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THE STRUCTURE AND MECHANICAL PROPERTIES OF WC–8 wt.% Co HARDMETAL PRODUCED BY COLD AND HOT ISOSTATIC PRESSING

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The structure and properties of tungsten carbide hardmetal with 8 wt.% Co produced by cold and hot isostatic pressing with the same starting powder mixture and conventional initial pressing of billets in metal dies were studied. The first batch of the samples was prepared by vacuum sintering of the pressed billets. The second batch was prepared by vacuum sintering followed by hot isostatic pressing of the billets at 5 MPa. The third batch was prepared by hot isostatic pressing at 200 MPa followed by vacuum sintering. The sintered samples had a density of 14.57 g/cm^3 in batch 1, 14.60 g/cm^3 in batch 2, and 14.63 g/cm^3 in batch 3. Microstructural analysis showed that cold isostatic pressing promoted more homogeneous and finer structure. According to the coercive force determination and structural analysis performed with scanning electron microscopy and X-ray diffraction, the average size of carbide grains was 1.315 μm for the samples in batch 1, 1.396 μm in batch 2, and 1.062 μm in batch 3. Determination of residual stresses indicated that they were compressive in both phases (WC and Co) of the batch 3 samples and tensile for the batch 1 and 2 samples. The average values of measured Rockwell hardness were 88, 87, and 90 HRA for the samples in batches 1, 2, and 3. Mechanical tests of the samples indicated that the bending strength and fracture toughness were 1820 + 110 MPa and 18.9 + 1.2 MPa · m$^{1/2}$ for the samples in batch 1, 2030 + 130 MPa and 18.2 + 1.1 MPa · m$^{1/2}$ in batch 2, and 2040 + 120 MPa and 18.6 + + 1.2 MPa · m$^{1/2}$ in batch 3. The high mechanical properties of the hardmetal are determined by structural variations and change from tensile to compressive residual stresses in the batch 3 samples.

Keywords: *hardmetal, cold isostatic pressing, mechanical activation, grains, cobalt layer, residual stresses, hardness, bending strength, fracture toughness.*

INTRODUCTION

Tungsten carbide–cobalt hardmetals feature exceptionally high mechanical properties, such as hardness, three-point bending strength R_{bm}, and fracture toughness [1]. Hardmetals are conventionally produced by die pressing and liquid-phase vacuum sintering. Sintering is a predominant process for the WC–Co hardmetals in acquiring their microstructure and, consequently, properties [2].

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We established in our previous paper [3] that cold isostatic pressing (CIP) influenced the powder mixture as early as in the compaction stage by increasing the billet density and thus improved the properties of the sintered samples. Cold isostatic pressing of billets prepared from tungsten carbide and cobalt powders to 400 MPa before their sintering allows the production of WC–Co hardmetals with higher hardness than that of the hardmetals manufactured conventionally. The VK8 hardmetal samples subjected to preliminary CIP at 200 MPa were found [3] to have the maximum density and minimum WC grain size [3].

The main techniques for the production of hardmetals, such as vacuum sintering (VS) and hot isostatic pressing (HIP), are widely used in industry as they are inexpensive and can be implemented on a large scale [4]. However, we did not find papers that would examine the effect of CIP on the mechanical properties of WC–Co hardmetals. If CIP qualitatively improves the functional properties of WC–Co hardmetals, this operation can become an effective step in the hardmetal production process.

Hot isostatic pressing can be employed both as the main method for the sintering [5] of billets or in the processing of preliminarily sintered samples [6, 7]. Hot isostatic pressing is used to avoid significant evaporation of cobalt in the liquid-phase sintering [8], densify preliminarily sintered samples, remove pores [5], and improve interphase bonding. Hot isostatic pressing improves strength and fracture toughness of the sintered materials [9]. Nevertheless, methods associated with long-term heat treatment cause grain growth [4], and the starting hardmetal mixtures thus need to be additionally doped with WC grain growth inhibitors [10], which increases the prime cost of hardmetal articles.

It was previously shown that the mechanical properties of hardmetals with finer grains could be substantially improved [11]. The most popular method to control the WC grain growth is to add grain growth inhibitors to the starting hardmetal mixtures. New methods for rapid synthesis of composites are under development as well.

Hot pressing [12], spark plasma sintering [13], impact sintering [14], and microwave sintering [15] allow the process temperature and time to be decreased as compared to vacuum sintering and promote the production of dense samples with finer grains [16]. However, the WC–Co hardmetals fabricated with these methods commonly show lower mechanical properties (strength and fracture toughness) than the composites produced by liquid-phase sintering [17]. These methods did not find wide application in the powder industry because of high production costs [18] and scaling complexity. They are primarily used for laboratory research [4].

Our objective is to examine the mechanical properties of VK8 hardmetal samples produced by conventional die pressing followed by vacuum sintering, vacuum sintering followed by hot isostatic pressing (VS + HIP), and preliminary CIP of billets at 200 MPa followed by vacuum sintering (CIP + VS).

EXPERIMENTAL PROCEDURE

The starting VK8 (WC–8 wt.% Co) hardmetal mixture was prepared from tungsten carbide and cobalt powders that were mixed in a 5% solution of synthetic rubber in gasoline, the proportion being 0.15 L of the solution to 1 kg of the mixture, in a vibration mill for 2 h. The mixture was sieved to acquire the necessary process characteristics. The average WC particle size determined according to [3] was equal to 0.49 μm. The paper [3] reported the average WC particle size of 0.65 μm.

After being pressed in a steel die at 70 MPa, the samples were dried at 120°C for 24 h. Then they were placed into latex shells, vacuumed, and subjected to CIP at 200 MPa since, as shown in [3], sintered samples with the maximum density and minimum WC grain size, which is a precondition for imparting high mechanical properties to the VK8 hardmetal, were produced from billets subjected to CIP exactly at 200 MPa. The objective of the paper [3] and this research effort is to find process options that would improve the service properties of hardmetal products and would be easy to implement.

Preliminary CIP was performed using multiplicative high-pressure equipment through compression of transformer oil in a high-pressure container with experimental samples. A part of the samples was not subjected to CIP. The samples were sintered in a vacuum furnace at 1440°C. Following sintering, the samples that did not undergo CIP were subjected to HIP in a '10 MPa vacuum pressure sinter–HIP' furnace at 1430°C and 5 MPa argon

pressure for 30 min. The main reason for using HIP after the sintering of WC hardmetals is that this operation transforms coarse pores present in the vacuum-sintered samples into fine ones. Following sintering and HIP, all samples were ground and polished. The samples were 5 mm × 5 mm × 35 mm in size.

The density ρ (g/cm^3) of all samples was measured by hydrostatic weighing with a VLR-200m laboratory balance. The coercive force H_c (kA/m) was measured with a Kobalt-1 meter. The bending strength R_{bm} was determined in compliance with a standard powder metallurgy procedure (ISO 3327 standard) [19]. Tests were performed using a U-10-1 universal machine to find the breaking load. The test bar moved at a speed of 2 mm/min. To ensure that the data are reliable, at least seven samples were used to measure the strength, the average deviation from the average value being no more than 3%.

The first batch of samples was prepared by vacuum sintering of pressed billets (VS). The second batch was prepared by vacuum sintering followed by HIP at 5 MPa (VS + HIP). The third batch was prepared by preliminary CIP at 200 MPa followed by vacuum sintering (CIP + VS).

The structure was examined employing a REM-106I scanning electron microscope. Quantitative metallography, involving processing of scanning electron microscopy images with the JMicrovision software [20], was used to determine the residual porosity [21], tungsten carbide grain size [22], and cobalt layer thickness. The stress strain state was analyzed using X-ray diffraction employing a Rigaku Ultima IV diffractometer and different methods such as the Rietveld and RIR methods in Cu–$K_{\alpha 1,2}$ radiation ($\lambda_{Cu-K\alpha 1}$ = 0.1541 nm). Residual stresses were found with the $\sin^2\psi$ method. Since the surface of VK8 hardmetal products wears out in operation and is subject to cracking that may cause damage, we examined the surface structure, phase composition, and hardness of the samples. The Rockwell hardness was measured with a TK-2 hardness tester at a load of 588 N. The three-point bending strength was determined with a U-10-1 mechanical testing machine (*Vymiryuvach* Company). The fracture toughness was measured on polished sample surfaces with the Palmqvist method including indentation with a diamond Vickers pyramid at a load of 490.5 N. For this purpose, a KhPO 250 hardness meter was used (WPM, Germany). The recalculation was performed in accordance with the standard procedure described in [23].

EXPERIMENTAL RESULTS

Table 1 summarizes the experimental results. Noteworthy is high hardness of the CIP + VS samples and the fact that it is much greater than hardness of the hardmetal HIP samples. Note also that fracture toughness decreases with greater strength both in our research effort and in the paper [24]. Structures of the sintered samples (Fig. 1) demonstrate differences in WC grain sizes. At the same time, the CIP + VS samples hardly contain coarse WC grains. It is also seen that all main structural characteristics are lowest in the CIP + VS samples (Table 1). The coercive force values correlate with the WC grain sizes and cobalt layer thickness [25], specifically: the thinner the cobalt layer, the higher the coercive force. The residual stresses in the batch 2 samples (VS + HIP) are tensile and those in the batch 3 samples (CIP + VS) are compressive. In the batch 1 samples (VS), compressive stresses are observed only in cobalt. Note that a breaking crack propagates along cobalt in the VK8 hardmetal and, hence, the cobalt layer parameters are important for imparting strength to the hardmetal as cobalt is much more ductile than tungsten carbide.

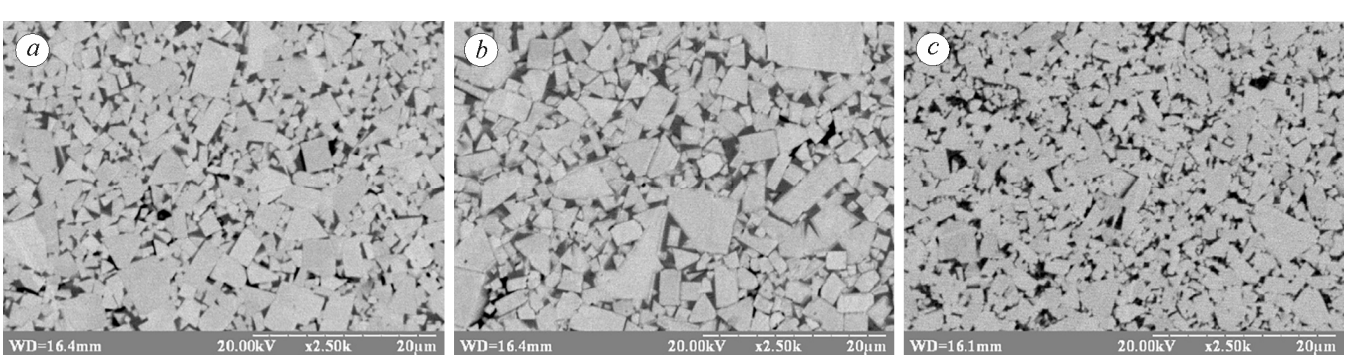

Fig. 1. SEM images of the sintered samples in different batches: VS (*a*); VS + HIP (*b*); CIP + VS (*c*)

TABLE 1. Mechanical Properties and Stereological Characteristics of Samples in Different Batches

Characteristics and properties	VS	VS + HIP	CIP + VS
Density, g/cm^3	14.57	14.60	14.63
Coercive force H_c, kA/m	7.7	6.6	10.0
Average WC grain size, L, μm	1.315	1.396	1.062
Average pore size, μm	0.292	0.201	0.167
Average cobalt layer thickness, μm	0.340	0.347	0.275
Residual stresses in WC, MPa*	+1692.30	+2741.41	–883.23
Residual stresses in cobalt, MPa*	–281.30	+2024.65	–1097.28
Rockwell hardness, HRA	88	87	90
Bending strength R_{bm}, MPa	1820 + 110	2030 + 130	1980 + 120
Fracture toughness, MPa · m$^{1/2}$	18.9 + 1.2	18.2 + 1.1	18.6 + 1.2

* Compressive stresses are shown with a minus sign and tensile stresses with a plus sign.

The JMicrovision software was used to process 20 structural photos for each ceramic batch and plot bar charts to show the distribution of WC grain sizes, pore sizes, and cobalt layer thicknesses (Fig. 2). The CIP + VS samples differ from the VS + HIP and VS samples by greater structural homogeneity, promoting increase in the mechanical properties. The statistical distribution shows that all phase components have minimum sizes in the CIP + VS samples.

Figure 3 displays fracture patterns for the VK8 samples in different batches. The fracture surface of the batch 1 samples significantly differs from those of the batch 2 and batch 3 samples: it is relatively smooth, without irregularities. This means that the moving crack did not change its path. The fracture surfaces indicate that cracks moved along more complex paths in samples of the two other batches.

DISCUSSION OF EXPERIMENTAL RESULTS

The powder subjected to CIP densifies as its particles slide relative to each other and an arch structure forms at this stage from the largest and coarse particles and fails when CIP pressure increases. In the process, the largest WC particles may refine and cobalt particles located between the coarse WC particles undergo plastic deformation. The minimum grain size in the CIP + VS samples may be determined by the following. The friction of particles tightly pressed against each other (mainly coarse particles, because they actively interact from the CIP beginning), which occurs in the densification process in quasiisostatic compression conditions, may substantially heat up the surfaces. The shear stresses induced by friction and temperature rise promote the origination and movement of dislocations in the surface layers of powder particles, i.e., lead to their mechanical activation. This phenomenon is used in cold welding. The stresses that remained in the WC particles after the mixture had been prepared (primarily through the mechanical effects in disintegration and grinding operations) in combination with the applied CIP force resulted in refinement of the carbide particles as they failed.

When coarse single crystalline WC particles are refined (Fig. 4), cracks originate and propagate through quasiisostatic compression of other WC and cobalt particles [26], which naturally complicates the crack propagation and thus can involve the dislocation saturation and mechanical activation of near-surface WC layers [27]. Cobalt is also saturated with dislocations in the plastic deformation process under CIP. Then, in the sintering process, the mechanically activated WC and cobalt layers may cause the eutectic to form at lower temperatures and probably not from fine WC particles, as is commonly the case for VK8 VS samples, but from fragments of the coarse fractured particles with activated surfaces. This, in turn, reduces the fragments of coarse particles and decreases the amount of cobalt required for the eutectic to form. As is shown in the paper [3], within 8 wt.% WC commonly dissolves in cobalt at the eutectic melting point (1320ºC). In case of the CIP + VS samples, this 8% may be presented exceptionally by mechanically activated surface layers of fragmentary coarse WC particles that started dissolving at

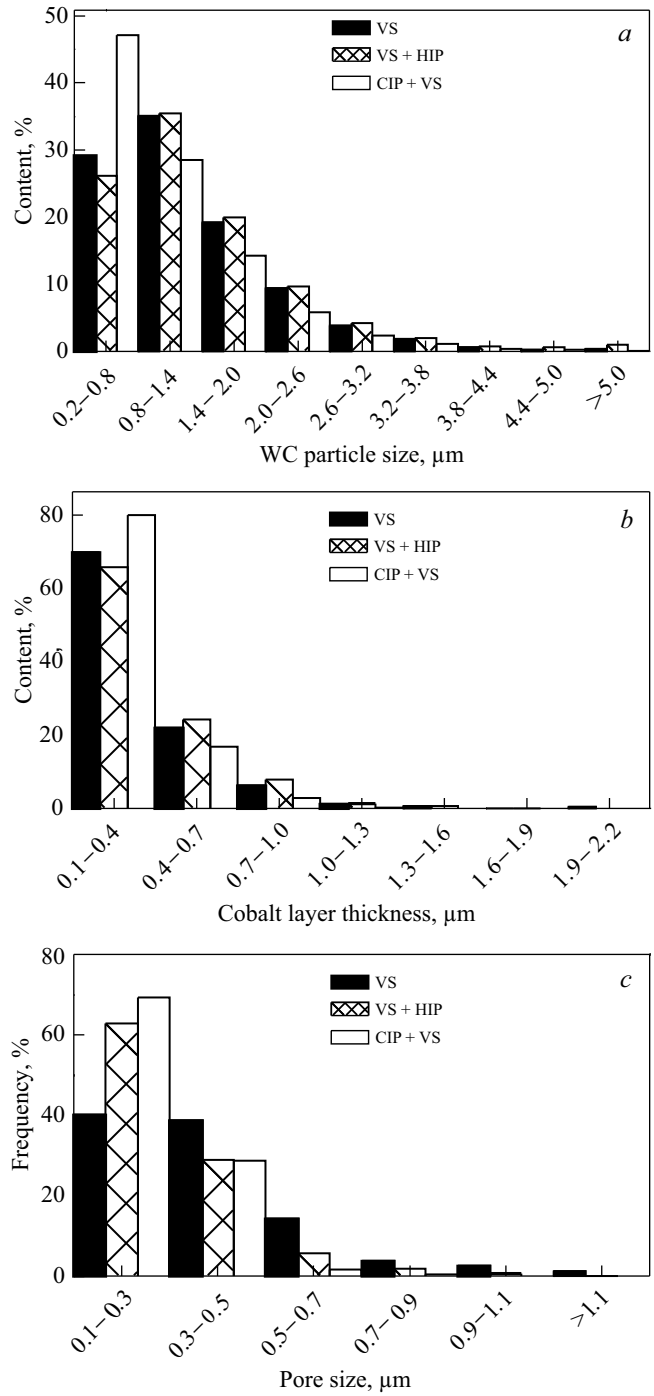

Fig. 2. Statistical distribution of the WC grain size (*a*), cobalt layer thickness (*b*), and pore size (*c*) for the sintered samples in three batches

a temperature probably somewhat lower than 1320ºC, because of the mechanical surface activation of WC and cobalt particles. Ordinary processes, described in [3], occur in further heating to 1440ºC. When the material cools down, a part of the dissolved fragments crystallizes as fine WC grains. Therefore, the average WC grain size decreases, but the grain size distribution in the CIP + VS samples becomes narrower; as a result, the cobalt layer thickness decreases and the mechanical properties improve compared to those of the hardmetal produced conventionally. Hence, we can state that preliminary CIP of the hardmetal samples leads to sintered final hardmetal products with finer average WC grains compared to the samples prepared in other equal conditions.

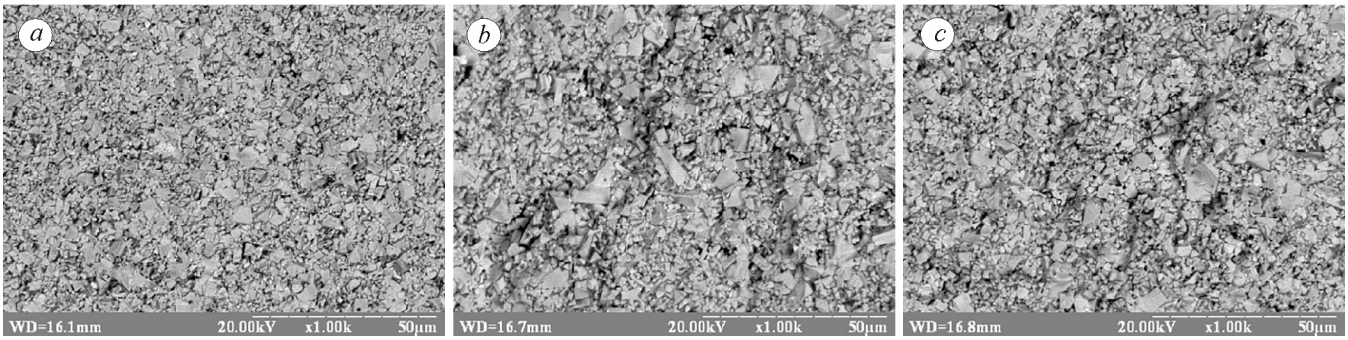

Fig. 3. SEM images of fracture surface structures for the samples in different batches: VS (*a*); VS + HIP (*b*); CIP + VS (*c*)

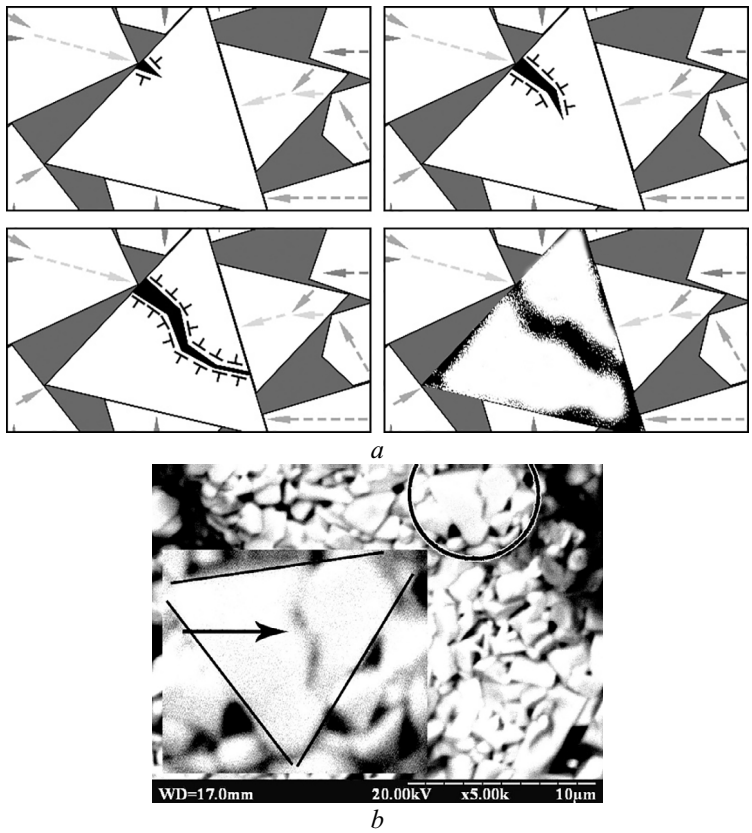

Fig. 4. Fragmentation of WC particles in the CIP process: crack origination and propagation (*a*); SEM image of a fractured WC particle (*b*)

The paper [28] described the role of residual stresses in the formation of VK8 hardness and explained how CIP of the billets could influence residual stresses in WC grains. Tensile residual stresses are known [29] to impair the mechanical properties and compressive ones to improve them. The role of residual stresses is further addressed in the discussion of experimental results. The WC particles in the starting mixture are characterized by tensile stresses (1127 MPa). The cobalt layer in the batch 3 samples (CIP + VS) is thinner than in the batch 2 samples (VS + HIP), which thus have lower ductility margin contributing to higher strength. Nevertheless, this is compensated by compressive residual stresses in cobalt in the CIP + VS samples and, as a result, strength of the batch 3 samples is insignificantly lower than that of the batch 2 samples. The highest hardness of the batch 3 samples (CIP + VS) is due to the finest WC grains and compressive residual stresses in them. Regarding the

properties of the batch 2 samples, they completely correspond to the HIP purpose, specifically: they have finer pores than the batch 1 samples that were not subjected to HIP, and the coarser WC grains and thicker cobalt layer are associated with recrystallization stimulated by additional pressure heating. The grain coarsening and high tensile residual stresses in WC and cobalt reduce hardness, while the thicker ductile cobalt layer promotes the highest strength.

Fracture toughness measurements focus on the cracks that propagate from corners of the indentation left by a diamond Vickers pyramid. For obvious reasons, these cracks propagate along the cobalt layer. Note that the fracture toughness corelates well with data from the fracture analysis of the samples. It is seen (Fig. 3) that the high fracture toughness of the batch 1 VK8 samples (VS) correlates with the smooth continuous fracture surface, and the lowest fracture toughness of the batch 2 samples (VS + HIP) is accompanied by large breaks on the fracture surface. The fracture surface values agree well with the magnitude and signs of residual stresses in the cobalt layers. The residual stresses in WC grains can be explained as follows. First, they were present in the starting mixture. Significant increase in residual stresses in the batch 2 samples may be associated with coarser grains, and change in the sign of residual stresses in the batch 3 samples is most likely due to, first, failure of coarse WC particles with great residual stresses and, second, their dislocation activity that could change the sign and magnitude of residual stresses in these samples.

In analysis of the experimental results, we used data on relation between the mechanical properties and structure from previously published research papers that examined the effect of high isostatic pressure on the failure of single crystals and the refinement of coarse single crystalline WC particles under quasiisostatic compression of powders in the CIP process. These papers show that the deformation and fracture of single crystals at high isostatic pressure are accompanied by a sharp increase in the dislocation density. Each WC particle is compressed on all sides by adjacent WC and cobalt particles, resulting in quasiisostatic compression. However, in quasiisostatic conditions, the forces acting on a particle are not equal and there is always one force that is greater than the others. This force causes fractures that lead to the mechanical activation of other WC particles. This mechanical activation can determine the properties of sintered VK8 samples subjected to preliminary CIP.

CONCLUSIONS

Preliminary CIP of ‘green’ hardmetal billets leads to final sintered hardmetal products with finer average WC grains than in the products fabricated from the hardmetal samples prepared in other equal conditions. The CIP effect is identical to the action of conventional inhibitors used in hardmetal production.

It has been experimentally established for the first time that CIP of the VK8 hardmetal billets at 200 MPa allows the production of samples with 90 HRA hardness after vacuum sintering. This exceeds the hardness of the VK8 samples produced using HIP (87 HRA) and that of the VK8 samples manufactured only by uniaxial compaction of the starting mixture (88 HRA). The three-point bending strength of the samples subjected to preliminary CIP is 1960 + 120 MPa, that of the samples subjected to HIP after sintering is 2030 + 130 MPa, and that of the samples sintered after uniaxial compaction is 1820 + 110 MPa. The stress intensity factor (fracture toughness) of the samples subjected to preliminary CIP is 18.6 + 1.2 MPa · $m^{1/2}$, that of the samples subjected to HIP after sintering is 18.2 + 1.1 MPa · $m^{1/2}$, and that of the samples sintered after uniaxial compaction is 18.9 + + 1.2 MPa · $m^{1/2}$.

The experimental results suggest that CIP of the billets to be sintered favorably influences the mechanical properties of the VK8 hardmetal and can be used in the manufacture of tungsten carbide hardmetal tools.

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