

The relationship between Palmqvist indentation toughness and bulk fracture toughness for some WC–Co cemented carbides

C. T. PETERS

Boart Research Laboratory, Krugersdorp 1740, South Africa

The Palmqvist indentation toughness (W) has been determined for a series of WC–Co hardmetals containing up to 10 wt % cobalt. Using a correlative parameter (the cobalt mean-free path) the Palmqvist toughness has been compared with true fracture toughness data (G_{IC} values) for similar materials, obtained by conventional testing techniques. It has been shown that a linear relationship between W and G_{IC} can be predicted, on the basis of a recent theory of the fracture process in hardmetals. The validity of such a relationship has been experimentally confirmed, over a limited range of hardmetal compositions and microstructures. The experimental difficulties and expense associated with fracture toughness determination for brittle materials such as hardmetals, by quasi-conventional techniques, are well known. It is concluded that the Palmqvist test provides a useful method of measuring fracture toughness on a routine basis, since it is simple to perform, non-destructive, and does not require a specialized testpiece geometry. Its application in hardmetal quality control is, therefore, indicated.

1. Introduction

The Palmqvist test [1] provides a measure of the resistance of brittle materials to crack propagation from the corners of a Vickers diamond indentation made on a polished surface. The Palmqvist toughness (W) is defined as the indenting load (P) divided by the sum of the four crack lengths measured at the indentation corners (L):

$$W = P/L. \quad (1)$$

Various attempts have been made (e.g. [2–4]) to utilize W values in comparative studies of the mechanical behaviour of different grades of cemented carbides. These were generally inconclusive for two main reasons, namely

(a) the difficulty of obtaining reproducible crack length data, and

(b) the absence of theoretical correlations between the Palmqvist toughness and conventional mechanical parameters.

Recent research by Viswanadham and Venables [5], Perrott [6] and Exner *et al.* [7] has attempted

to establish a relationship between W and G_{IC} for hardmetals, based on fracture mechanics theories. Viswanadham and Venables found that a direct proportionality was indicated by their results, whilst the other authors concluded that a simple relationship did not adequately describe their data.

In the present work, the Palmqvist toughness has been measured for a series of WC–Co hardmetals spanning a range of cobalt contents up to 10 wt %. These values have been compared with fracture toughness (G_{IC}) data recently published by Murray [8] and quantitatively correlated by means of a microstructural parameter, the cobalt mean-free-path (λ).

2. Experimental materials and methods

2.1. Hardmetal compositions

A series of eight WC–Co hardmetals was selected for investigation, spanning a wide range of microstructures. Four alloys (A to D) were of sub-stoichiometric composition, and contained varying

amounts of the eta phase double-carbide $\text{Co}_3\text{W}_3\text{C}$. Details of these materials have been published recently in a separate article [9]. The remainder of the materials were industrial stoichiometric grades, comprising a 5% cobalt fine-grained alloy (5F), a 5% cobalt medium-grained alloy (5M), an 8% cobalt coarse-grained alloy (8C) and a 10% cobalt coarse-grained alloy (10C).

2.2. Evaluation of structural parameters

The mean WC grain size (\bar{d}) of each alloy was determined by metallography, using the mean linear intercept method. Owing to the wide grain-size distribution of the coarse structures, a small range of \bar{d} has been reported, based on statistical analysis of the linear intercept data.

The cobalt volume fraction (f_{Co}) was calculated from the observed saturation magnetization, in the case of the stoichiometric alloys. For the substoichiometric materials, f_{Co} values were obtained using ancillary measurements of the volume fraction of eta phase in the microstructure [9].

Knowing the free cobalt content, and the mean grain size, the binder mean-free-path (λ) was calculated using the relationship:

$$\lambda = f_{\text{Co}} \cdot \bar{d} / (1 - f_{\text{Co}}). \quad (2)$$

2.3. Palmqvist technique

It is of great importance to ensure that the Palmqvist indentations are made on a polished surface which is free from residual stresses introduced by the polishing procedure. This can be achieved by annealing after rapid polishing or by extended polishing with successively finer abrasives, until a maximum indentation crack length is observed [3]. In this work, the latter

method was adopted and the optimum polishing procedure was found to be as follows:

- (1) preliminary grinding using a diamond impregnated grinding wheel;
- (2) 30 min polishing with 20 to 40 μm diamond powder;
- (3) 30 min polishing with 8 to 16 μm diamond powder;
- (4) 20 min polishing with 3 to 6 μm diamond powder;
- (5) 20 min polishing with 1 μm diamond powder;
- (6) 20 min polishing with 0.25 μm diamond powder.

After polishing, ten Vickers diamond indentations were made on each sample, using an indenting load of either 300 or 1177 N. The total length of cracks for each indentation was then measured at a magnification of $\times 100$. The mean crack lengths and their coefficients of variation were then evaluated for each alloy.

Examples of typical Palmqvist cracks for the 5M and 10C alloys are shown in Fig. 1a and b, respectively.

3. Experimental results

The calculated values of cobalt binder content (f_{Co}), WC mean grain size (\bar{d}) and binder mean-free-path (λ) for each alloy are given in Table I. The mean Palmqvist crack lengths (\bar{L}) and their coefficients of variation are given in Table II, together with the corresponding values of Palmqvist toughness (W) calculated from Equation 1.

4. Discussion

The Palmqvist cracks do not always appear to originate from the indentation apex. This phenomenon has been noticed in previous work (see, for

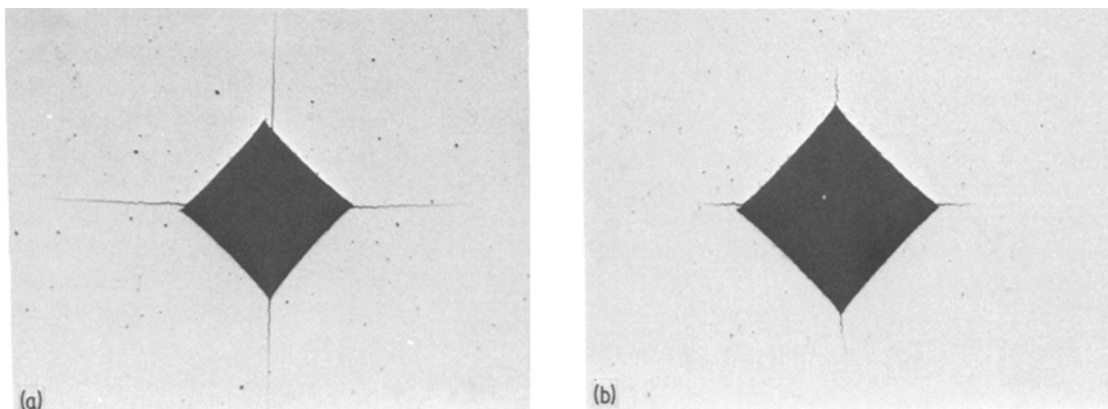


Figure 1 Palmqvist cracks in (a) 5 M hardmetal ($\times 60$); (b) 10 C hardmetal ($\times 60$).

TABLE I Microstructural parameters of the experimental hardmetals

Grade	f_{Co}	\bar{d} (μm)	λ (μm)
A	0.03	1.3	0.040
B	0.043	1.3	0.057
C	0.05	1.3	0.067
D	0.06	1.3	0.082
5F	0.085	1.3	0.122
5M	0.085	1.5–2.0	0.14–0.19
8C	0.135	3.0–4.0	0.47–0.63
10C	0.17	3.0–4.0	0.61–0.82

example, Fig. 1 of Viswanadham and Venables [5] and Fig. 4b of Ogilvy *et al.* [10]) but no explanation has been proposed. In view of the exceptional attention paid to sample preparation in all indentation fracture research, it is concluded that sample misalignment, relative to the diamond indenter, is not the factor responsible for the observed asymmetry of cracks. A possible explanation is as follows.

It is known that Palmqvist cracks in the present grades of hardmetal are initiated at indenting loads significantly below those used in the experimental work. The observed position of the Palmqvist cracks after testing hence depends on the mode of propagation of the initial crack, under the increasing indenting load. The crack path may deviate somewhat from the direction of the indentation diagonal. Since the initial crack zone is consumed within the expanding indentation, the final crack will then appear to have originated from an indentation edge, as observed in Fig. 1. The extent to which this occurs may be expected to increase as the difference between the crack initiation load and the final indenting load increases. The brittle 5M hardmetal should, therefore, show a larger shift in apparent crack origin than the tougher 10C hardmetal; this agrees with

TABLE II Experimental Palmqvist test data

Grade	Crack length data		Palmqvist toughness W (kN m^{-1})
	\bar{L} (μm)	C.V. (%)	
A*	725	7	406
B*	660	7	446
C*	640	3	460
D*	580	6	507
5F*	460	5	640
5M	1153	3	1021
8C	433	8.5	2718
10C	338	8	3482

*Indented at 300 N

the experimental observations, as exemplified by Fig. 1a and b.

The coefficients of variation of the crack length data ranged from 3.5% to 8.5%, which represents a reasonably good level of reproducibility among the individual results for each alloy. Experimental scatter of data is, therefore, not a problem, when the polishing procedure is carefully optimized.

From the Palmqvist toughness values given in Table II it can be seen that this test provides an intuitively correct ranking of the hardmetal grades in terms of toughness, which is expected to increase with increasing cobalt content and grain size.

It is of obvious interest to find a meaningful quantitative correlation between the Palmqvist toughness (W) and the true fracture toughness (G_{IC}). This has been attempted by analysing the present experimental data in terms of a recent theory of hardmetal fracture, as given below.

In a recently developed theory of crack propagation in WC–Co hardmetals, Murray [8] has shown that G_{IC} should vary linearly with the cobalt mean-free-path (λ). Using conventional fracture toughness data from various sources, he demonstrated that a linear relationship was closely obeyed. Thus we may write:

$$G_{IC} = \beta + \gamma\lambda \quad (\beta, \gamma = \text{constants}). \quad (3)$$

The most simple relationship between Palmqvist toughness and G_{IC} , is direct proportionality between the two quantities, i.e.

$$W = \alpha G_{IC}. \quad (4)$$

By substitution of Equation 3 into Equation 4 we obtain:

$$W = \alpha\beta + \alpha\gamma\lambda. \quad (5)$$

Therefore, if Equation 4 is valid, a linear variation of Palmqvist toughness (W) with binder mean-free-path is predicted by Equation 5. The experimentally observed variation of W with λ is shown in Fig. 2; a good linear correlation is apparent. The correlation coefficient of this line was 0.995.

In order to establish fully the validity of Equation 4, the two straight lines defined by Equations 3 and 5 must now be numerically compared. Inspection of the latter relationships shows that the ratio of their slopes ($\alpha\gamma/\gamma$) and of their intercepts ($\alpha\beta/\beta$) should be identical. The actual experimental data did not exactly fulfil this

requirement; agreement to within about 7% was obtained, however, on the basis of Murray's stated experimental accuracies. When our own experimental errors in W values (arising from crack length measurements) were also taken into account, a range of coincidence of slope and intercept ratios was achieved. This shows that the two sets of experimental data can be manipulated to verify Equation 4, although the value of α cannot be defined exactly from the data available. The approximate solution is given by:

$$\text{Palmqvist toughness } (W) = 4.9 (\pm 0.2) \times 10^3 G_{IC} \quad (6)$$

In order to achieve the above correspondence between Murray's data and the present experimental results, it was necessary to utilize data from the extremes of the error bands. This may indicate that the simple proportionality $W = \alpha G_{IC}$ is not the optimum correlation between these quantities, despite its intrinsic attractiveness. The next simplest correlative description (taking into account the linear variation observed between W and λ) has the form:

$$W = \delta + \epsilon G_{IC} \quad (\delta, \epsilon \text{ constants}) \quad (7)$$

Although empirical, the above relationship provides a very accurate fit with experimental

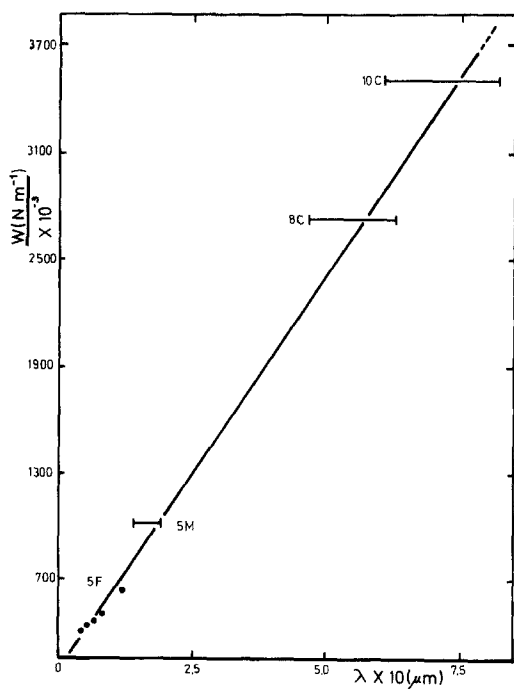


Figure 2 Experimentally observed variation of Palmqvist toughness (W) with binder mean-free-path (λ).

data. The values of δ and ϵ can be obtained from the best-fitting lines given by Equations 3 and 5, yielding the following relationship:

$$W = 5.74 \times 10^3 G_{IC} - 8.63 \times 10^4 \quad (8)$$

The G_{IC} values for the experimental alloys have been calculated using Equations 6 and 8 and are given in Table III. If the elastic constants (E, ν) of the materials are known, the more practical toughness parameter, K_{IC} , can be calculated, using the relationship:

$$K_{IC}^2 = EG_{IC}/(1 - \nu^2) \quad (9)$$

Perrott [6] and Exner *et al.* [7] have independently established that linear relationships such as those given by Equations 6 and 8 do not adequately describe the dependence of W on G_{IC} over a wide range of cobalt contents. A marked divergence from linearity is observed when tougher grades of hardmetal are evaluated. This has been attributed to changes in the relative amounts of crack growth occurring during loading and unloading, respectively, of the indenter.

In the present work, we have noticed that deviation from Equation 8 commenced at a cobalt content of 11 wt%. In this material, it was observed that indentation generated cracks which were markedly different from those shown in Fig. 1. A large number of short cracks were observed at the corners of the indentation; these exhibited a large degree of transgranular cleavage of the WC grains, in contrast to the intergranular cracking normally observed in the more brittle grades. In many cases, WC cleavage had propagated through several adjacent WC grains without visible signs of fracture in the intervening cobalt interlayers.

We have, therefore, attributed the deviation from linearity in tough grades to a change in the fracture mechanism, at high values of cobalt

TABLE III Calculated fracture toughness values for experimental hardmetals

Grade	Fracture toughness G_{IC} ($J m^{-2}$)	
	From Equation 6	From Equation 8
A	83	86
B	91	93
C	94	95
D	103	103
5F	131	127
5M	208	193
8C	555	489
10C	711	622

mean-free-path. In the low cobalt alloys fracture occurs predominantly by rupture of the Co/WC interlayers, whilst at higher binder contents, WC cleavage takes place, presumably via the dislocation pile-up stress generated by plastic deformation in the binder [11, 12].

The results of both Perrott and Exner *et al.* indicate that a linear correlation between W and G_{IC} is satisfactory over the present experimental range of G_{IC} values, and show a reasonable quantitative agreement with Equation 6. The range of materials over which the linear correlation is valid includes a large number of materials of industrial importance. It is concluded, therefore, that whilst the Palmqvist test does not have universal validity as a fracture toughness test, it provides a simple, inexpensive and non-destructive method of evaluating and comparing commercial WC-Co hardmetal grades with cobalt contents up to 10 wt %.

Further work is envisaged to investigate the means by which the lengthy specimen preparation procedure may be shortened, so that the Palmqvist technique can be made more attractive for routine quality control. Routine metallographic preparation, followed by a short annealing treatment may possibly offer an adequate alternative to the extended polishing sequence, for reducing residual surface stress to a minimum.

4. Conclusions

(1) Satisfactory reproducibility of Palmqvist crack length data can be achieved by close control of the surface preparation technique.

(2) Over a limited range of WC-Co hardmetal compositions (up to 10 wt % Co), the Palmqvist

toughness (W) is linearly related to the bulk fracture toughness G_{IC} , by the relationship:

$$W = 5.74 \times 10^3 G_{IC} - 8.63 \times 10^4.$$

(3) For the above range of materials, the Palmqvist test may be used routinely to evaluate fracture toughness, on specimens from normal production batches, without the need for sophisticated testing apparatus.

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Received 8 August and accepted 16 November 1978.