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A New Method for Evaluating the Indentation Toughness of Hardmetals

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Abstract: This paper proposes a new method of evaluating the indentation toughness of hardmetals using the length of Palmqvist cracks (C) and Vickers indentation diagonal size (d_i). Indentation load "P" is divided into two parts: P_i for plastic indentation size and P_c for Palmqvist cracks. Pi depends upon the square of the indentation size (d_i^2) and Pc depends upon ($C^{3/2}$). The new method produces a very good linear relationship between the calculated indentation toughness values and the standard conventional linear elastic fracture mechanics toughness values with the same cemented carbide materials for a large number of standard Kennametal grades for both straight WC-Co carbide grades and grades containing cubic carbides. The new method also works on WC-Co hardmetal data selected from recently published literature. The technique compares the indentation toughness values of WC-Co materials before and after vacuum annealing at high temperature. The indentation toughness values of annealed carbide samples were lower than for un-annealed WC-Co hardmetals.

Keywords: WC-CO cemented carbide materials; Vickers hardness; Palmqvist indentation cracks; indentation toughness; linear elastic fracture mechanics toughness, K_{IC}, G_{IC}

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1. Introduction

WC-Co based cemented carbide materials, also known as hardmetals, with and without the addition of cubic carbides such as TiC, TaC, and NbC to the base material, are extensively used in metalcutting, mining, metalforming, and other speciality wear-resistant applications. Many hardmetal components rely on material hardness, and while a number of application-relevant properties, such as strength, elastic modulus, and hardness are easy to measure, the conventional linear elastic fracture mechanics approach for measuring the fracture toughness, critical energy release rate (G_{IC}), and critical stress intensity factor (K_{IC}) requires considerable effort. Specifically, the pre-cracking of specimens has remained a serious obstacle.

The Palmqvist indentation cracking test is sometimes used for the characterization of the toughness of cemented carbides [1]. The test provides a measure of the indentation crack resistance of a brittle material from the length of cracks induced with a Vickers diamond hardness impression and applied load as per Equation (1)

$$W = P/C \quad (1)$$

where W is the Palmqvist indentation toughness, P is the indentation load on a Vickers diamond indenter, and C is the sum of the four Palmqvist cracks lengths, $(C_1 + C_3) + (C_2 + C_4)$ emanating from the four corners of the indentation after the load has been removed. Crack length $C_1 + C_3$ is measured along one indentation diagonal length and $C_2 + C_4$ is measured along the other indentation diagonal length [2]. It is to be noted that W has the unit of kg/mm, similar to G_{IC} in linear elastic fracture mechanics formulation.

Palmqvist cracks geometrically different from half-penny cracks are essentially confined to the specimen surface and therefore surface preparation is extremely important and critical for the evaluation of indentation toughness. Exner [2] further examined the issue of specimen surface preparation techniques such as diamond polishing of the ground specimen so that the deformed binder phase layer near the surface and surface residual compressive stress observed in the WC phase are minimized, and further recommended a high-temperature (1000–1100 °C) vacuum annealing procedure after diamond-polishing procedures so that reproducible Palmqvist cracks are generated at each indentation load.

2. Indentation Toughness versus Linear Elastic Fracture Mechanics Toughness

In recent years, considerable efforts have been directed at relating indentation toughness W or equivalent K values with conventional linear elastic fracture mechanics (K_{IC}) or equivalent G_{IC} values for the same cemented carbide materials. Niihara [3] and Warren and Matzke [4] independently suggested the relationship in Equation (2)

$$K_{IC} = b(H \cdot W)^{1/2} \quad (2)$$

The above relationship is based upon the formation of half-penny cracks which have not been observed in cemented carbide materials. In the above equation, "b" is a non-dimensional constant dependent on the ratio of Young's modulus"E" and Vickers hardness "H" in Niihara's analysis. The value of constant in Warren and Matzke's analysis is unspecified. These investigators collected a large body of experimental data on WC-Co hardmetals and showed good linear correspondence with Equation (2) for K_{IC} values up to ~17 MPa·m$^{1/2}$. The latest model is that of Shetty and colleagues [5], who used a wedge loaded crack as a fracture mechanical analogue to the situation in Palmqvist cracks and showed that K_{IC} can be evaluated as Equation (3)

$$K_{IC} = 0.0889(H \cdot W)^{1/2} \quad (3)$$

Shetty's model has become an accepted model for evaluating the indentation toughness of hardmetals and is being used extensively by the carbide industry for that purpose [6] The indentation toughness values have a good linear relationship with K_{IC} values determined by the conventional linear elastic fracture mechanics procedures for values up to ~20 MPa·m$^{1/2}$ but the linear relationship breaks down for carbide materials with very high toughness values. The reason for this discrepancy is that Palmqvist cracks are extremely small compared with indentation diagonal size, so that ratio of $C/2d_i$ is extremely small, at much less than 1. In that case indentation toughness values are very large compared with K_{IC} values. This paper proposes a new method to address this problem.

3. The New Approach for Evaluating the Indentation Toughness

Two effects are observed whenever a flat and properly polished specimen of a cemented carbide material is indented with a Vickers indenter with load "P". One can observe Vickers plastic indentation with size "d_1" and "d_2" along with Palmqvist cracks emanating from the four corners of the Vickers indentation. The size of the average indentation diagonal $d_i = (d_1 + d_2)/2$ and lengths of cracks depend upon the mechanical properties (plastic deformation and toughness properties of a given carbide material which in turn depend upon the chemical composition of WC-Co, WC grain size, and the average thickness of the binder phase). Sometimes the indentation load has to be sufficiently large to induce Palmqvist cracks on all four corners of the Vickers indentation in very-high toughness cemented carbide materials.

The technical approach adopted here is as follows:

One can divide indentation load "P" into two components, P_i and P_c. Pi is responsible for causing average indentation "d_i" and P_c for causing Palmqvist cracks $C = C_1 + C_2 + C_3 + C_4$. One can write the Equation (4) as

$$P = P_i + P_c \quad (4)$$

It is well-known that P_i is proportional to the square of the average indentation "d_i". Therefore, Equation (5) can be written as

$$P_i = X_i \cdot d_i^2 \quad (5)$$

Also, P_c is proportional to $C^{3/2}$ and therefore Equation (6) is as follows:

$$P_c = X_c \cdot C^{3/2} \quad (6)$$

Therefore, one can write the two equations into Equation (7)

$$P = X_i \cdot d_i^2 + X_c \cdot C^{3/2} \quad (7)$$

Now, if the indentation load is in "kg", the indentation size is in mm and C is in mm. Then, "X_i" is described in kg/mm^2) and "X_c" is in $kg/mm^{3/2}$. Assuming that $X_i = 1$; and $X_c = 1$, this leads to Equation (8)

$$P = d_i^2 + C^{3/2} \quad (8)$$

One can combine these equations and arrive at Equations (9) and (10)

$$K_m = P_c / C^{3/2} \quad (9)$$

$$W_m = P_c / C \quad (10)$$

Therefore, one can calculate "K_m" and "W_m" by using the P_c and C from the measured values of indentation size and total lengths of Palmqvist cracks. It should be understood that K_m and W_m are different from conventional indentation toughness "W", as is mentioned in Equation (1).

4. Results and Discussions

The results are presented in three sections as follows.

4.1. Application to Kennametal Cemented Carbide Grades

Detailed investigations [7] were undertaken in early 1980s on the Palmqvist toughness and the linear elastic fracture mechanics toughness (K_{IC}) of a large number of commercially available Kennametal carbide grades covering metal cutting, mining, metal forming, and specialty grades. Metalcutting grades contained fair amounts of cubic carbides such as TiC, NbC etc., whereas others were essentially straight WC-Co grades with less than 0.5% cubic carbides. The properly polished samples were indented at various indentation loads varying from 30 to 120 kg. Three measurements were conducted at each load for indentation size and Palmqvist crack measurements. Considerable variation in Palmqvist crack lengths was noted even within a single indentation from one corner to the opposite corner. Linear elastic fracture mechanics measurements (K_{IC}) were also conducted from the same batch of carbide samples using the Terra Tek procedure [8]. Indentation toughness "K_m" was calculated at 100 kg indentation load and compared with the average value of K_{IC}. Figure 1 shows the K_m versus K_{IC}. The linear agreement between K_m and K_{IC} is quite reasonable across the whole range of carbide materials.

4.2. Application to Recently Published Crack Length and Vickers Hardness Data

Recently, Seikh and colleagues published a paper [9] measuring the indentation toughness and K_{IC} values on a large number of straight WC-Co cemented carbide samples using an indentation load of 30 kg for both Vickers hardness and Palmqvist crack measurements. Ten measurements were performed for each WC-Co material for a total of eight different carbide materials. K_{IC} measurements were also conducted for all of the eight carbide materials. The sum of Palmqvist crack lengths "C" was calculated from the given data and indentation toughness (K_m) was calculated for each carbide material. Figure 2 shows the plot of indentation toughness "K_m" versus "K_{IC}" for all of the samples. The linear agreement between K_m versus K_{IC} is excellent across the whole range of carbide materials. This shows the clear difference indentation toughness between the method adopted in this approach versus the previous methods, as detailed in Section 2 of this paper.

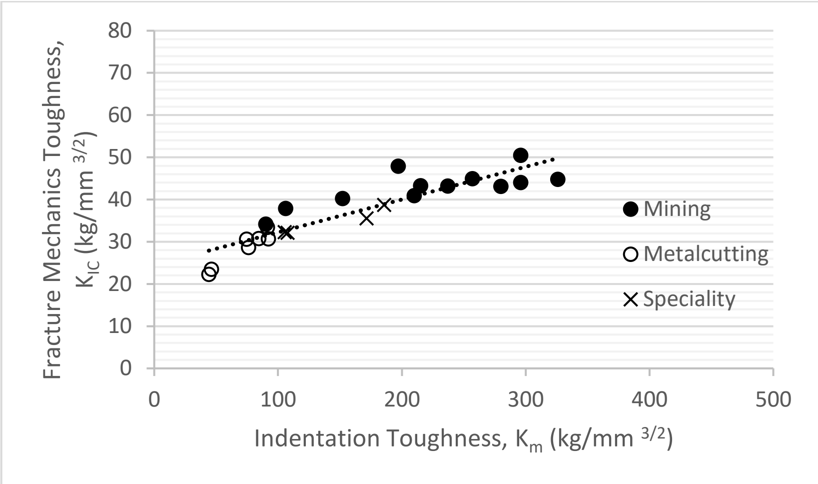

Figure 1. Indentation toughness (K_m) versus fracture mechanics toughness (K_{IC}) for Kennametal grades.

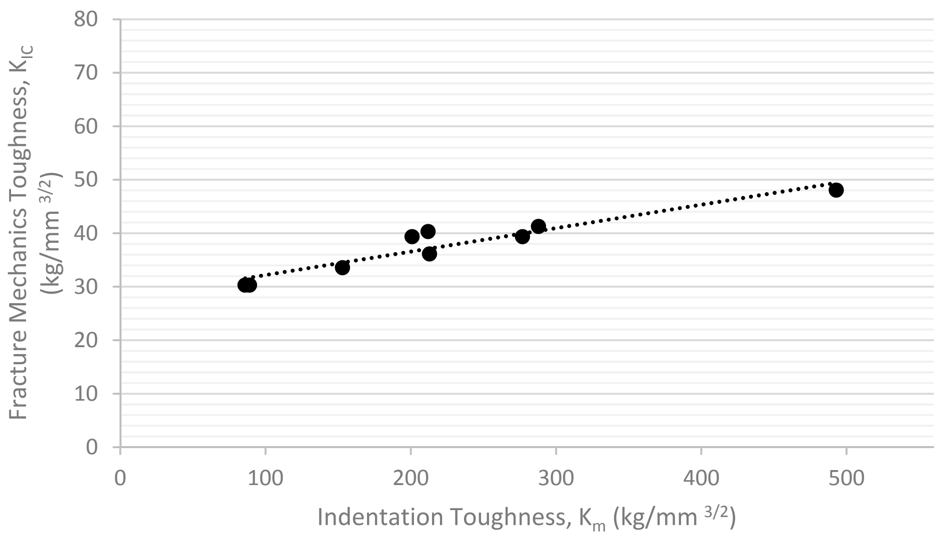

Figure 2. Indentation toughness (K_m) versus fracture mechanics toughness (K_{IC}), for WC-Co hardmetals.

4.3. Effect of Vacuum Annealing on Indentation Toughness of Carbide Materials

Exner et al. [10] conducted Palmqvist crack measurements on a number of straight WC-Co carbide grades, which were vacuum annealed at 1100 °C before Palmqvist crack lengths were carried out at indentation loads of 30, 45, 60, 100, and 150 kg. It was not possible to compare indentation toughness before and after vacuum annealing in that work because no crack measurements were conducted on the as-sintered un-annealed samples. However, it was possible to compare the results with the published data of Seikh and colleagues [9], who performed extensive Palmqvist crack measurements at an indentation load of 30 kg. Therefore, indentation toughness was calculated on a few vacuum annealed WC-Co samples at an indentation load of 30 kg and the indentation toughness results were compared with the indentation toughness data taken from the work of Seikh and colleagues [9]. The results are summarized in Tables 1 and 2.

Table 1. Data from Exner et al. [10] on Co Vol %; Vickers hardness and toughness.

SP# (Co Vol %)	Vickers Hardness	K_m
5.1	1705	27.2
10.1	1603	30.8
14.8	1390	38.1

Table 2. Data from Seikh et al. [9] on Co Vol %; Vickers hardness and toughness.

SP# (Co Vol %)	Vickers Hardness	K_m
4.2	1782	30.5
7.5	1748	29.8
10	1591	39.0
15.6	1483	39.9

One can note that the K_m value is higher for the as-sintered WC-Co materials (samples from Seikh et al. [9]) as compared with the vacuum-annealed carbide materials (samples from Exner et al. [10]) for essentially similar WC-Co compositions, in spite of the fact that un-annealed specimens have higher Vickers hardness values. In general, toughness is inversely proportional to Vickers hardness for these hardmetal materials. This result indicates that vacuum annealing reduces the indentation toughness of WC-Co carbide materials.

This result is completely unexpected and contradicts the results of various investigators [10,11] who compared vacuum annealed indentation toughness values with K_{IC} and G_{IC} values, which were generally measured on un-annealed as-sintered carbide samples assuming explicitly that vacuum annealing of WC-Co material should not reduce or degrade any mechanical properties of the as-sintered carbide materials. This is probably based on the fact that Vickers hardness does not change after annealing. To the best of our knowledge, uniaxial yield stress and K_{IC} measurements have not been conducted on high-temperature vacuum-annealed WC-Co materials and reported in the open published literature.

The work of Pickens and Gurland [12] is worth mentioning to explore this issue further. These authors evaluated the K_{IC} and G_{IC} of a large number of WC-Co materials with varying volume fraction of cobalt, WC grain sizes, and cobalt-based binder phase layer thickness, and proposed Equation (11) to explain the results:

$$G_{IC} = a \cdot \sigma_y \cdot l \quad (11)$$

where "a" is a constant, σ_y is the in-situ yield stress of the binder phase, and "l" is the average thickness of the binder phase.

Vacuum annealing at (1000–1100 °C) is not expected to change the value of binder phase thickness. Also, it has been observed during routine X-ray diffraction of the polished carbide samples that the major cobalt-based binder phase XRD peak becomes sharper and of higher intensity for the annealed sample than that of the un-annealed as-sintered polished sample, which is broad and of low intensity. This observation indicates that in situ yield stress of the binder phase (σ_y) has decreased, resulting in a lower G_{IC} value after annealing. This result is consistent with the lower indentation toughness of annealed samples compared with un-annealed samples as shown in our results.

This result is also consistent with lower transverse rupture strength of CVD-coated carbide samples routinely observed in CVD-coated samples as compared with uncoated polished samples.

It has also been well established that carbide materials coated with CVD coatings (multi-layer TiCN/TiC/Al_2O_3) have performed poorly in metal cutting machining operations, especially for rotating tools (interrupted cutting operations such as milling applications) relative to high quality ion-plated PVD TiN, TiCN, and TiAlN coatings, even though CVD coatings have higher abrasive wear resistance (hot hardness) and also higher crater wear resistance (chemical inertness) than PVD TiN, TiCN, and TiAlN coatings. The primary reason is that CVD coatings routinely deposited at high temperatures (~1050–1250 °C) reduce the toughness of the base carbide materials. PVD coatings are generally deposited at around ~500 °C and do not degrade the transverse rupture strength of the base material.

5. Conclusions

1. A new method of evaluating the indentation toughness of hardmetals has been proposed.
2. The new measured indentation toughness values provide very good linear agreement with K_{IC} values measured by conventional linear elastic fracture mechanics procedures.
3. Vacuum annealing of as-sintered cemented carbide materials at 1000–1100 °C lowers the indentation toughness of cemented carbide materials.

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