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Properties of WC–Co hardmetals as a function of their composition and microstructural parameters

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Abstract: Research into WC–Co submicron hardmetals involving measurement of hardness, coercivity and microstructural characterization, as well as analysis and comparison of results from recent literature led to the development of a unified constitutive expression for Vickers hardness in a form that separates the effects of the tungsten carbide grain size from those of the cobalt binder volume fraction. With the proposed expression for *HV* one may recalculate and compare hardness values for hardmetals featuring the same average grain size but differing in the binder matrix content. The paper shows that, in contrast to the Lee-Gurland model, the proposed constitutive expression framework treats the hardmetal hardness as a function of the carbide skeleton hardness (H_{WC}) and contiguity (C) described as $HV = CH_{WC}$. The carbide skeleton hardness depends on the WC grain size only, and it is described by the Hall-Petch equation. The results of parallel hardness and coercivity measurements led to an empirical equation relating H_c to the WC grain size and the Co volume fraction. Based on the complete experimental data, the relationship between the coercivity and Vickers hardness was explored, and a simplified relationship between these physical values was proposed to carry out the primary *HV* evaluation based on the measured coercivity values. As noted in the paper, the above equations are valid for relatively narrow WC grain size distributions with a maximum coefficient of variation of 0.5.

Keywords: hardmetal, microstructure, Vickers hardness, coercivity, grain size, binder fraction, carbide skeleton, contiguity.

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Зависимость свойств твердых сплавов WC–Co от их состава и характеристик микроструктуры

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Аннотация: В ходе проведенных исследований субмикронных твердых сплавов системы WC–Co, включавших в себя диагностику твердости, коэрцитивной силы и параметров микроструктуры, а также анализ и сопоставление результатов из современных литературных источников, представлена объединенная модель, согласно которой выражение для твердости по Виккерсу можно представить в виде, позволяющем разделить влияние размера зерна карбида вольфрама и объемного содержания кобальтовой связки. Предложенное выражение дает возможность проводить перерасчет и сопоставлять значения *HV* для твердых сплавов с одинаковым средним размером зерна и различным содержанием связки. В работе показано, что в отличие от модели Ли-Герланда в рамках представляемой модели твердость сплава определяется твердостью карбидного каркаса (H_{WC}) и его смежностью (C) и задается соотношением $HV = CH_{WC}$. При этом величина H_{WC} зависит только от размера зерна карбида вольфрама и описывается уравнением типа Холла–Петча. По результатам параллельных измерений твердости и коэрцитивной силы (H_c) получено эмпирическое уравнение зависимости величины H_c от размера зерна WC и объемного содержания Co. На основании всей совокупности экспериментальных данных исследованы связи коэрцитивной силы и твердости по Виккерсу и предложено упрощенное соотношение между этими физическими показателями, позволяющее проводить первичную экспрессную оценку величины *HV* по измеренным значениям коэрцитивной силы. В работе отмечается, что приведенные соотношения справедливы для относительно узкого распределения зерен WC по размерам с коэффициентом вариации не более 0,5.

Ключевые слова: твердый сплав, микроструктура, твердость по Виккерсу, коэрцитивная сила, размер зерна, содержание связки, карбидный каркас, смежность.

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Introduction

Many reviews and research papers have investigated the relationship between the microstructure and composition of WC—Co grades and their properties such as hardness (*HV*) and coercivity (H_c) [1—4]. Most studies assume that *HV* and H_c values are functions of the WC grain size (d_{WC}) and the volume fraction of the Co binder (V_{Co}). Empirical *HV* and H_c vs. d_{WC} and V_{Co} relationships are used to build the relevant physical models [5—10]. For most d_{WC} measurements, various versions of a single approach, the linear intercept method, are used. Unfortunately, in the case of grades with close hardness and coercivity values, these works report very different WC grain sizes. Such discrepancies are particularly evident in the submicron grain size range. For example, in the case of grades containing 10 wt.% of Co and the average grain size $d_{\mathrm{WC}} = 0.5$ μm, the reported hardness ranges from 1540 to 1820 HV_{30}, for $d_{\mathrm{WC}} = 0.6$ μm, 1610 to 1798 HV_{30}, and for c $d_{\mathrm{WC}} = 0.7$ μm, 1431 to 1720 HV_{30}. The empirical relationships are also very different and therefore of little use.

Papers [11—13] consider the metrological aspects of WC grain size measurements. The results presented in [3, 6, 10] seem to be highly reliable. We believe that the empirical hardness vs. grain size and binder content relationships from [3, 6] can be used to find the relationships with other hardmetal properties.

The hardness vs. WC grain size relationships in [3] measured by the linear intercept (d_{c}) are Hall—Petch-type relationships for grades with 6 and 10 wt.% Co, respectively:

$$HV = 970 + 540 d_{\mathrm{c}}^{-1/2}, \qquad (1)$$

$$HV = 850 + 485 d_{\mathrm{c}}^{-1/2}. \qquad (2)$$

Kresse T. et al. [6] obtained an empirical equation for hardness, and tungsten carbide grain size (where d_{F} is the max Feret diameter) vs. cobalt volume content from the experimental results for WC—Co grades in a wide range of Co contents (5—25 wt.%):

$$HV = \alpha(V_{\mathrm{Co}})[729 + 718(d_{\mathrm{F}} + 0.13)^{-1/2}], \qquad (3)$$

wherein the first factor $\alpha(V_{\mathrm{Co}}) = 0.5/(V_{\mathrm{Co}} + 0.331)$ represents the contribution of the binder, and the second factor, that of the carbide component as a Hall—Petch relationship with the complex argument (d_{F} + 0.13). It limits the infinite growth of *HV* as d_{F} approaches zero. Comparing the estimations with equations (1)—(2) and (3) is of considerable interest, but first, we need to find the d_{c} vs. d_{F} relationship.

The study objectives are as follows:

— to analyze the WC—Co grades' hardness vs. composition and microstructure relationship;

— to use the measured HV and H_c values for a number of grades, and to identify the coercivity vs. composition and microstructure relationship;

— to prove the existence of a correlation between the HV, H_c, and d_{WC} values for the WC—Co hardmetal under certain conditions.

Test samples and methods

The samples consisted of submicron WC—Co tool hardmetal from Sandvik (Sweden), Konrad Friedrich and Konrad Micro Drill (Germany), Iscar (Israel), Gesac (China), and Virial (Russia). The carbon content is close to the stoichiometric value.

In order to evaluate Vickers hardness, the polished microsections were analyzed using a Falcon 508 hardness tester (Netherlands) at 294 N.

A Koerzimat 1.096 CS measuring system (Germany) was used to measure coercivity. We estimated the microstructural features using the Fiji software (USA) by analyzing the images obtained with a Mira 3 scanning electron microscope (SEM) (Tescan, Czech Republic). The images represented the surfaces of the samples etched and pre-polished with diamond suspensions. The etching process consisted of soaking the samples in the Murakami solution for 60s followed by rinsing and a 10-minute ultrasonic bath cleaning. Five SEM images were used to estimate the grain size. The FOV was 7.22 μm, with $80^{\times}$ magnification.

Hardness vs. WC grain size and Co binder content

The linear intercept (d_c) method assumes that the single WC grain size is the length of an arbitrary chord. Therefore, the grain size is the «average» chord length. The grade's average grain size estimated with the linear intercept method is double-averaged and does not significantly depend on WC grain shape. If we assume the equivalent circle diameter (d_{eqv}) to be the grain size, its average value does not depend on the WC grain shape, and the d_c to d_{eqv} ratio loosely depends on the grain morphology: $d_{eqv} \approx 1.15\, d_c$ [13].

When the max Feret diameter (the longest chord) is assumed to be the grain size, the average grain size (d_F) strongly depends on the grain isometricity. We can expect that in commercially available grades the degree of grain isometricity varies in a relatively narrow range. Therefore, in order to find the d_c/d_F ratio, we selected 6 grades from different manufacturers. Their average grain size dc was 0.39—0.68 μm. We used the samples to measure d_c and d_F. We obtained $d_c/d_F = 0.70 \pm 0.04$.

Since the variation did not exceed 10 %, we used the result to modify equation (3):

$$HV = [0.5/(V_{Co} + 0.331)] \times \times [729 + 601(d_c + 0.09)^{-1/2}], \quad (3a)$$

For grades with 6 and 10 wt.% Co respectively we obtain:

$$HV = 842 + 694(d_c + 0.09)^{-1/2}, \quad (4)$$

$$HV = 735 + 606(d_c + 0.09)^{-1/2}. \quad (5)$$

These equations are more suitable for comparison of the grade hardness vs. WC grain size relationships presented in [3] and [6].

Fig. 1 shows the hardness vs. grain size relationships for grades with 6 and 10 wt.% Co content obtained from equations (1), (4) and (2), (5), respectively. Our experimental data and the data from the studies [3, 6, 10] are also indicated.

Within the experimental data scattering for the 0.2—5.0 μm grain size range, the approximations presented in [3] and [6] give similar results. In the nanoscale range ($d_c < 0.2$ μm), the plastic deformation mechanisms change, and equations (1) and (2) are no longer valid. Therefore, it is preferable to use modified equation (3a). For example, in the case of grades with 10 wt.% Co and 0.14 μm [14] and 0.061 μm [15] grain sizes, equation (3a) gives 2000 and 2294 HV hardness values, respectively. This is in satisfactory agreement with the experimental values (2036 and 2356 HV).

The model by Kresse T. et al. [6], where the grade's hardness is determined by the hardness of its carbide network while the contribution of the Co content is expressed as a normalizing function. For $d_c > 0.2$ μm the model unifies the Hall—Petch factors for different Co concentrations:

$$HV = [0.5/(V_{Co} + 0.331)](850 + 485 d_c^{-1/2}). \quad (6)$$

For example, for 10 wt.% Co, $\alpha(V_{Co}) \approx 1$, we obtain equation (2). For 6 wt.% Co, $\alpha(V_{Co}) \approx 1.155$,

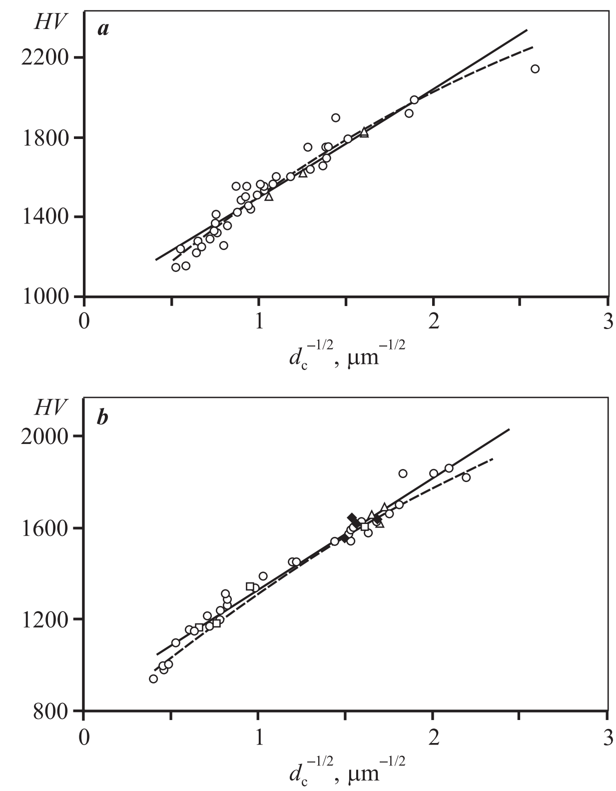

Fig. 1. Hardness of 6 wt.% Co grades (***a***), and 10 wt.% Co grades (***b***) as a function of WC grain size

Solid lines mean calculation as per Equation (1) (***a***) and Equation (2) (***b***) [3], dashed lines mean calculation as per Equation (4) (***a***) and Equation (5) (***b***);
symbols mean ○ – experimental data from [3];
△ – experimental data from [6]; □ – experimental data from [10];
◆ – Virial's own measurements

Рис. 1. Зависимость твердости сплавов с 6 мас.% (***a***) и 10 мас.% Co (***b***) от величины зерна WC

Сплошные линии – расчет на основании уравнений (1) (***a***) и (2) (***b***) [3], штриховые – расчет по уравнениям (4) (***a***) и (5) (***b***);
значки – экспериментальные данные [3] (○), [6] (△), [10] (□) и собственные измерения ООО «Вириал» (◆)

then $HV = 982 + 560d_c^{-1/2}$, which is almost identical to (1).

For $V_{Co} = 0$, equation (6) becomes:

$$HV_{WC} = 1284 + 733d_c^{-1/2}, \qquad (7)$$

where the factors are similar to the results presented in [16] (1382 and 731) and [17, 18] (1112 and 911). Equations (3a) and (6) enable comparison of the hardness of grades with different binder volume contents (at least for the $0.08 \le V_{Co} \le 0.24$ range), and to estimate the grain sizes.

The physical meaning of $\alpha(V_{Co})$ becomes clear, if we compare it to the carbide network contiguity (connectivity). C. Roebuck B. et al. [12] proposed the following equation for contiguity:

$$C = 1 - 1.27V_{Co}^{0.75}. \qquad (8)$$

Fig. 2 shows a comparison of $\alpha(V_{Co})$ and C values for WC—Co grades with the $V_{Co} \le 0.24$ cobalt volume content. It follows that:

$$\alpha(V_{Co}) \approx 1.5C. \qquad (9)$$

Then the expression for hardness can be expressed as

$$HV = C(1284 + 733d_c^{-1/2}). \qquad (10)$$

HV vs. tungsten carbide grain size and binder content in WC—Co grades is well described using the comprehensible phenomenological model. The grade's hardness depends on the hardness of its carbide network, while the volume content of the binder governs the contiguity of the network.

It should be noted that, as shown in [19], the WC—WC interfaces may or may not contain Co interlayers 3 to 30 nm thick, depending on the carbon content. It is not clear how the presence of such nanosheets affects the mechanical properties of the grade and whether we should adjust the concept of the carbide network and

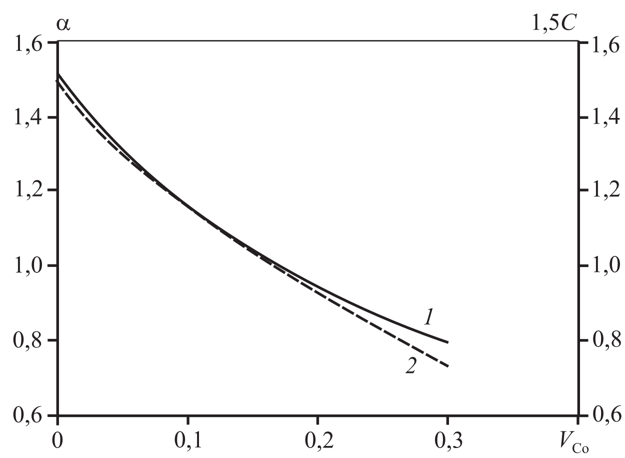

Fig. 2. Ratio between the coefficient $\alpha(V_{Co})$ (***1***) and contiguity C (***2***) depending on Co binder fraction

Рис. 2. Соотношение между коэффициентом $\alpha(V_{Co})$ (***1***) и смежностью C (***2***) в зависимости от содержания Co-связки its contiguity in grades with nearly stoichiometric carbon content.

Coercivity vs. WC grain size and Co binder content

In order to find the coercivity vs. WC grain size relationship for a number of tool hardmetals from different manufacturers (6, 10, and 12 wt.% Co content), we concurrently measured H_c and HV. Then we applied combined equation (6) to the measured HV values, in order to estimate the grain size (d_p). The solid lines in Fig. 3 show the curves for 6, 10, and 12 wt.% Co alloys. They are well fitted by the following empirical equations:

$$H_c = 54.7 + 102d_\mathrm{p}^{-1}, \quad (12)$$

$$H_c = 50.8 + 83d_\mathrm{p}^{-1}, \quad (13)$$

$$H_c = 53.0 + 76d_\mathrm{p}^{-1}. \quad (14)$$

Within the margin of error, we can assume the free terms in equations (12)—(14) are all equal to 53 Oe,

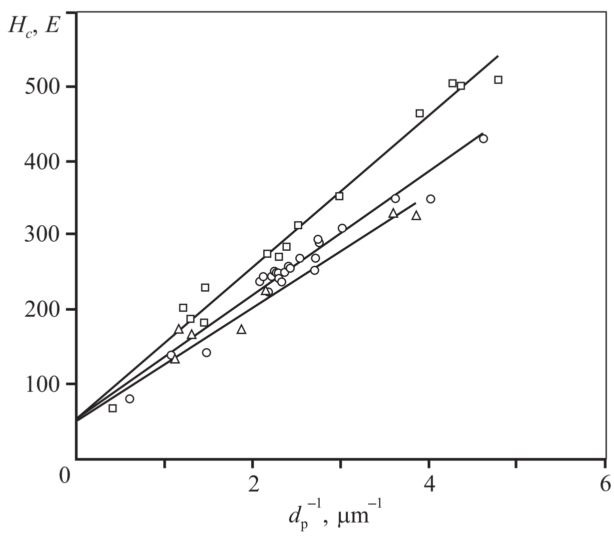

Fig. 3. Coercivity HC as a function of WC grain size in grades with different Co binder content:
□ – grades with 6 wt.% Co; ○ – grades with 10 wt.% Co; △ – grades with 12 wt.% Co

Рис. 3. Зависимость коэрцитивной силы от размера зерна WC в сплавах с различным содержанием Co-связки, мас.%: 6 (□), 10 (○) и 12 (△)

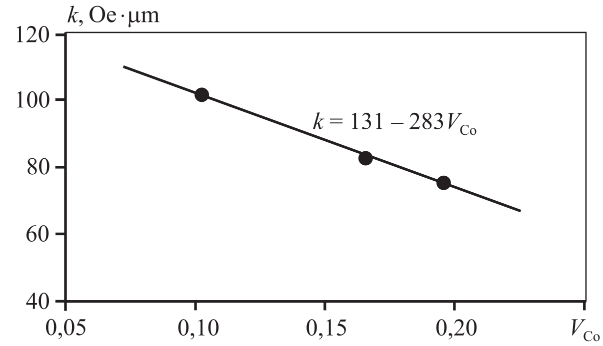

Fig. 4. Coefficient k as a function of cobalt volume fraction

Рис. 4. Зависимость коэффициента k от объемного содержания кобальта

while the k multiplier of d_p^{-1} linearly varies with the binder volume content (Fig. 4) within the 6—12 wt.% Co range. This relationship can be expressed as $k = 131 - 283V_\mathrm{Co}$.

By substituting the calculated d_p value with d_c in equations (12)—(14), we obtain the following expression for the H_c coercivity vs. grain size and binder content:

$$H_c = 53 + (131 - 283V_\mathrm{Co})d_\mathrm{c}^{-1}. \quad (15)$$

Since the empirical dependences (12)—(14) and, respectively, (15) were obtained with the grain size (d_p) values estimated by equation (6), they are valid for the 0.2—5.0 μm grain size.

Hardness vs. coercivity relationship

Fig. 5 shows our concurrent H_c and HV measurements for 6, 10, and 12 Co wt.% alloys. The solid lines indicate the estimated coercivity vs. hardness relationships obtained from equations (6) and (15). It follows from the figure that the relationships satisfactorily describe the experimental data. The data variation is due to certain variations of the hardmetal manufacturing process variables. The manufacturers usually specify [20—25] a fairly wide range of acceptable hardness and coercivity values: ±50 HV and ±35 Oe, respectively. Such a variation should be considered when analyzing the relationships for many different grades.

In hardmetal manufacturing, magnetic measurements are one of the first express NDT stages of sintered

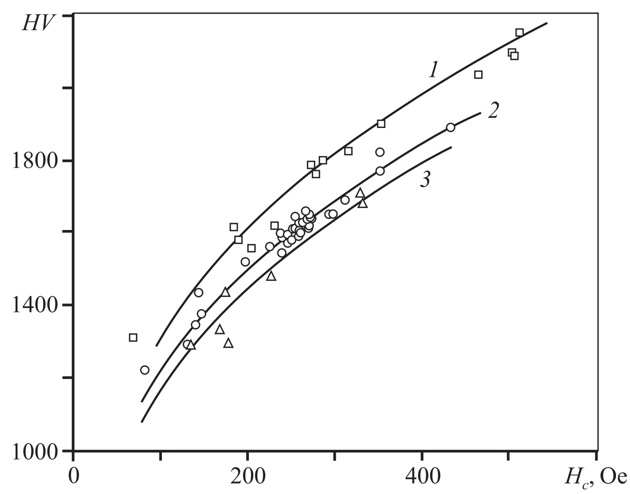

Fig. 5. Relationship between hardness and coercivity in the researched grades

Solid lines mean *HV* and H_c calculated as per Equation (6) and Equation (15), respectively, for grades with 6 wt.% Co (***1***); 10 wt.% Co (***2***); 12 wt.% Co (***3***); symbols mean experimental data: □ – grades with 6 wt.% Co; ○ – grades with 10 wt.% Co; △ – grades with 12 wt.% Co

Рис. 5. Соотношение между твердостью и коэрцитивной силой в исследованных сплавах

Сплошные линии – расчетные данные для *HV* и H_c сплавов с 6 (***1***), 10 (***2***) и 12 мас.% Co (***3***) по уравнениям (6) и (15) соответственно; значки – экспериментальные данные для сплавов с 6 Co (□), 10 Co (○) и 12 мас.% Co (△)

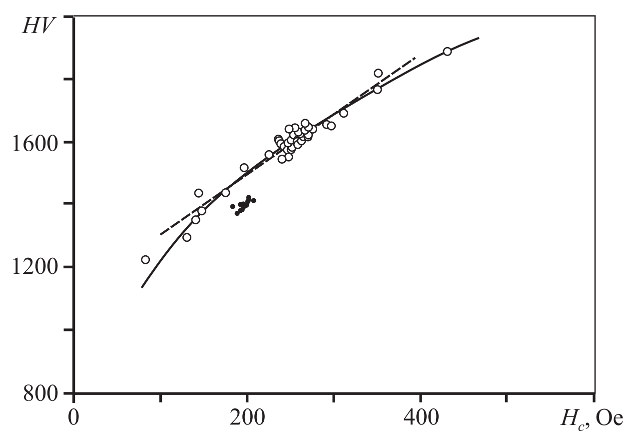

Fig. 6. $HV(H_c)$ ratio for grades with 10 wt.% Co (part of Fig. 5)

Solid line corresponds to theoretical curve *2* in Fig. 5, dashed line corresponds to linear approximation as per Equation (16); • – mean experimental data for VHS11 grade (Virial)

Рис. 6. Соотношение $HV(H_c)$ для сплавов с 10 мас.% Co (фрагмент рис. 5)

Сплошная линия соответствует расчетной кривой ***2*** на рис. 5, штриховая – линейная аппроксимация согласно уравнению (16); • – экспериментальные данные для сплава VHS11(ООО «Вириал»)

products. As Fig. 6 shows, for 10 wt.% Co grades, the following linear approximation

$$HV = 1{,}932H_c + 1112. \quad (16)$$

can be used as an express hardness assessment from coercivity (in the 110—350 Oe range).

It should be noted that the hardness and coercivity of WC—Co grades depend not only on the average grain size and Co binder content, but also on a number of other factors [3]. Therefore, we can expect some deviations from the proposed relationships for grades with wide or bimodal WC grain size distributions [26]. For example, the VHS11 grade, 10 wt.% Co (from Virial) was developed to offer both relatively high hardness (≈1400 HV) and fracture toughness (≥ 13.5 MPa·$m^{1/2}$). It has a wide WC grain size distribution. As shown in Fig. 6, it results in a noticeable change in the *HV* to H_c ratio compared to the grades with a narrower grain size distribution (CV less than 0.5).

Discussion

As we noted earlier, Roebuck B. et al. [3] provided the experimental approach to building the models by describing the hardness vs. WC grain size and binder content relationships. Kresse T. et al. [6] proposed a semi-empirical model in which grade's hardness is determined by the hardness of its carbide network normalized with a binder content factor. The carbide network hardness in their model is a Hall—Petch function of a complex argument. The max Feret diameter is assumed to be the WC grain size. It makes it impossible to directly compare the results since most publications refer to the average grain size determined by the linear intercept method.

We obtained the experimental hardness vs. average WC grain size relationships using the linear intercept method (d_c), the max Feret diameter (d_F), and modified equation (3) to (3a), in which dc is included. Such a modification makes it possible to compare a large number of published experimental datasets with the estimated values. The comparison showed that the hardness vs. average grain size and binder volume content relationship (3) [6] provides a good agreement with experimental data in a wide range of grain sizes: from the nanoscale to coarse.

In the case of $d_c > 0.2$ μm, we applied the $\alpha(V_{Co})$ normalization factor, in order to produce equation (6) which expresses the grade's hardness as a single Hall—Petch relationship for different Co binder contents. The $\alpha(V_{Co})$ to C ratio for hardmetals which we found makes the unified model clearer: The hardness of a grade with a nearly stoichiometric carbon content is a product of the carbide network hardness and contiguity.

In order to find the coercivity vs. grain size relationship, we used d_p estimated from the hardness values produced by equation (6). The *HV* variation of the same grain sizes can lead to a d_p estimation error. We evaluated the average deviation of the experimental hardness values from the trend curve presented in [3]. It does not exceed 30 HV, which corresponds to a less than 9 % uncertainty of the estimated grain size value. Since hardness values deviations are random, the estimated d_p errors are also random. This will cause more scattering relative to the trend curve, as we analyze the coercivity vs. grain size relationship. If the number of samples is sufficient, it does not affect the trend curve equation. Another confirmation of this is the good agreement between the experimental and estimated *HV* and H_c values as shown in Fig. 5.

Conclusions

1. We analyzed studies [3] and [6] and proposed a combined model to express *HV* as:

$$HV = [0{,}5/(V_{Co} + 0{,}331)](850 + 485d_c^{-1/2})$$

and to compare the harnesses of grades with the same grain sizes and different binder volume contents in the $0.08 \le V_{Co} \le 0.24$ range for $d_c > 0.2$ μm.

2. As a part of the model, we obtained a simple relationship between the grade's hardness, the carbide framework hardness (HV_{WC}), and contiguity (*C*):

$$HV = C \cdot HV_{WC},$$

$$HV_{WC} = 1284 + 733d_c^{-1/2}.$$

3. The coercivity vs. grain size and binder content relationship was found for grades with a 6—12 wt.% Co content:

$$H_c = 53 + (131 - 283V_{Co})\, d_c^{-1}.$$

4. We proposed an equation for the coercivity vs. hardness for grades with a 10 wt.% cobalt binder content:

$$HV = 1{,}932H_c + 1112.$$

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