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Fracture toughness testing of hardmetals using compression–compression precracking

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Compression–compression precracking of brittle materials has recently been applied to fracture toughness testing. This paper reports the results of an experimental programme of fracture toughness testing of a WC–Co alloy containing 10% cobalt by weight. Tests were performed on specimens precracked by cyclic compression and on specimens in which this compression–compression precrack was subsequently extended by tensile fatigue. Toughness data obtained in this way were compared with the results of short-rod toughness tests. The causes of differences between these various data are discussed.

1. Introduction
Hardmetals are used primarily for their high hardness and good wear resistance, but also require adequate toughness to avoid failure by chipping or cracking. The quantification of hardmetal toughness has therefore received considerable attention over the last two decades. Earlier techniques such as the transverse rupture strength (TRS) test [1–3], impact chipping [1] and the Palmqvist test [1, 3, 4] have suffered from sensitivity to surface preparation and finish, and lack of reproducibility of results, as well as from the fact that they may not provide the required material constant parameter quantifying crack growth resistance. In this respect, however, it should be noted that there have been recent attempts to correlate Palmqvist values with fracture energy [3–5].

The application of linear elastic fracture mechanics to toughness determination, through use of the plane strain fracture toughness (K_{Ic}), has afforded a considerable improvement in reproducibility and provides a material constant parameter which is independent of specimen geometry. An essential requirement in obtaining valid K_{Ic} values is the generation of a sharp precrack of regular shape, prior to performing the actual toughness test. Current standards for toughness testing, e.g. BS 5447 [6], specify tensile fatigue to generate this precrack from a notch, but for brittle materials the stress intensity required to initiate a crack from a blunt notch is close to the fracture toughness and control of crack growth is difficult.

Consequently, numerous alternative methods of precracking hardmetal and ceramic toughness specimens have been proposed. Currently used techniques include indentation, either by a wedge [7] or a pyramid [8], stiff tensile loading (displacement control) of notched specimens [9], impact loading of notched specimens with a zone of lateral compression below the notch [8] and V-notched short rod specimens [10]. A very useful review of precracking and fracture toughness test methods has been given by Bolton and Keeley [11].

In determining the toughness of hardmetals there are two major requirements of test methods:

1. that they should be relatively simple and utilize relatively inexpensive equipment so that they are suitable for use in routine assessment of batches of hardmetal (and give as little scatter as possible);
2. that they should allow accurate, standard comparison both among different hardmetal grades and with other materials.

These are difficult to meet simultaneously with a particular test method. The first requirement is met largely by techniques like the short-rod or Fractometer test, although it has been suggested that data obtained with this method may be influenced by artefacts of specimen preparation, at least for some materials [12]. To meet the second requirement, it would be advantageous if tests could be performed that meet all the conditions found in accepted standards for toughness testing, such as BS 5447 [6]. The development and application of compression–compression precracking to brittle materials appears to offer this possibility.

2. Compression–compression fatigue
Observations of controlled crack initiation and growth from notches in metallic specimens subject to cyclic compression date back to the 1960s [13]. Recently, Suresh and co-workers have used the technique with ceramic and hardmetal specimens and have proposed mechanisms to explain the phenomenological observations of crack growth [14–17]. In ceramic–metal composites fatigue crack growth under cyclic compression has been proposed to occur by an interaction between the mechanisms observed in metals and ceramics [16]. In essence, these mechanisms involve:

(a) generation of residual tensile stresses in the cobalt binder as a consequence of cyclic plasticity;

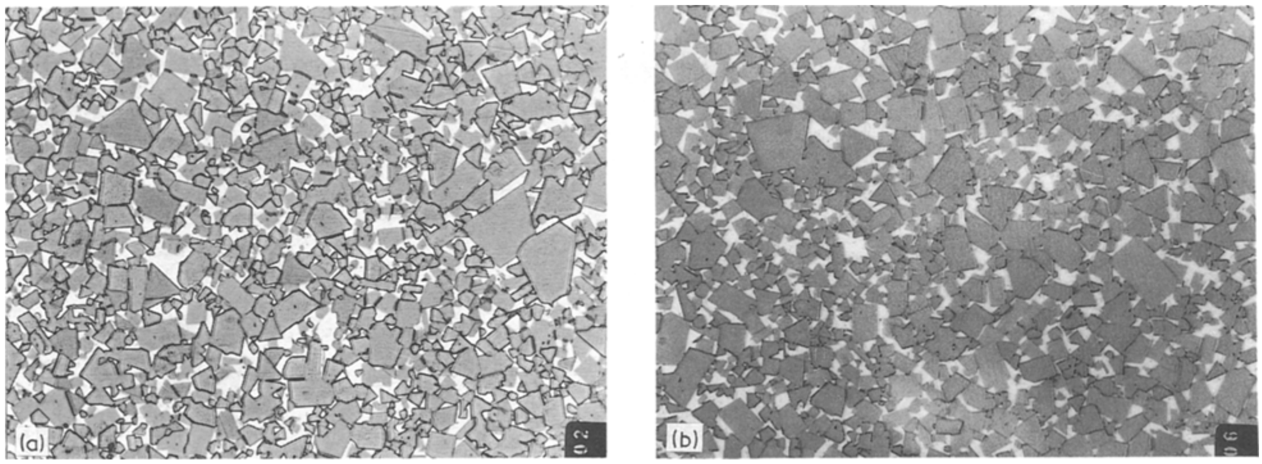

Figure 1 Typical micrographs of the two 10% cobalt grades considered. (a) IC11. (b) G10.

(b) creation of a damage zone at the notch tip, characterized by microcracks at the carbide–binder interface. If a fraction of these microcracks remains open during the unloading half-cycle, residual tensile stresses will be generated at the notch tip [16]. Additional factors influencing crack growth include residual tesselated stresses (due to thermal contraction mismatch) and sliding at the carbide–binder interfaces [16].

The phenomenon of fatigue crack growth under cyclic compression therefore requires the presence of a relatively blunt notch and involves crack growth at a progressively decreasing rate until arrest occurs, presumably at the boundary of the notch damage/residual tensile stress zone. The extent of crack growth from the notch root is a function of the magnitude of the minimum load in the compressive cycle and the applied stress ratio.

Some experimental evidence has indicated [18] that once a crack has naturally arrested, the maximum amount of damage ahead of its tip does not affect subsequent fracture toughness measurements. Thus the technique has been used to precrack ceramic fracture toughness specimens [17]. However, both the results reported in this paper and recent work by Godse *et al.* [19] indicate that residual damage ahead of the crack tip does affect values of fracture toughness. It is therefore necessary to extend the crack in tensile fatigue which is a relatively simple process once a sharp crack exists in the material.

3. Experimental details

The hardmetal used in this work was a WC–Co alloy containing 10% cobalt by weight. Specimens were supplied by two manufacturers to their specifications G10 and IC11, which represent a nominally similar standard grade used in the mining industry, with a coarse grain size of 2 to 3 μm. For the IC11 specimens dewaxing and pre-sintering were carried out simultaneously. Specimens were then cut from these "green" blocks, 20% oversize to allow for shrinkage during sintering, which was carried out in a vacuum furnace at 1420° C for 80 min. For the case of the G10 grade, a separate dewaxing cycle was used and sintering took place in hydrogen at 1400° C for 45 min. Sintered testpieces were then ground to size.

TABLE I Properties of the two hardmetal grades

	IC11	G10
Average grain size (μm)	2.92	3.05
Standard Deviation (μm)	1.55	1.85
Young's modulus (GPa)	590*	584*
Compressive strength (MPa)	4200*	4010*
Density (g cm^{-3})	14.60	14.51
Vicker's hardness (30 kg load)	1236	1203
Coercivity (Oe)	106	92

*Manufacturer's data.

Properties of the two hardmetal grades are given in Table I and typical micrographs are given in Fig. 1.

Simple bar specimens were used in all testing, with dimensions 60 mm × 12.7 mm × 6 mm. Notches were machined by electric discharge using a 0.25 mm diameter wire to depths of between 3.4 and 5.1 mm.

Compression–compression precracking was carried out between parallel platens in a 100 kN capacity Amsler vibrophore testing machine at a frequency of 170 Hz. A stress ratio $R = 10$ was used, where $R = \sigma_{min}/\sigma_{max}$ and σ_{min} corresponds to the most negative load (maximum compressive stress). Values of the maximum compressive stress in the cycle were estimated from dividing the compressive strength by the approximate notch stress concentration factor obtained from Roark and Young [20]. Using this as an initial load, the load range was incrementally increased and the arrest length of the notch root crack was measured.

These data are shown in Fig. 2 for specimens with notch depths of 3.4 to 3.7 mm and indicate that, although there is significant scatter in observed crack lengths for any given nominal stress (calculated as load divided by average cross-sectional area), there is a trend towards longer cracks with increasing maximum compressive stress, as found by other workers [18]. The trend line has a decreasing slope indicating that there is likely to be an upper limit to the length of crack which can be obtained. For notch depths between 3.4 and 4.3 mm, tested at nominal maximum compressive stress values between 920 and 1050 MPa, the arrested crack length only varied from 0.31 to 0.51 mm. Two tests were performed with initial notch

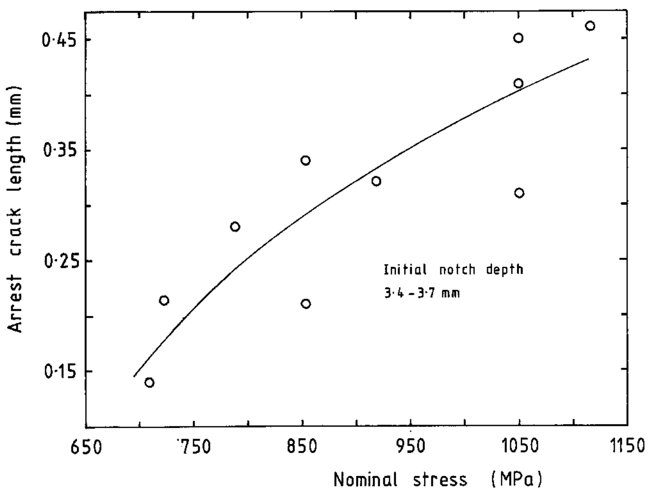

Figure 2 Crack arrest lengths as a function of nominal stress observed during compression fatigue of notched specimens with notch depths between 3.40 and 3.70 mm.

depths of approximately 5 mm, however, which gave crack lengths of 1.69 and 1.88 mm. The reason for this rather large increase in arrest length is not known and further work is required to clarify the effect.

A light micrograph of a typical small compression–compression precrack is shown in Fig. 3. The central planar crack is the one of interest, which extends through the thickness of the specimen. The roughly semicircular cracks correspond to local surface damage in the form of a cone, which is presumably related to increased local deformation which would occur under plane stress conditions.

Following this compression–compression precracking, certain specimens were subjected to normal fracture toughness testing in three-point bend at a strain rate of 1.5 MPa $m^{1/2}$ sec^{-1}, using a servohydraulic testing machine fitted with a 5 kN load cell. Notch mouth displacement was recorded via a small clip gauge and traces of load against displacement were produced.

In other specimens, however, the fatigue crack was subsequently extended by tension–tension fatigue in three-point bend, both to give a/W values of around 0.45 (as required by BS 5447) and to grow the fatigue crack through any residual damage zone ahead of the arrest position observed during compressive fatigue. The initial maximum stress intensity value was set at 5 MPa $m^{1/2}$ and a stress ratio of 0.2 was used. In order to maintain crack growth it was found necessary to incrementally increase the applied load, as seen in Fig. 4.

Dye penetrant was used to assist in measurement of fatigue crack length from the fracture surfaces. Typical fatigue crack shapes are shown in the fractographs of Fig. 5. The compression–compression cracks can be seen to be remarkably uniform in length with the exception of small regions near the specimen surfaces (Fig. 5a). This relatively small deviation disappears during the tensile fatigue, as can be seen in Fig. 5b.

Fracture toughness data were also obtained for the grade G10 alloy using V-notched short rod specimens [10] in a commercially available Terratek 'Fractometer' testing machine. These specimens were 14.3 mm long with a diameter of 9.5 mm.

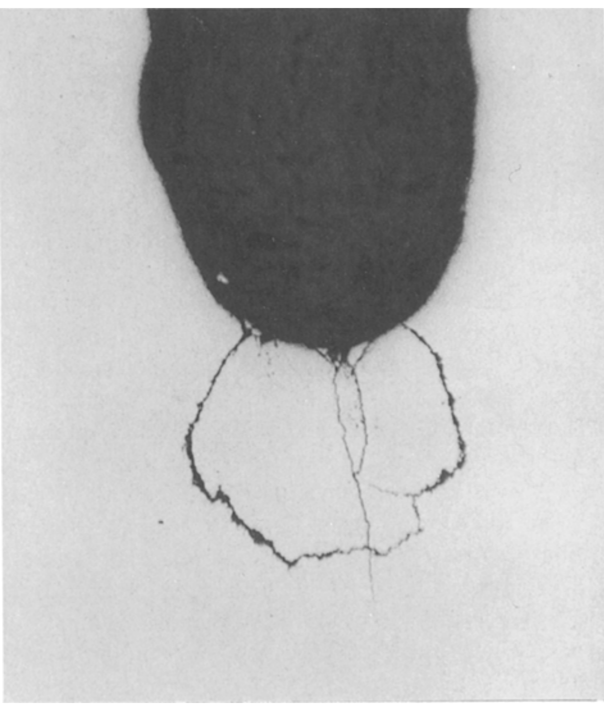

Figure 3 Typical cracking observed at the surface during compression fatigue. The central planar crack extends through the thickness of the specimen.

4. Results and discussion

The fracture toughness data obtained for the two 10% cobalt hardmetal grades used in the present investigation are given in Table II for the precracked specimens, while Table III presents the fractometer results

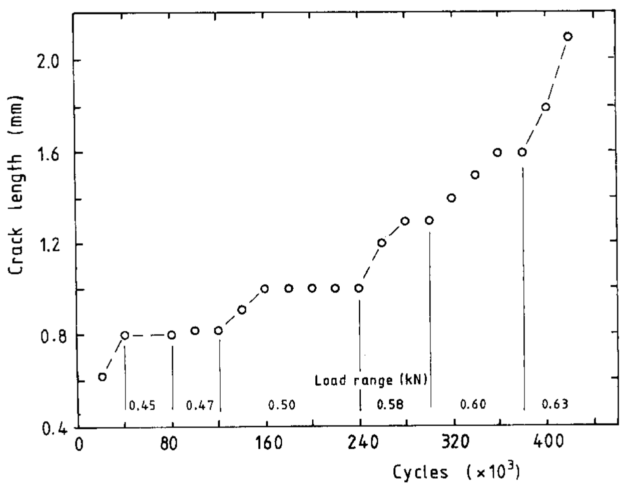

Figure 4 Crack growth during tensile fatigue. Crack arrest occurred repeatedly necessitating load increases.

TABLE II Fracture toughness data

Specimen no. and grade	Notch depth (mm)	Fatigue crack length (mm)	a/W	K_{Ic} (MPa m$^{1/2}$)
1 IC11	3.40	0.45	0.30	10.77
2	3.48	0.46	0.31	10.88
3	3.49	0.41	0.31	10.72
4	3.59	0.41	0.31	10.43
5	5.02	1.69	0.53	10.40
6	5.08	–	0.40	17.36
7	5.09	–	0.40	16.56
8	3.52	1.95 (T)	0.43	13.37
9	3.43	2.10 (T)	0.44	13.06
10 G10	4.29	0.45	0.37	10.14
11	4.17	0.42	0.36	10.54
12	3.97	0.41	0.34	10.76
14	4.15	1.53 (T)	0.45	13.49
15	4.30	1.09 (T)	0.43	13.19

(T) indicates crack extension by tensile fatigue.
W = 12.7 mm.

TABLE III Fractometer results for the G10 grade

	Specimen no.						
	1	2	3	4	5	6	7
K_{Ic} (MPa m$^{1/2}$)	13.71	14.95	13.92	13.92	14.78	14.35	14.11

obtained with the G10 grade. From these data several general observations can be made.

Firstly, there appears to be little variation evident in mean toughness values for the 10% cobalt grades supplied by the two manufacturers. For specimens precracked using only compression fatigue, the five IC11 tests gave an average toughness of 10.51 MPa m$^{1/2}$ and the three G10 tests gave an average of 10.48 MPa m$^{1/2}$. In view of the fact that the fracture toughness of cemented carbides depends on binder phase mean free path (or, synonomously, binder phase volume fraction and mean carbide grain size) and carbide contiguity, this agreement is quite reasonable. Fig. 1 indicates that the microstructures of the two hardmetal grades are closely similar in terms of average grain size and range of grain sizes. Although the coercivity values vary somewhat, and the coercivity is a measure of the mean free path of the binder for the fixed cobalt content, it is also affected by other factors such as the residual stresses in the specimens. Increasing coercivity indicates a decreasing binder phase mean free path and hence an increase in average grain size. Table I indicates that the G10 grade does indeed have a slightly larger grain size.

Secondly, although only a limited number of specimens had the precrack extended by tensile fatigue, the data from these tests are significantly higher than those obtained from the specimens in which precracking only involved compressive fatigue. This observation lends some support to the suggestion by Godse *et al.* [19] that, when a crack arrests during compressive fatigue, there is still a significant zone of tensile residual stress ahead of the crack tip. A peculiar aspect of these residual stresses, which merits further attention, relates to the arrest sequence under tensile loading observed in Fig. 4. Presumably, as the crack grows and relieves the tensile residual stresses this is affecting local crack closure and leading to the observed periods of crack arrest. When the crack length increases monotonically, the crack is believed to be outside the zone of residual damage developed during compression fatigue and an accurate fracture toughness value should be obtained. In the present work cracks were grown by tensile fatigue to a/W values of 0.43 to 0.45 prior to fracture toughness testing.

Comparing the data in Table II for the G10 grade with the Fractometer results in Table III, it appears that the two specimens in which the precrack was extended by tensile fatigue gave fracture toughness values significantly lower than the average Fractometer value of 14.25 MPa m$^{1/2}$. Indeed, both K_{Ic} values are lower than any of those determined using the short-rod specimens. General indications of the data in Table II also imply that the scatter in K_{Ic} values is less using the three-point bend specimens than that found with short-rod specimens.

Toughness data for specimens 6 and 7 in Table II were obtained from notched specimens without any precrack, to see the magnitude of the overestimation of K_{Ic}. The average toughness value from these tests is 28% higher than the average of the tensile fatigue tests.

Thus on the basis of the experimental work reported here it appears that, in the 10% cobalt grade

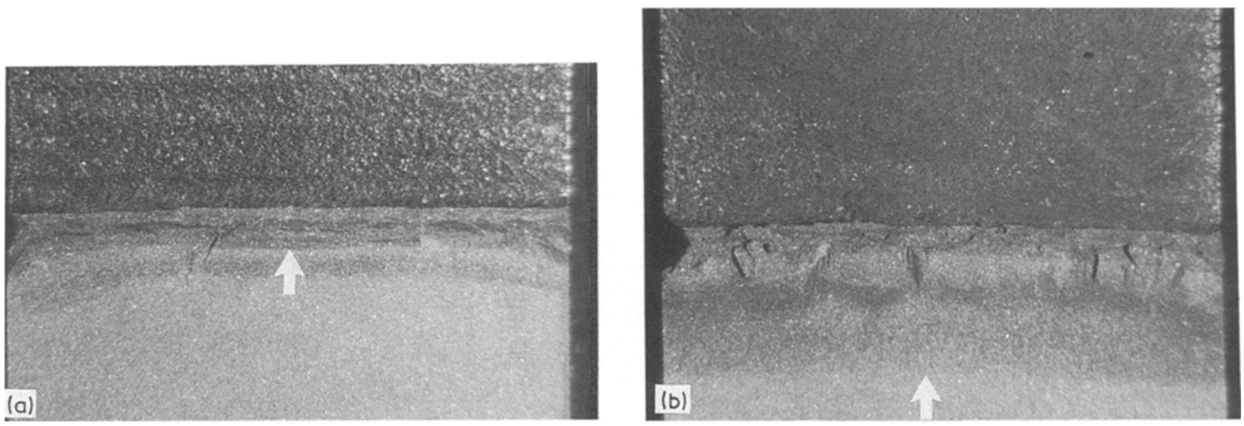

Figure 5 Fractographs of a (a) typical compression–compression fatigue precrack and (b) a precrack after compression fatigue followed by tensile fatigue. The arrows indicate the end of the precrack.

of WC–Co examined, the fractometer test may be overestimating the true K_{Ic} value by some 7%. Suresh *et al.* [17] have suggested that some evidence exists which indicates that fracture toughness determinations using the short-rod technique may depend on specimen geometry and initial crack length in some materials. Further work is required to elucidate the causes of the higher K_{Ic} values obtained in this study using the fractometer technique.

In the present case, using compression fatigue followed by tensile fatigue to develop a precrack to a/W values of approximately 0.45, and then performing a K_{Ic} test to the requirements of BS 5447, an average toughness value of 13.28 MPa m$^{1/2}$ was obtained for the 10% cobalt grade considered.

5. Conclusions

1. Precracking of WC–Co specimens by a combination of compressive and tensile fatigue allows fracture toughness testing to be performed which meets the requirements of BS 5447 (Methods of Test for Plane Strain Fracture Toughness (K_{Ic}) of Metallic Materials).

2. In the 10% cobalt grade examined, it appears that the fractometer, or short-rod, test technique may be overestimating the K_{Ic} value by some 7% and exhibiting higher scatter than obtained from three-point bend tests of precracked specimens.

3. At crack arrest during compression–compression precracking of a notched specimen, a zone of residual damage/stresses exists ahead of the crack tip which influences subsequent toughness determinations.

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