reducing the activation energies of the reactions, this novel method has the distinct advantage of lower reaction temperature and shorter holding time [17,18]. It has been found that it is possible to achieve dense cemented carbide bulk by in situ synthesis in the SPS system [19].

In our previous paper [20] we presented the mechanical properties and micro-structure of hardmetals produced by carbothermal reduction of oxide powders, and it was found that the properties of recycled WC-Co materials were mainly influenced by the content of additional graphite. The same phenomenon also occurs with reactive sintering of bulk Cr_3C_2-Ni parts from Cr, Ni and C mixtures [21]. Carbon content has a great effect on the properties of sintered WC-Co composites. There is a very narrow range of ±0.1 wt% carbon where the WC-Co microstructure remains in the two-phase region. At the W : C atomic ratio of less than 1 the carbon precipitates in the form of graphite, and at the W : C ratio significantly over 1, Co_xW_yC (η-phase) is formed in the structure. During carbo-thermal reduction and sintering, reduction of the oxides and a loss of carbon occur. However, this drawback can be overcome through the addition of some "extra" carbon to the mixture to compensate for the carbothermal reduction and decarburization in the furnace during sintering [22].

Free carbon content in sintered parts is generally considered detrimental to the mechanical properties of sintered WC-Co, since its presence reduces hardness

and wear resistance. Thus, it is necessary to adjust the free carbon concentration in $CoWO_4$ and WO_3 powder mixtures, depending on the application.

According to [23], shrinkage of fine-grained WC-Co composites occurs mainly during the solid state phase and is completed in the liquid phase stage. Solid-state shrinkage results in Co spreading to the WC particles and the rearrangement of WC particles, which dissolve in Co and become facetted. Grain growth during liquid phase sintering of the WC-Co alloys can be described as an Ostwald ripening process [24]. According to this model, the reduction in surface energy of solid particles is the major driving force behind the dissolving of small grains and growth of large grains. Although this solution/re-precipitation requires diffusion through the liquid phase binder, the rate is controlled by interface kinetics due to the faceted morphology of the WC grains, where growth occurs by a 2-dimensional nucleation or defect assisted process [25]. Another mechanism for WC grain growth is particle coalescence, involving atomic diffusion across solid /solid grain boundaries.

Due to the disadvantages of conventional recycling methods, in this paper we propose a new route for the recycling of hardmetals by a combination of oxidation, carbothermal reduction and reactive sintering. This new method has the advantage of simpler processing. We examine the influence of different graphite contents in the initial WO_3 and $CoWO_4$ powder mixture, produced by ball milling and high energy milling, on the shrinkage of test specimens during carbothermal reduction and reactive sintering, microstructure evolution, and the mechanical properties of recycled WC-Co hardmetals.

2. MATERIALS AND EXPERIMENTAL PROCEDURE

Commercially available WC-Co hardmetals scrap was used as raw material. The scrap parts were manufactured using Boart Longyear G30 hardmetals powder by the conventional hardmetals production method. Hardmetals scrap with 15 wt% Co binder phase content was used. The WC-Co scrap was washed with distilled water. The WC-Co scrap was oxidized in a rotary kiln at 850 °C in a flowing stream of air. During oxidation the surfaces of the specimens developed a green-blue-yellow oxide coating, and many microcracks formed. As the tube inside the furnace rotated, the soft oxide layer was removed from the surfaces of the WC-Co specimens. Two oxide phases of $CoWO_4$ and WO_3 formed during oxidation. The oxide of the WC-15 wt% Co hard metal had low strength due to its sponge-like microstructure and the presence of microcracks.

Eight types of powder mixtures were investigated with the same chemical composition but different graphite content and different milling procedures. The crushing and mixing were carried out in both an attritor (high energy milling) and ball mill. The powders were mixed for 72 h using a ball mill with WC-Co balls and pure ethanol as the liquid medium. The ball-to-powder weight ratio was 5 : 1, and the rotation rate of the mill was 60 rpm. In the attritor, the powders were

mixed for 6 h, using pure ethanol as the liquid medium. In order to minimize any possibility of contamination, the vial, impellers and milling balls were made from WC-Co hardmetals. The starting powder mixture was placed in the vial (1 l) with a ball-to-powder weight ratio of 5 : 1. The vial was cooled with water circulation throughout the process. The milling speed was set at 560 rpm. The composition of the alloys investigated is given in Table 1.

The initial concentration of free carbon (in the form of graphite) in the WO_3 and $CoWO_4$ powder mixtures was 16, 16.5, 16.8, and 17 wt%. No grain growth inhibitors were added. After milling, the powders were air-dried at 40 °C and pelletized into small spheres of about 200 μm in diameter. The mechanically activated WO_3, $CoWO_4$ and C mixtures were compacted into $6 \times 6 \times 40$ mm blocks by uniaxial pressing at 80 MPa. The green compacts with a green density of 3.25 g/cm^3 were directly sintered at different temperatures for 1 h in a graphite-heated furnace in a vacuum greater than 10^{-4} bar. Interrupted sintering experiments were performed in the Sinter/HIP furnace.

Sample length was measured with a precision of 0.01 mm before and after sintering. The microstructure was investigated by SEM (JEOL JSM 840A) after various stages of sintering. Phase identification was carried out using X-ray diffraction (XRD) methods with CuKa radiation (Bruker AXS D5005). Shrinkage was determined from recorded changes in sample dimensions during heating at different temperatures.

The hardness of the samples was measured using a Vickers pyramid indenter. Measurements were made under a load of 10 kgf using a load time of 30 s. An average hardness value was determined based on 5 indentations.

Transverse rupture strength was determined in accordance with the ASTM Standard B406-95 by three-point method using the device "Instron 8516". Each test point indicates the average value of 5 measurement results.

The microstructure and mechanical properties of the recycled WC-Co materials were compared with those of the original conventional Boart Longyear G 30 hardmetal.

Table 1. Nominal composition of alloys investigated

Material notation	Amount of additional carbon, wt% C	Powder preparation
R16B	16.0	Ball mill
R16.5B	16.5	Ball mill
R16.8B	16.8	Ball mill
R17.0B	17.0	Ball mill
R16A	16.0	Attritor
R16.5A	16.5	Attritor
R16.8A	16.8	Attritor
R17.0A	17.0	Attritor

3. RESULTS AND DISCUSSION

3.1. Carbothermal reaction and phase formation

A number of studies have shown that WC-Co composites can be synthesized from a mixture of metal oxides and carbon by carbothermal reaction at 950°C to 1100°C in a vacuum or in a flow of inert gas [17,18,22,26,27].

Figure 1 shows the changes in chemical composition during carbothermal reduction at different temperatures in vacuum. The oxide powder mixture consists of WO_3 and $CoWO_4$ (line A). The dilatometer tests and TGA showed that the reduction process starts at 900°C, involving a mass loss of about 30%. After reduction for 1 h at 950°C, peaks of W_2C and WC appear, and the mixture also contains dual carbides and some remains of WO_3 (line B). It was found that 1 h is not sufficient time for reducing green parts with a weight of less than 10 g. The phenomena can be explained by different reduction conditions on the surface layer and at the core of the green parts. After reduction at 1150°C, the material mainly consists of WC and Co (line C). The appearance of W peaks show that the formation of WC continues at higher temperatures. After sintering at 1400°C for 30 min, the recycled hardmetals consists of WC and Co.

Figure 2 illustrates microstructure evolution during sintering over a temperature range of 1250°C to 1400°C. During sintering a significant increase of WC grain size and decrease of porosity was detected. As seen in Fig. 2a, sintering at 1250°C leads to a hardmetals microstructure with high porosity, which can be attributed to the lack of a liquid binder phase at lower temperatures.
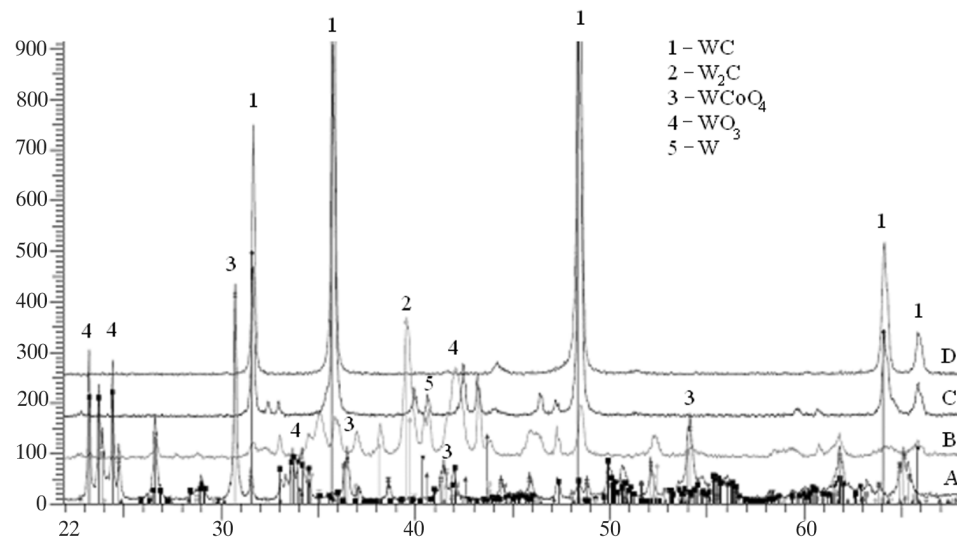

Fig. 1. XRD patterns for powder mixtures of WO_3 and $CoWO_4$ with a 16.8 (C) wt% of additional carbon at different temperatures: A – without carbothermal reduction; B – reduction at 950°C for 1 h; C – reduction at 1150°C for 1 h; D – sintering at 1400°C for 30 min.
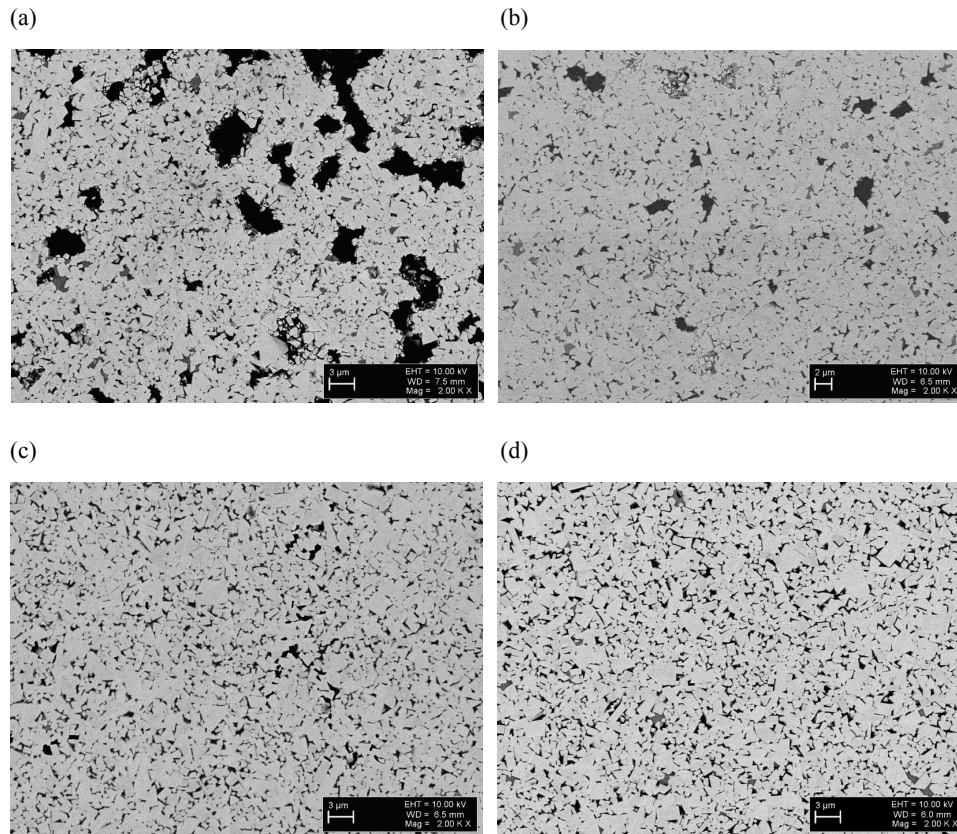
Fig. 2. Structural evolution of WC-15 wt% Co hardmetals at different temperatures during reactive sintering: a – 1250 °C; b – 1330 °C; c – 1380 °C; d – 1400 °C.

The liquid phase appears at about 1300 °C, but even after sintering at 1330 °C, the hardmetals still exhibits some porosity, due to the high viscosity of the binder material (Fig. 2b). A non-porous microstructure can be achieved by sintering at 1380 °C to 1400 °C (Fig. 2c and d).

3.2. Densification and shrinkage behaviour

The densification behaviour of the W-xC-15 Co powder compacts, sintered between 900 °C and 1400 °C, is shown in Fig. 3. The shrinkage of the compacts increased with an increase in sintering temperature. Sintering at temperatures between 900 °C and 1050 °C has a significant effect on the sample dimensions, indicating that remarkable densification occurs at these temperatures. As seen from Fig. 3, the linear shrinkage of sintered blocks at 900 °C is between 5% and 10%, depending on chemical composition and particle size distribution (attritor vs ball mill). Shrinkage is caused by the reduction of oxides and the formation of carbides. Intensive shrinkage by the rearrangement of particles can be explained
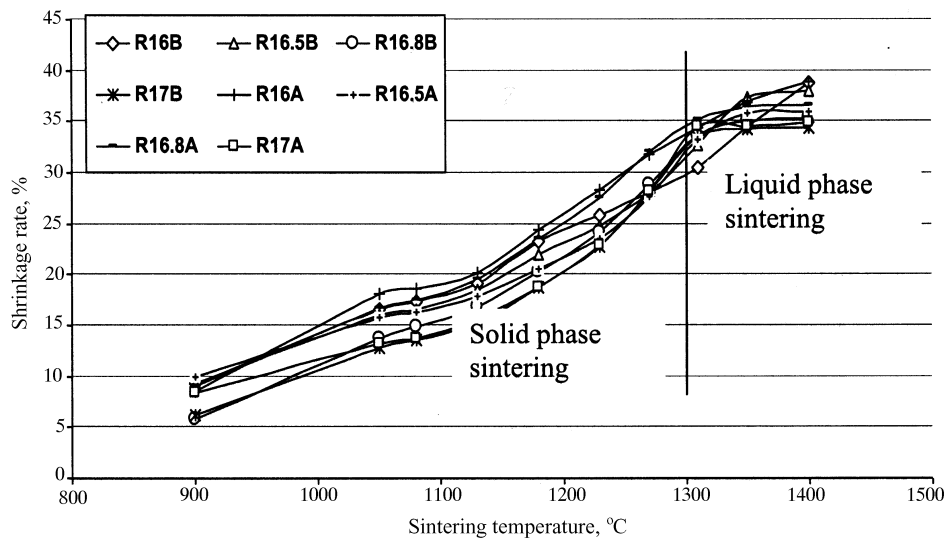
Fig. 3. Linear shrinkage of W-xC-15Co hardmetals with differing amounts of additional carbon and different methods of powder preparation.

by a low effective viscosity, generated by defects in the contact region between particles. The creep of the Co binder is assumed to be the local process, producing solid state rearrangement.

It was found that the CO/CO_2 gas, produced during the carbothermal reduction, does not damage the green parts. This can be explained by the low green density and open porosity, which allow the gases to exit the compact without damaging the structure. The relatively high linear shrinkage rate at lower temperatures indicates that the sintering processes of mechanically activated materials starts at lower temperatures. As seen in Fig. 3, the mixtures, prepared by high energy milling (R16.8A), have higher shrinkage rates at lower temperatures than the materials, prepared by conventional ball milling (R16.8B).

Rapid densification occurred at the temperature range of 900 °C to 1050 °C and 1130 °C to 1300 °C. Using TGA tests, it was found that the liquid phase appears at 1295 °C. This shows that during carbothermal reduction and subsequent to reactive sintering more than 90% of the densification took place in solid phase sintering. The WC-15Co hardmetals, sintered from oxide powders, showed rapid shrinkage and full densification at a lower temperature than conventional W, Co and C powder mixtures, which underwent reactive sintering [21]. During heating, the solubility of WC in the cobalt binder increases uniformly as the temperature increases. However, during isothermal holding, the equilibrium solubility is quickly reached due to very short diffusion distances in the binder phase, and thereafter, no further dissolution occurs. The linear shrinkage of the samples, produced by carbothermal reduction in combination with reactive sintering, was 40%–45%, which is about twice more than that of samples, produced with conventional technology.

3.3. Microstructure

The microstructure of the WC-Co hardmetals consists of WC grains, embedded in a cobalt-rich binder phase. Since WC is known to be precisely composed stoichiometrically and does not dissolve any cobalt, the liquid cobalt phase ought to dissolve W and C in atomically equal proportions during the liquid phase sintering. There is a very narrow range of 6.12 ± 0.1 wt% carbon where the WC-Co microstructure will remain in the two-phase region. In practice, the total amount of C in the material may not agree with the stoichiometric ratio. It should be noted, that too much or too little carbon results in the formation of free graphite or η-phase ($W_x Co_y C$), respectively. Figure 4 shows the microstructure of the material, produced by oxidation and carbothermal reduction with the optimal amount of additional graphite and the microstructure of the original hardmetals.

The powder mixture of hardmetals in Fig. 4a was prepared by ball milling and compacted by uniaxial pressing at 80 MPa and sintered at 1400°C for 30 min. As seen in Fig. 4, the hardmetals produced by carbothermal reduction and reactive sintering have a fine-grained microstructure. However, an investigation of the cross-sections of the hardmetals produced revealed that depending on the content of additional carbon before carbothermal reduction and sintering, inner zones with abnormal phase compositions formed. As seen in Fig. 5a, the lack of free carbon during WC phase formation leads to the formation of η-phase rich areas in the outer layer and to a lack of binder material at the core of the sintered specimen. This can be explained by the onset of WC formation and sintering on the surface layer of the blocks produced. The lack of carbon causes the formation of η-phase which results in the consumption of Co from the inner layers of the sintered part.
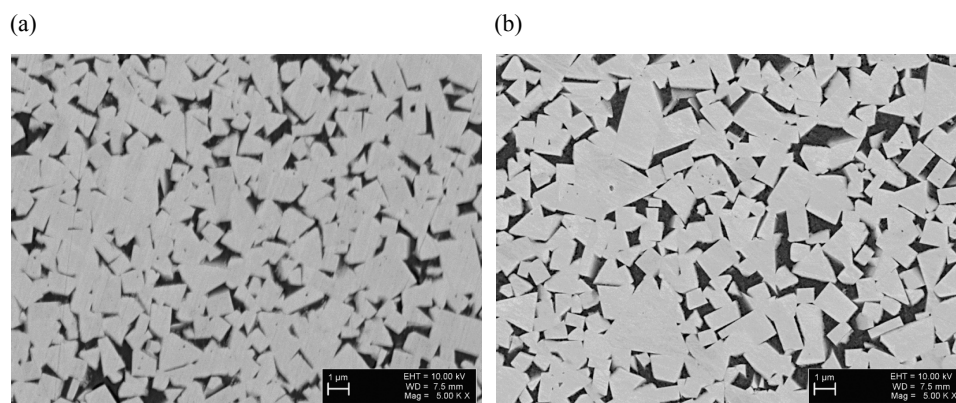
(a)   (b)

Fig. 4. The microstructure of recycled W-xC-15Co hardmetals with 16.8 wt% C of additional carbon (a) and the original Boart Longyear G30 hardmetal (b).

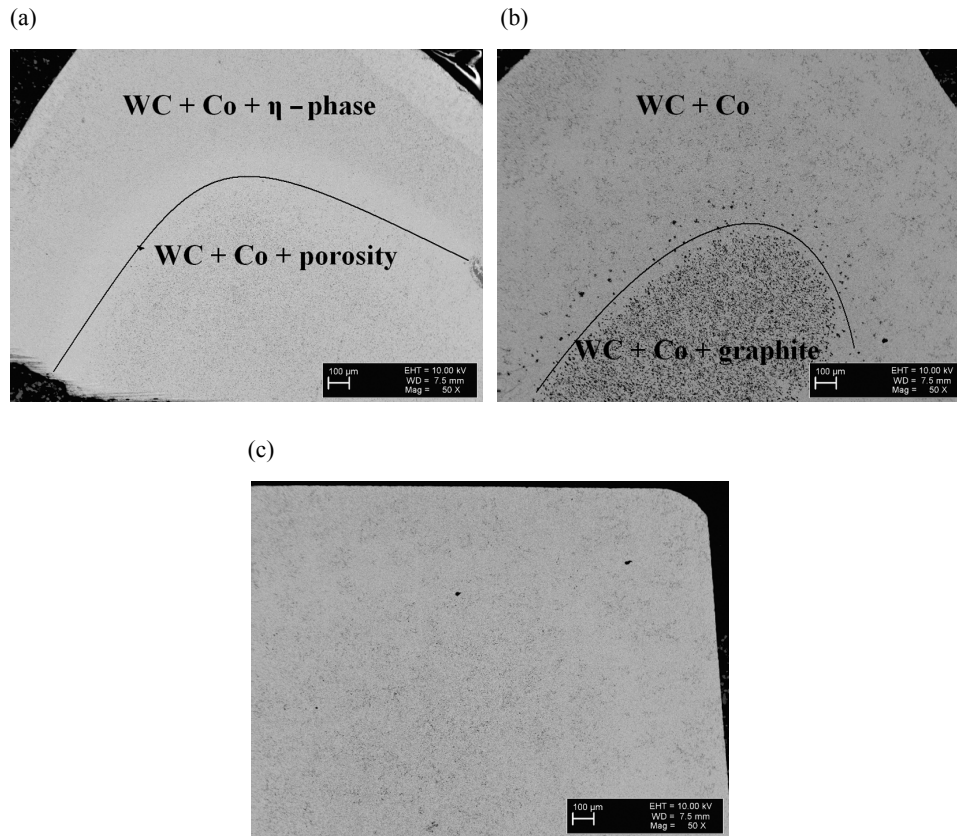(a)   (b)

(c)

Fig. 5. Phase formation after sintering by 16.5 wt% (a), 17.0 wt% (b) and 16.8 wt% (c) of additional carbon before reduction and sintering (all mixtures were milled in a ball mill).

Having too much additional carbon in the mixture (Fig. 5b) causes the formation of a graphite rich zone at the core of the sintered material. It was noted that both porosity and graphite flakes arose only at the inner core of the sintered material. In both cases the microstructure of the outer layer of the sintered specimens consists of fine-grained WC and Co phases. In the case of compacts obtained from a mixture with 16.8 wt% of additional carbon (Fig. 5c), WC and Co phases filled the cross-sections of the specimens, and the formation of η-phase and free graphite was not detected.

Figure 6 illustrates the microstructures of sintered hardmetals, produced by ball milling and high energy milling in an attritor. It was found that the hardmetals prepared by high energy milling in an attritor has coarser WC grain than materials prepared by ball milling. Grain size analysis shows that the hardmetals, prepared by attritor and ball mill have similar grain size distribution (Fig. 7), but the mean grain size of hardmetals prepared by ball milling is 0.88 μm compared to 1.08 μm for hardmetals prepared using an attritor. The

phenomenon can be explained by the more intensive grain growth of the mechanically activated mixture due to the relatively long heating period (the heat-up rate was 2 °C/min) and holding time.
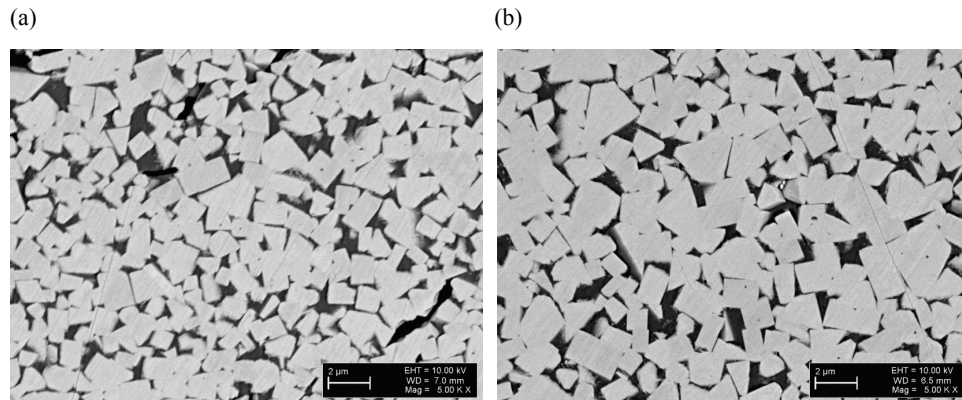
(a)   (b)

Fig. 6. The microstructures of WC-15Co hardmetals prepared by ball mill (a) and high energy milling in an attritor (b).

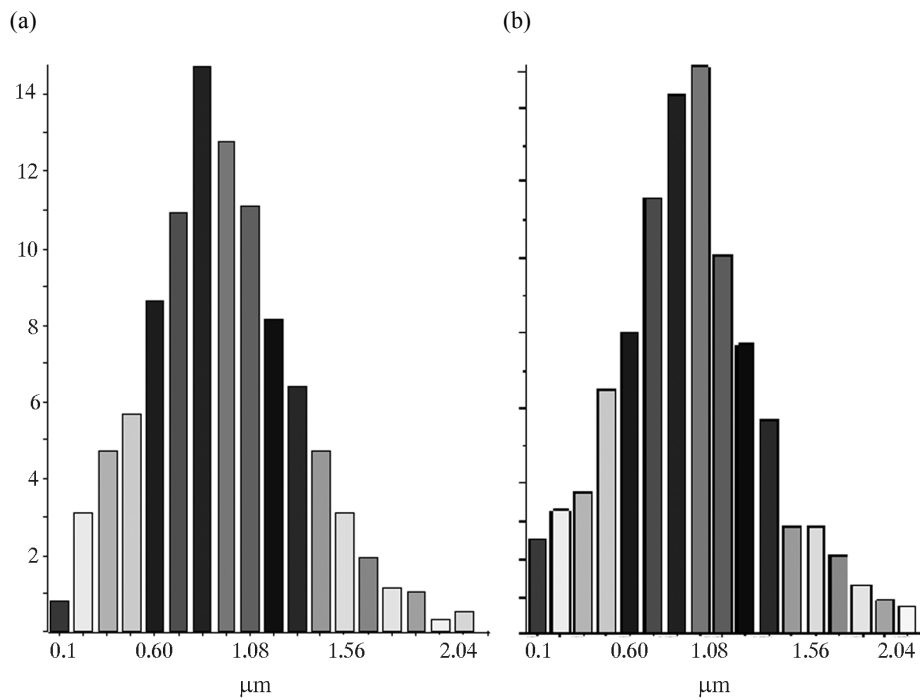(a)   (b)

Fig. 7. Grain size distribution of hardmetals prepared by ball mill (a) and high energy milling in an attritor (b).

3.4. Mechanical properties

The mechanical properties of tested materials are presented in Table 2. It was found that materials with a sufficient amount of additional carbon have the same mechanical properties as the hardmetals before recycling. The density of hardmetals, produced using 16.8 wt% of additional carbon, was 14.0 g/cm^3, which is the same as that of the hardmetals, produced using conventional WC-Co powder and the conventional production method. The relatively high hardness and very low strength of hardmetals produced, using a smaller amount of additional carbon, can be explained by the formation in the hard phase of a rich surface layer and porous core, as seen in Fig. 5.

4. CONCLUSIONS

The following conclusions can be drawn from the present work.
1. A novel method for recycling WC-Co composites from waste hardmetals scrap lies in the oxidation and carbothermal reduction of compacted parts in combination with subsequent reactive sintering in a vacuum furnace.
2. The optimal graphite content in the initial oxides powder mixtures was determined. The amount of 16.8 wt% graphite powders in initial mixtures guaranteed two-phase WC-15 wt% Co alloys without free graphite and γ-phase after reactive sintering.
3. The relatively high linear shrinkage rate at lower temperatures (950 °C) indicates that the sintering processes of mechanically activated powders starts at lower temperatures. The linear shrinkage of samples was up to 40%.
4. The recycled WC-15Co hardmetals have the same chemical composition as the original material, as well as similar grain size and mechanical properties.

Table 2. Mechanical properties of W-xC-15Co hardmetals with different levels of carbon additions and methods of powder preparation

Material notation	Amount of additional carbon, wt% C	Hardness HV10	TRS, MPa
R16B	16.0	1350	–
R16.5B	16.5	1200	1500
R16.8B	16.8	1350	2500
R17.0B	17.0	1100	2100
R16A	16.0	1300	–
R16.5A	16.5	1250	1300
R16.8A	16.8	1300	2500
R17.0A	17.0	1200	2100
Original Boart Longyear G30		1300	2500

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WC-Co kõvasulamjäätmete ümbertöötlemine oksüdeerimise ja karbotermilise taandamise teel koos reaktsioonpaagutusega

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Uus taaskasutusmeetod seisneb WC-Co kõvasulamjäätmete oksüdeerimises, mille tulemusena tekib WO_3 ja $CoWO_4$ pulbrisegu, ja selle termilises taandamises grafiidiga koos järgneva kõrgenergeetilise jahvatuse, pressimise ning reaktsioonpaagutusega. Uuriti grafiidi sisalduse mõju lähtepulbreis karbotermilisele taandamisele, volframmonokarbiidi tekkele, sulami mikrostruktuuri moodustumisele, toorikute kahanemisele ja kõvasulami mehaanilistele omadustele. Meetodi teiseks iseärasuseks on see, et taandamine, karbiidi süntees ja sulami struktuuri moodustumine toimub ühes tsüklis – reaktsioonpaagutuse käigus. Leiti, et uudsel meetodil jäätmeist ümbertöödeldud WC-15Co kõvasulamite mikrostruktuur ja mehaanilised omadused on sarnased originaalsulamitele.