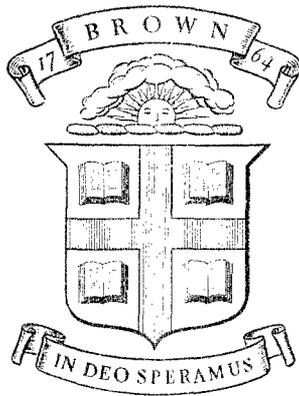


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BROWN UNIVERSITY
PROVIDENCE, R. I.

THE INFLUENCE OF SAMPLE PREPARATION
ON THE SURFACE STRESS STATE AND
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TESTING OF CEMENTED CARBIDES

H. E. EXNER

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THE INFLUENCE OF SAMPLE PREPARATION ON THE SURFACE STRESS STATE
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H. E. Exner^{*}

Division of Engineering
BROWN UNIVERSITY
Providence, Rhode Island 02912

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*Research Associate at the Max Planck Institute für Sondermetalle, Stuttgart, Germany. This work was finished at Brown University on a fellowship sponsored by the Materials Research Program of the Advanced Research Projects Agency under Contract No. SD-86.



ABSTRACT

Different views exist on the influence of surface preparation on crack formation at Vickers indentations in the test used by Palmqvist [3-7] to evaluate the toughness of cemented carbides. The present experimental results clearly show that grinding diminishes the thermal tensile stresses in the cobalt phase and thus reduces the crack length. The deformed surface layer is removed by polishing and a stress pattern is approached corresponding to that in the bulk material. Cracks of maximum length are characteristic of this stress state. Deformation stresses can be removed by annealing above 800°C. On the basis of these results, sample preparation for the crack length test can be simplified, the accuracy of measurement is improved and a simple and significant parameter is found to describe crack resistance of cemented carbides.

1. INTRODUCTION

Following suggestions by Amman and Hinnüber [1,2], Palmqvist [3-7] developed a method for testing the toughness of cemented carbides using as a measure the crack length at corners of Vickers hardness indentations. This method has several advantages over the bend test commonly used in carbide technology:

- a) Specially formed samples are unnecessary,
- b) Sufficient information can be obtained from one sample,
- c) The scatter is relatively small,
- d) The range of actual values for commercial cutting alloys is wide.

Thus, this test is both cheaper and more precise than the bend test.

Nevertheless, Palmqvist's method is not widely used, mainly for two reasons:

- a) The relationship between crack length and the technological properties of the material (wear and shock resistance) are not clear.
- b) The crack length is strongly dependent on surface treatment. It is necessary, therefore, that surface preparation be very precise and reproducible, conditions usually hard to obtain.

While any other simple mechanical test has this first drawback, the second is crucial in the applications of the crack length method as a practical test. On the other hand, the sensitivity of crack length to surface preparation provides a good method of determining the stresses introduced by different grinding and polishing techniques [5-7]. Furthermore, study of this problem could give new information on crack propagation in cemented carbides.

Öhman, Pärnama and Palmqvist [8] summarized their views on the experimental results in an intensive study which will be discussed in detail. Two critical observations are that the crack length in a given alloy is greater after grinding with diamond wheels than with silicon carbide wheels, and that the crack length increases when the surface is polished. Palmqvist [3-7] attributed

these phenomena to the effect of tensile stresses introduced into the cobalt phase by diamond grinding and polishing. Another explanation was proposed by Dawihl and Altmeyer [9] and independently by Exner [10]: they suggest that compressive stresses are introduced into the cobalt phase by silicon carbide grinding which produce shorter cracks. Little attention, however, was given to this point of view in the most recent papers [8,11]. Since X-ray measurements have disproven Palmqvist's initial suggestion, Öhman, Pärnama and Palmqvist [8] propose that polishing work-hardens and thus embrittles the cobalt phase, thereby increasing crack length. This suggestion, however, is disproven by the experiments described below. Miyoshi, Hara and Sugimoto [11] consider work-hardening of tungsten carbide by polishing as an explanation. They present experimental evidence that an increase in microhardness of tungsten carbide after polishing accompanies an increase in crack length, but do not prove their statement that the crack proceeds through the carbide. According to the views generally accepted at present [4, 7-9, 12-16], crack propagation in commercial WC-Co alloys with grain sizes up to 2 μm occurs mainly in the binder phase. In this case, work-hardening or any other change in properties of tungsten carbide should not have much influence on crack formation at Vickers indentations.

In this paper we put forward the following explanation for the changes in crack length with surface preparation. Grinding introduces compressive stresses into the surface. These diminish the thermal tensile stresses present in the cobalt phase after cooling from the sintering temperature and thereby decrease the crack length. Polishing removes the layer deformed in grinding, increasing the crack length. On the basis of this interpretation, which is consistent with present views on both the grinding and polishing processes and the fracture of cemented carbides, a more complete understanding of the influence of surface preparation on crack length and on the surface stress state is obtained.

Improvements in the method of crack length testing are proposed.

2. PREVIOUS EXPERIMENTAL RESULTS

In his first attempt to use the cracks at Vickers impressions as a qualitative measure of the toughness of cemented carbides, Palmqvist [3] calculated the work necessary to initiate cracks by the formula

$$S_k = k \cdot P_k \cdot \sqrt{P_k/HV} \quad (1)$$

where k = numerical constant = 6.49

S_k = critical work to initiate cracks (p cm)

P_k = critical load (load necessary to initiate cracks) (kp)

HV = Vickers hardness (kp/mm²)

The difficulties involved in experimental determination of the critical load were overcome by the observation that a linear relation holds between the applied load and the sum of the crack lengths at the four corners of the indentation. This linear function, given by Eq. (2) below, was confirmed by Dawihl and Altmeyer [9] and by our experiments (see Fig. 1).

$$\sum l = a_1 \cdot P + a_2 \quad (2)$$

where $\sum l$ = sum of the crack lengths at a Vickers indentation

P = applied load

a_1, a_2 = parameters dependent on the toughness of the alloy and the surface preparation.

The critical load, P_k , is now given by a_1 , which can be obtained by extrapolating the load-crack length line to zero crack length (see Fig. 1). This simple relationship (Eq. (2)) led Palmqvist [4,7] to a parameter which was more reproducible and easier to obtain experimentally than the critical work: the work, S_{300} , put into the sample to produce an average crack length

of 300 μm per indentation. This parameter is calculated using the formula

$$S_{300} = k \cdot P_{300} \sqrt{P_{300}/HV} \quad (3)$$

where the load, P_{300} , giving a crack length of 300 μm , can be taken from the load-crack length plot (Fig. 1) or calculated from Eq. (2) by putting $l = 300 \mu\text{m}$.

The significance of the work for crack formation as a measure of toughness was discussed by Dawihl and Altmeyer [9] and by Holzberger [17], who pointed out that S is not directly related to toughness in a practical sense, because of the different character of the loading in hardness testing and in machining or drilling. Palmqvist [4] claims that the variation in work of crack formation corresponds to that in technological properties and that toughness in the sense of resistance to impact and cracking is very well measured by this parameter. He demonstrates a close correlation between the S_{300} value and the composition of WC-Co and WC-TiC-Co alloys. The crack length method is sensitive to plastic deformation of a sample previous to testing while the hardness test per se, the transverse rupture strength or other mechanical tests are not, and thus provides additional information on the mechanical properties of cemented carbides [8,18].

The influence of surface preparation on crack length has been thoroughly investigated, and the following reproducible observations have been made [4-9]:

- a) After grinding with a silicon carbide wheel, the crack length in a given alloy is a minimum.
- b) Diamond grinding produces longer cracks.
- c) Honing and polishing extend the cracks to a maximum which is typical for every step of surface treatment.
- d) Electrolytic polishing after silicon carbide grinding also extends the cracks.

- e) Annealing after polishing does not change crack length significantly.
- f) The slope of the load-crack length function (a_1 , Eq. (2)) changes during surface treatment, increasing in the finer steps.
- g) The intercept of the load-crack length function (a_2) tends to zero after prolonged polishing.

Öhman, Pärnama and Palmqvist [8] measured the strains in the tungsten carbide phase using the two-exposure X-ray diffraction method, which makes possible accurate calculation of the residual stresses. They obtained the following results:

- a) The stresses in the tungsten carbide are compressive after any kind of surface preparation.
- b) Ground surfaces show the highest compressive stresses. Polishing decreases these stresses to a constant level which is higher than that in a fracture surface.
- c) The magnitude of the stresses present in a polished surface is not significantly changed by heat treatments up to 800°C.

3. INTERPRETATION OF PREVIOUS EXPERIMENTAL OBSERVATIONS

Öhman, Pärnama and Palmqvist [8] summarized their views on the experimental results in the following statements:

- a) No compressive stresses are introduced into the cobalt phase.
This is a direct corollary of the assumption that the stress states in the two phases are coupled. Compressive stresses in the cobalt phase would then produce tensile stresses in the tungsten carbide; such tensile stresses have not been observed.
- b) More tensile stress is introduced into the binder by diamond grinding than by silicon carbide grinding.

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- c) Crack length is mainly a function of strain- and work-hardening; these occur independently in the two phases and result in an embrittlement of the cobalt phase.
- d) The cobalt phase is only slightly strain-hardened after sintering. Polishing, a cyclic deformation process, strain-hardens the cobalt phase to the extent that the crack propagation mechanism changes from the ductile, Griffith-Orowan type to a completely brittle, Griffith type.
- e) Strain-hardening by polishing reaches a saturation value which is independent of previous treatment and which results in maximum crack length.
- f) Heat treatment does not reduce the stresses, but might diminish the strain-hardening.

Some of these statements seem rather complicated, not fully consistent with the experimental results, or completely inconsistent with the usual points of view on the relative influences of grinding and polishing. In essence, these difficulties arise, we feel, from two assumptions which Palmqvist has stated as axioms in all of his papers:

- a) The stresses in the two phases are coupled in such a way that all compressive stresses in the tungsten carbide must be balanced by tensile stresses in the cobalt phase, and vice versa, in any volume element of the sample.
- b) The cracks for a given alloy are shortest in an "ideally" ground surface, where no additional effects are introduced by surface treatment and the stress pattern is equivalent to that in the bulk material. Any increase in crack length, therefore, shows a deviation from this stress pattern and should be avoided.

Both assumptions, in our opinion, are incorrect. Let us look at the coupled stress system first. Thermal stresses due to the different coefficients of thermal expansion are compressive in the tungsten carbide and tensile in the cobalt phase. The stresses of opposite signs in the two phases balance each other in volume elements comparable with the grain size, and are thus completely coupled. Stresses introduced by grinding, on the other hand, are a function of the distance from the ground surface. Compressive stresses near the surface are balanced by tensile stresses towards the middle of the specimen. Residual stresses due to grinding may thus very well be of the same sign and even of the same order of magnitude in different phases.

Residual stresses resulting from grinding in steels have been the subject of extensive investigation [19-26]; these studies give us an estimate of the stresses to be expected in cemented carbides. It has been shown experimentally (and is to be expected theoretically) that the residual thermal stress pattern in cemented carbides is not influenced by annealing, and therefore no effect of the heating and cooling cycle in grinding is to be expected. The stress pattern in hardened steel ground so as to avoid phase transformations and heat effects is shown in Fig. 2. The depth and the magnitude of the stresses may be different in cemented carbides, but the principal features should be the same: compressive stresses are present near the surface and are balanced by tensile stresses further down in the sample. The stress distribution predicted in a tungsten carbide-cobalt alloy is shown in Fig. 3. Grinding superimposes compressive stresses in the surface layer, increasing the compressive stresses in the tungsten carbide and decreasing the tensile stresses in the cobalt phase.

The second assumption has been discussed in previous papers [9,10]. Dawihl and Altmeyer [9] gave the following explanation for the short crack lengths after silicon carbide grinding: The silicon carbide wheel removes material by impact working and thereby introduces compressive stresses, while

in diamond grinding, the material is removed by preferential cutting. The compressive stresses introduced by silicon carbide grinding must be overcome by the tensile stresses in loading before cracking appears; thus, crack length is decreased. However, Dawihl and Altmeyer follow this line of reasoning only as far as grinding is concerned. They go back to Palmqvist's original concept in discussing the influence of honing and polishing, and state that additional tensile stresses must be introduced by the finer steps of mechanical surface preparation. Because of this surprising step, they are not able to give a consistent explanation of their experimental results.

Let us look at this problem in more detail. Because cobalt contracts about three times as much as tungsten carbide when cooled from the sintering temperature, the residual tensile stresses in the cobalt add to the effective load and facilitate crack propagation. Any compressive stresses introduced in the surface will diminish the tensile stresses and increase resistance to crack propagation. But even if the sum of the stress changes in the surface is zero, heavy machining, such as silicon carbide grinding, could decrease the crack length. When a carbide grain is hit by a hard particle of the grinding wheel it might break, thus allowing the cobalt matrix to relax, or it might change its position so that the cobalt matrix is under greater tensile stress on one side and compressive stress on the other side of the particle. Some of the cobalt layers will therefore be stronger than in the unmachined state; these layers will resist the applied tensile stress and stop the crack.

It is much more probable that the crack length is decreased (rather than increased, as Palmqvist states) by any change of the stress pattern present after cooling. This change is largest for coarse grinding. The original stress state is approached to a certain extent by honing, polishing, electro-polishing or heat treatment. If one takes this point of view, almost all of the observed phenomena can be interpreted very easily.

4. EXPERIMENTAL PROCEDURE

All experiments were carried out on a fine grade of a 93 wt% WC, 0.8 wt% TaC, 0.2 wt% NbC and 6 wt% Co alloy, the mean linear grain size and Vickers hardness of which were 0.5 μm and 1740 kp/mm^2 respectively. The samples were cylindrical platelets (25 \times 5 mm) ground on their flat surfaces (Superfix diamond wheel type B212, grain size 150 μm , concentration 75, speed 22 m/sec). The samples were polished on an automatic machine (Buehler Whirlimet, 285 rpm, specific pressure ~ 0.7 kp/cm^2), using diamond paste (15, 7, 3 and 1 μm) manufactured by Winter & Son, on perlon cloth. The thickness of the layer removed was measured by the decrease in the diameter of the Vickers indentation and calculated by the formula

$$A = \frac{D_1 - D_2}{4} \cdot \sqrt{2} \cot \frac{\alpha}{2} = 0.143 (D_1 - D_2) \quad (4)$$

where A = thickness of the layer removed,

D_1, D_2 = diagonals of the Vickers indentation mark before and after polishing, respectively,

α = angle between the faces on the top of the Vickers pyramid (136°).

The polishing conditions were kept as constant as possible. It was found that there was no direct relationship between polishing time and amount of surface removed: often more material was removed after 5 than after 30 minutes of polishing. The variation in the depth removed (1-5 μm) each time new diamond paste is added is large, but the mean values are nearly the same for all grades of diamond paste (3 μm depth for the 15 and 7 μm grades, 2.5 μm depth for the 3 and 1 μm grades). As a standard procedure, therefore, polishing was stopped every 5 minutes and new diamond paste was added. The thickness of the layer removed was measured after every step and is used as a measure of polishing action.

One sample was electropolished in 2% aqueous NaOH. A layer 7 μm thick was removed, but the surface was quite rough and thus the crack length measurements are not very accurate. Two other samples were heat-treated in hydrogen.

Hardness indentations were made on a Frank universal hardness tester and measured on a Zeiss Ultraphot II microscope at a magnification of 300 X. Measurement of the crack length is accurate to about $\pm 2 \mu\text{m}$. During the experiments it was necessary to decrease the test load from 150 to 50 kp in order to avoid damaging the diamonds. The parameter used to describe crack length is the sum of the crack lengths at each indentation mark. Crack lengths presented in Figs. 1 and 4-7 are the mean values for five indentations.

5. EXPERIMENTAL RESULTS

As a first step the relationship between applied load and crack length was checked for a ground and a polished sample (1 h, 15 μm diamond, thickness of layer removed 40 μm). The results, shown in Fig. 1, agree completely with those given by Palmqvist [4-7] and Dawihl and Altmeyer [9]. The relationship is linear, the slope, a_1 , of the line is higher for the polished than the ground surface and the intercept, a_2 , on the load axis (the critical load) disappears after polishing.

Our results on the influence of progressive polishing are similar in principle to those of previous experiments [4-7, 9, 11]. As shown in Fig. 4, crack length increases proportionally to the thickness of the layer removed and reaches a constant value at 30 μm thickness removed. The data in Fig. 4 are the results of three independent test series, showing good reproducibility. Figure 5 shows the influence of polishing in several steps. After a layer of 20 μm was removed a finer grade of diamond paste was applied. At first, the increase in crack length was great, but after 30 μm of surface was removed a constant value is again reached and is not influenced by the grade of diamond

paste used thereafter.

Figure 6 shows the change in crack length with annealing for a ground sample. The surface was polished slightly (5 min, 15 μm diamond paste) and the crack length after annealing could be measured without any further surface finishing. There is a marked increase in crack length after heat treatment at 700°C for two hours. At 800°C a crack length value is obtained which is slightly higher than the final value in polishing (see Fig. 5) and which is not altered by subsequent heat treatment up to 1100°C. The results of the heat treatment of a second sample were essentially the same, but after annealing at 1100°C the cracks were much shorter. This observation could be explained by postulating the formation of a cobalt-rich surface layer [8]. As shown in Fig. 7, the crack length in an annealed sample is decreased slightly by polishing and coincides with the final crack length in the sample polished without previous heat treatment (see Fig. 5). Again, there is no noticeable influence of the grade of diamond paste used.

The cracks in the electropolished sample were much longer than those in the ground samples, which is in agreement with previous results [9]. However, here the maximum crack length value reached in the annealing experiments and approached in the polishing experiments was not obtained, even when mechanical polishing preceded electropolishing. Before trying to explain this observation on the basis of the stress state, more probable explanations must be excluded, such as the enrichment of the surface by cobalt (which is insoluble in NaOH) or the fact that the cracks are less visible after electropolishing than after mechanical polishing. This question can be settled only if a better electropolished surface is achieved.

6. DISCUSSION OF EXPERIMENTAL RESULTS

The results of the annealing experiments clearly prove that the short crack length in ground samples is caused by surface deformation and that the removal of this deformed layer by polishing produces a stress state almost equivalent to that in a completely undeformed surface. Figure 6 shows that the compressive stresses introduced in the surface by grinding (see Fig. 3) can be relaxed by annealing in cemented carbides as well as in steel [21,22]. In annealing experiments by Öhman, Pärnama and Palmqvist [8], the crack length and the stress state of a polished sample were to a great extent unchanged. A change would have supported their assumption that the cobalt phase is embrittled by polishing. Their X-ray measurements of the surface stresses show, in contrast, that the stress state in an annealed or polished surface is about the same as that in a fracture surface (which can be regarded as the best approximation of the original stress state). The crack length measured after annealing (Fig. 6) (which is approached to a certain degree by removing the layer deformed in grinding by polishing (Figs. 4, 5 and 7)) corresponds to the original stress state produced exclusively by residual thermal stresses. The same reasoning disproves the conclusions reached by Miyoshi, Hara and Sugimoto [11], who claim that work-hardening of the tungsten carbide is essential for the production of long cracks after polishing. No influence of heat treatment was observed (in agreement with [8]); therefore, the increase in crack length in ground specimens cannot be explained on the basis of their concept.

Öhman, Pärnama and Palmqvist [8] observed that samples after sintering are stronger in bending than ground samples, and cited this observation as evidence against the idea that compressive stresses are introduced by grinding, since, if this were true, the bend strength should increase after grinding because of compressive stresses in the cobalt phase. This argument, however, seems invalid, because in grinding the cobalt-rich sintering layer is removed

and flaws and scratches may be introduced. These effects may reduce the bend strength more than any compressive stress in a layer about 30 μm thick would improve it. Polished and ground samples should be compared for evidence on the effect of compressive stresses. Filimonenko [27] has shown that an alloy whose bend strength was about 170 kp/mm^2 in the as-sintered and polished states had a bend strength of more than 200 kp/mm^2 after grinding with a 100 μm diamond wheel. This result is not cited as additional evidence for our point of view, since the bend strength is very insensitive to the residual stress pattern [18]. It disproves, however, the argument against it mentioned above.

Dawihl and Altmeyer [9] pointed out that the small critical loads (a_2) after polishing indicate that little surface deformation has taken place. This implies that the high critical loads (up to 80 kp) extrapolated by Palmqvist [4-7] are a result of superimposed compressive stresses. The disappearance of the critical load shows that only very small additional stresses are necessary for crack propagation from a sharp notch such as the corner of a Vickers indentation. This is reasonable since the strength of notched specimens is only a small fraction of their nominal bend strength. The critical work for crack formation, which was initially used by Palmqvist [3] as a measure of toughness, is determined by the surface stress state exclusively and is therefore not a valid parameter for a bulk property of an alloy. We cannot agree to the conclusion of Dawihl and Altmeyer [9] that these restrictions are true for the slope (a_1) of the load-crack length curve as well. We consider this slope the best parameter for the crack resistance of an alloy if it is measured at a constant stress state (which should preferably be the one given exclusively by the stress state of the bulk material).

As Palmqvist [5-7] has pointed out, the damage caused by surface preparation can be measured by the crack length test. Knowledge of whether

long or short cracks indicate deformation is, of course, essential for the correct interpretation of the results. In our opinion, crack length measurements [4,7,9] show that diamond grinding does not produce as much damage as silicon carbide grinding. The depth of the layer deformed by grinding in our experiments was about 30 μm (see Figs. 4 and 5). Crack length after honing is smaller than that after prolonged polishing [9], indicating that appreciable compressive stress is introduced by honing. A surface with minimum deformation and superimposed stresses is reached by polishing. Any influence of the grade of diamond paste used could not be observed in the range between 15 and 1 μm (Figs. 5 and 7). Whether or not a stress free surface can be approached even more closely by using finer grades of diamond paste has yet to be tested. The conclusions suggested by the crack length test are in full agreement with the common views on the influence of the different steps of surface preparation [28].

Furthermore, the results of the annealing experiments (Fig. 6) give some estimate of the temperature at which the thermal stresses become zero. Values for this temperature are necessary in the calculation of the residual stresses at room temperature [8], and are important in evaluating the influence of residual stresses on the mechanical strength of cemented carbides [12]. Since the crack length of a sample annealed at 700°C increases when it is annealed at 800°C but remains constant for higher temperatures, this critical temperature should be higher than 700 but lower than 800°C. This value is in rough agreement with that calculated from X-ray measurements of compressive stresses in tungsten carbide [8], but much higher than that assumed by Kreymer, Alekseyeva and Vakhovskaya [12].

As suggested by Fig. 3, high compressive stresses in the surface tungsten carbide should, in our opinion, be coupled with low rather than high tensile stresses. Thus, shorter cracks should be found at higher compressive stresses.

Figure 8 shows experimental results of Öhman, Pärnama and Palmqvist [8] which give qualitative support to this view.

Not all our experimental results may be interpreted without difficulty. It has already been mentioned that the maximum crack lengths were not obtained by electropolishing, as had been expected. Furthermore, cracks longer than any on the annealed specimens were occasionally found after extended polishing with 3 μm diamond. X-ray measurements [8] showed a slight increase in compressive stress in tungsten carbide, accompanied by a decrease in crack length, when polished samples were annealed at 800°C. However, we shall assume here that these anomalous results were caused by experimental errors. There is a possibility, however, that cracks after mechanical polishing may be longer than those for the "ideal" surface, or that an "ideal" stress state is not obtained by annealing and some influence of previous deformation still remains. Another explanation could be that polishing causes some work-hardening and thus embrittles the cobalt phase (as suggested by Öhman, Pärnama and Palmqvist [8]). Further experiments are desirable, and it may be necessary to modify some of the conclusions which follow; the principal concepts, however, should remain essentially unaltered.

7. CONCLUSIONS

Our interpretation of the experimental results may be summarized as follows.

- a) Coarse grinding reduces the residual tensile stresses in the cobalt phase. The cracks at Vickers indentations become shorter with increasing surface deformation and damage by the mechanical surface treatment.
- b) Annealing at temperatures about 800°C removes the damage produced by grinding and restores a surface stress state corresponding to that

- of the bulk material.
- c) The increase in crack length resulting from honing and polishing is caused by the removal of the layer deformed in grinding and not by introduction of additional tensile stresses into the cobalt phase.
 - d) Embrittlement of the cobalt phase or of the tungsten carbide by work-hardening after prolonged polishing cannot be completely excluded. Its contribution, however, is not very important, and may even be negligible.
 - e) Mechanical surface treatment influences the crack length mainly by altering the stress state from that produced by residual thermal stresses.
 - f) Crack length in a given alloy reaches a maximum if the surface preparation does not introduce additional stresses into the cobalt phase, but reproduces a stress state corresponding to that in the bulk material.
 - g) This maximum crack length is determined exclusively by the load and the properties of the alloy (residual thermal stresses, composition and structure) and is the best measure for the crack resistance of an alloy.

8. SUGGESTIONS FOR IMPROVEMENTS IN THE CRACK LENGTH TEST

The conclusions above suggest a substantial change in surface preparation of samples for the crack length test. In contrast to the preparation method recommended hitherto [4-7], the surface should be prepared so that the maximum crack length for a given alloy is obtained. In comparison to the directions given by Palmqvist [4-7], which are very difficult to follow in practice and are not very reproducible [17], the grinding and polishing can be done in arbitrary steps with the machines and wheels available. The only important

requirements are the complete removal of the layer deformed in the coarse machining steps (grinding and honing) and the consequent achievement of a stress state typical of a polished surface. Mechanical removal of the deformed layer can be replaced by annealing at 800 to 1000°C. Polishing the annealed specimen with a fine grade of diamond paste for a short time will produce the same stress state as that achieved by long polishing.

The accuracy of the measurements is improved substantially by this method for three reasons: the cracks are more visible on a scratch-free surface; the cracks are longer; and the surface treatment is easy to reproduce.

The following procedure for sample preparation was found useful, though, of course, it can be varied. After diamond grinding the sample is polished with 15, 7 or 3 μm diamond paste. The paste is renewed ten times at short intervals (3 to 5 minutes), and the sample is finally polished with 1 μm diamond paste. After testing another layer is removed (in 2 to 3 steps) and testing is repeated to assure that equilibrium has been obtained.

A second fact which is important for simplifying the crack length test and which has not yet been emphasized adequately [4,9] is the disappearance of the critical load after polishing. The relationship between the crack length, $\{l$, and the load, P , then becomes

$$\{l = a_1 \cdot P \quad (5)$$

and one single measurement gives a pair of values sufficient to establish it. This suggests a reformulation of the parameter used. Instead of the crack length or the work for crack formation, both of which require a definition of testing conditions (constant load and constant crack length, respectively), the resistance to crack formation can be expressed by the reciprocal slope of the load-crack length line a_1 (Fig. 1), which we shall call the "crack resistance", W :

$$W = \frac{P}{l} \quad (6)$$

The advantages of this parameter are that its numerical value is independent of the load and of the hardness of the alloy (compare Eqs. (1) and (3)) and that it is very easy to calculate. The dimension (force per unit length) corresponds to energy per unit area and suggests a simple relationship to a physically significant property of the material. A sufficiently accurate analysis of the stress field around a Vickers indentation has not yet been achieved, nor is the fracture mechanism in cemented carbides completely understood. The practical advantages should therefore be the deciding factor for using the crack resistance, as defined in Eq. (6), as the characteristic parameter for the crack length test.

The wear on the diamond indenter, which influences the test results [17] and is most severe for high loads and low-cobalt alloys, can be reduced by using an optimum testing load. For hard alloys small loads may be used, but for tough, cobalt-rich alloys high loads (up to 200 kp) must be used in order that the cracks produced are long enough to assure fair accuracy. Since the crack resistance, W , is independent of the load, values of W obtained for different alloys can be compared directly.

With these modifications the crack length test becomes an extremely simple one, and standardized precepts are unnecessary to give comparable results. The next question, essential for its use as an industrial test, is that of the relationship between crack resistance and material properties in practical use. This relationship is not yet clear, mainly because quantitative parameters characterizing wear resistance or other properties in application are hard to define and difficult to measure. It is quite well established, however, that the crack resistance (or work of crack formation) is intimately connected to the composition, the structure and other properties (coercive

force, hardness) of cemented carbides [4-7, 13, 14]. In addition, Palmqvist [4-7], Dawihl and Altmeyer [9] and Exner and Gurland [18] have found experimental evidence that the crack length test gives information on mechanical behavior which cannot be obtained by hardness or fracture strength measurements.

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REFERENCES

1. Amman, E. and Hinnüber, J., Stahl und Eisen, 71, p. 1081 (1951).
2. Hinnüber, J., Stahl und Eisen, 62, p. 1083 (1942).
3. Palmqvist, S., Jernkont. Ann., 141, p. 300 (1957).
4. Palmqvist, S., Arch. Eisenhüttenwes., 33, p. 629 (1962).
5. Palmqvist, S., Jernkont. Ann., 147, p. 107 (1963).
6. Palmqvist, S., paper presented at meeting of SVIF, tool section, Stockholm (1963).
7. Palmqvist, S., Jernkont. Committee for Powder Metallurgy, Report JK-U8 62-15 (1962).
8. Öhman, S., Pärnama, E. and Palmqvist, S., Jernkont. Ann., 151, p. 126 (1967).
9. Dawihl, W. and Altmeyer, G., Z. Metallkde., 55, p. 231 (1964).
10. Exner, H. E., Research Report of the Cemented Carbide Laboratory of Söderfors Bruk, Söderfors, Sweden (1964).
11. Miyoshi, A., Hara, A. and Sugimoto, Y., Planseeber. Pulvermetallurgie, 15, p. 187 (1967).
12. Kreymer, G. S., Alekseyeva, N. A. and Vakhovskaya, M. R., Phys. Met. Metallogr., 15, p. 88 (1963).
13. Exner, H. E., Thesis, Montanistische Hochschule (Institute for Mining and Metallurgy), Leoben (1964); Jernkont. Committee for Powder Metallurgy, Report JK-U8 64-20 (1964).
14. Fischmeister, H. and Exner, H. E., Arch. Eisenhüttenwes., 37, p. 499 (1966).
15. Fischmeister, H., Tekn. Tidskrift, 95, p. 761 (1965).
16. Dawihl, W. and Frisch, B., Arch. Eisenhüttenwes., 36, p. 449 (1965).
17. Holzberger, J., Diskussion, Arch. Eisenhüttenwes., 33, p. 634 (1962).
18. Exner, H. E. and Gurland, J., to be published.
19. Letner, H. R., Trans. ASME, 77, p. 1089 (1955), and 79, p. 149 (1957).
20. Litmann, W. E., Proc. CIRP Intern. Conf. on Manufacturing Technology (1967); SAE Paper 793-A (1964), and paper presented at the Fall Meeting of the Metallurgical Society of the AIME, Cleveland (1967).

References (continued)

21. Letner, H. R. and Sauvageot, A. B., Metal Progress, p. 79 (1957).
22. Halverstadt, R. D., Trans. ASME, 80, p. 929 (1958).
23. Hendriksen, E. K., Trans. ASME, 73, p. 69 (1951).
24. Roberts, J. G., SAE Paper 467-B (1962).
25. Frisch, J. and Thomsen, E. G. ASME Paper 50-F-10 (1950).
26. Field, M. and Kahles, J. F., paper presented at the Fall Meeting of the Metallurgical Society of the AIME, Cleveland (1967).
27. Filimonenko, V. N., Thesis, Institute of Electrical Engineering, Novosibirsk (1966).
28. Petzow, G. and Exner, H. E., in Handbuch der Mikroskopie in der Technik, edited by F. Freund (Umschau Verlag, Frankfurt) (in press).

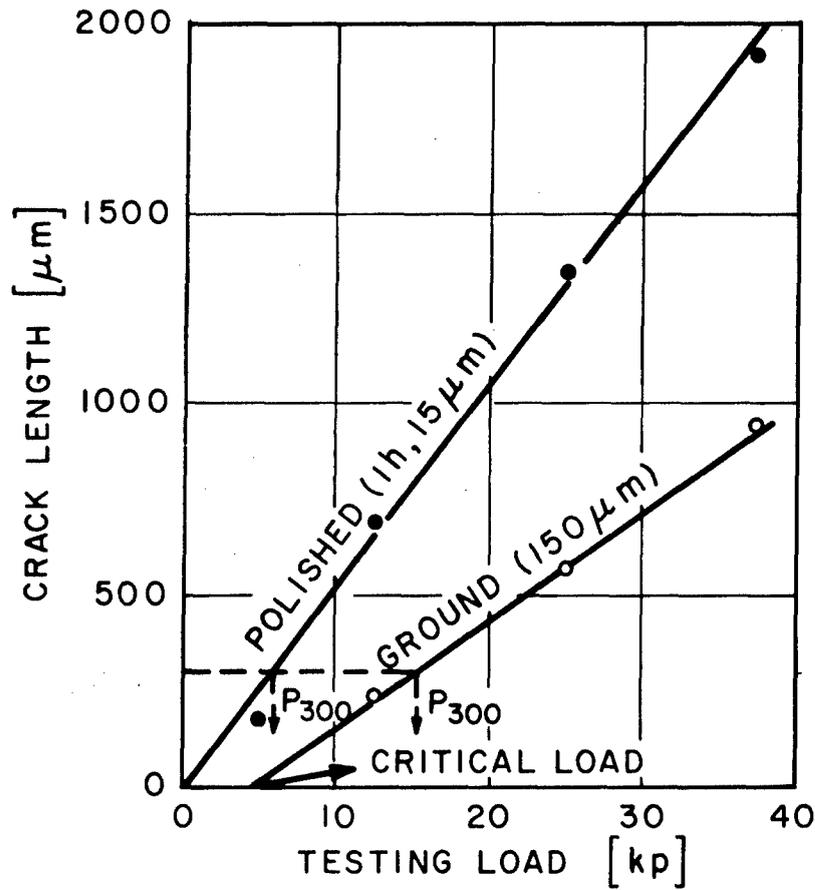


FIG. 1 CRACK LENGTH AS A FUNCTION OF APPLIED LOAD.

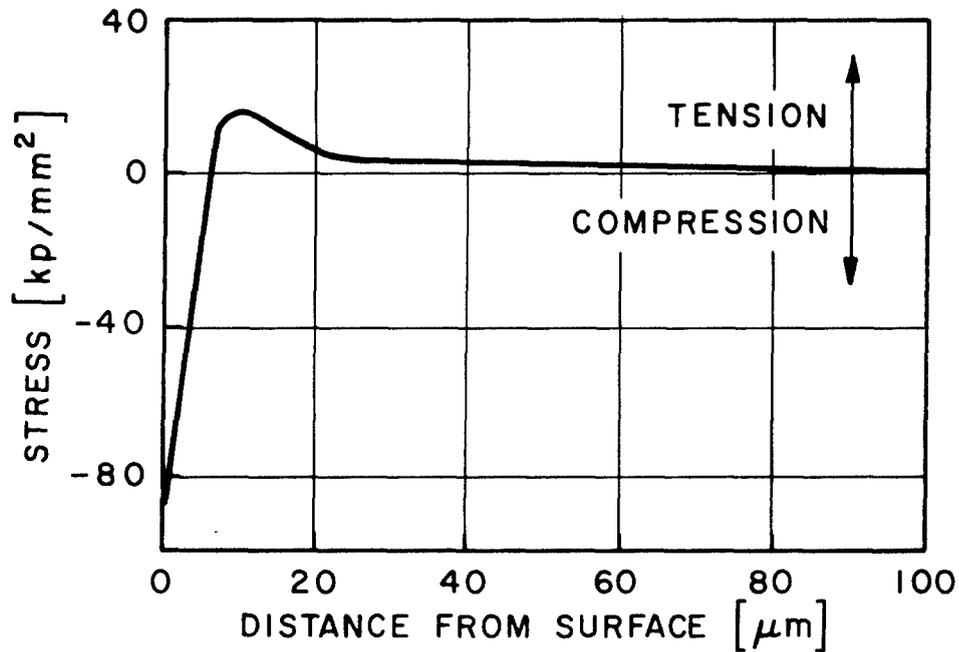


FIG. 2 STRESS DISTRIBUTION VS. DISTANCE FROM SURFACE IN HARDENED STEEL. (AFTER LETNER (19, 20))

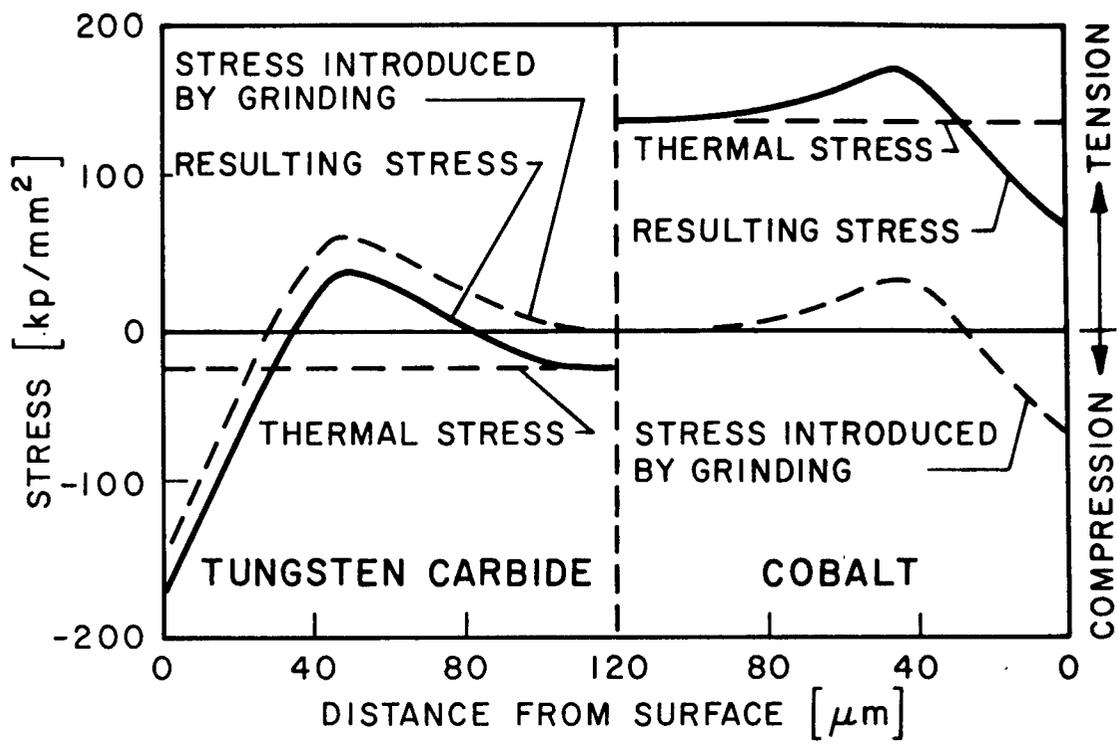


FIG. 3 STRESS DISTRIBUTION VS. DISTANCE FROM SURFACE IN A WC-CO ALLOY. (SCHEMATIC)

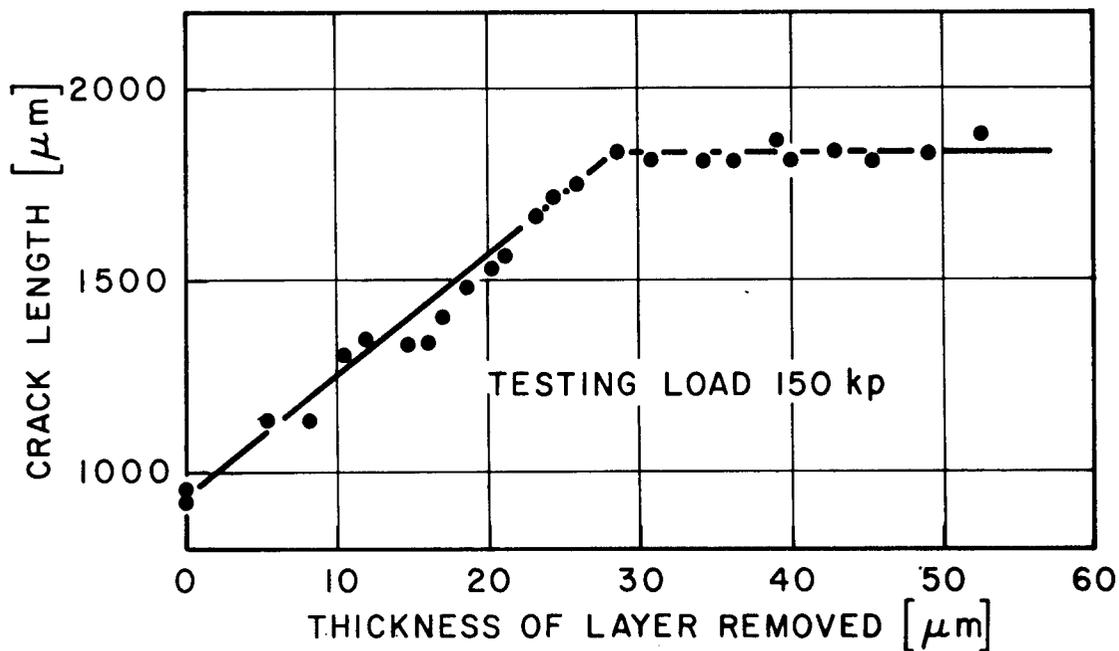


FIG. 4 CRACK LENGTH AS A FUNCTION OF THICKNESS OF DEFORMED LAYER REMOVED BY POLISHING.

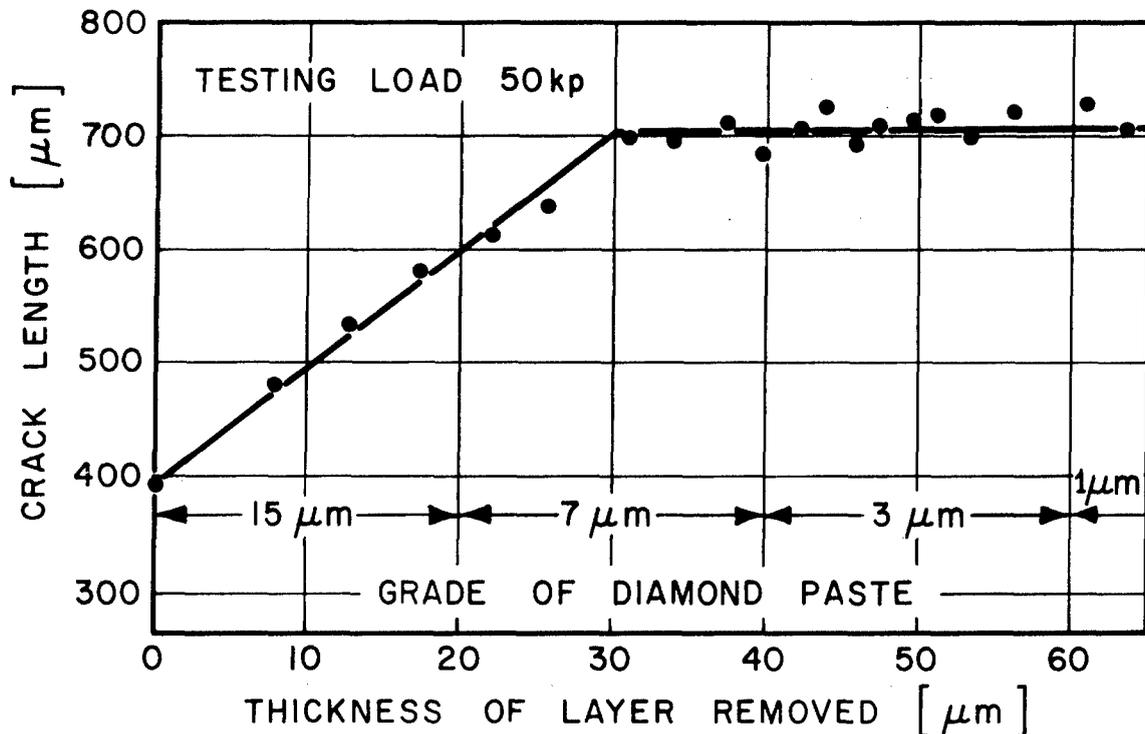


FIG. 5 CRACK LENGTH AS A FUNCTION OF THICKNESS OF DEFORMED LAYER REMOVED BY POLISHING WITH DIFFERENT GRADES OF DIAMOND PASTE.

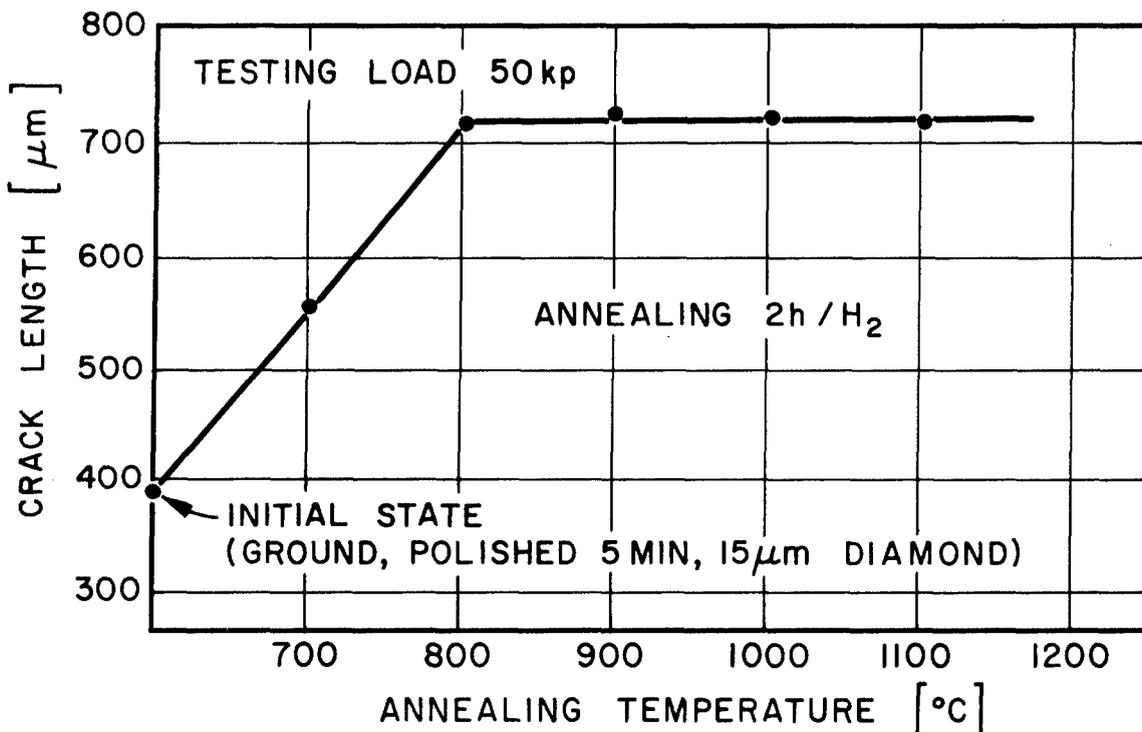


FIG. 6 CRACK LENGTH AS A FUNCTION OF ANNEALING TEMPERATURE.

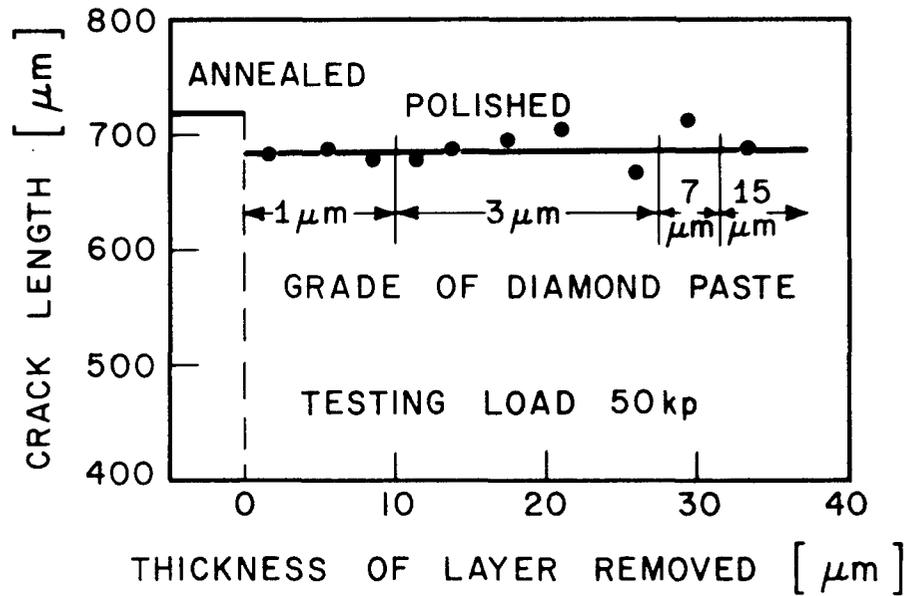


FIG. 7 EFFECT OF POLISHING AFTER ANNEALING

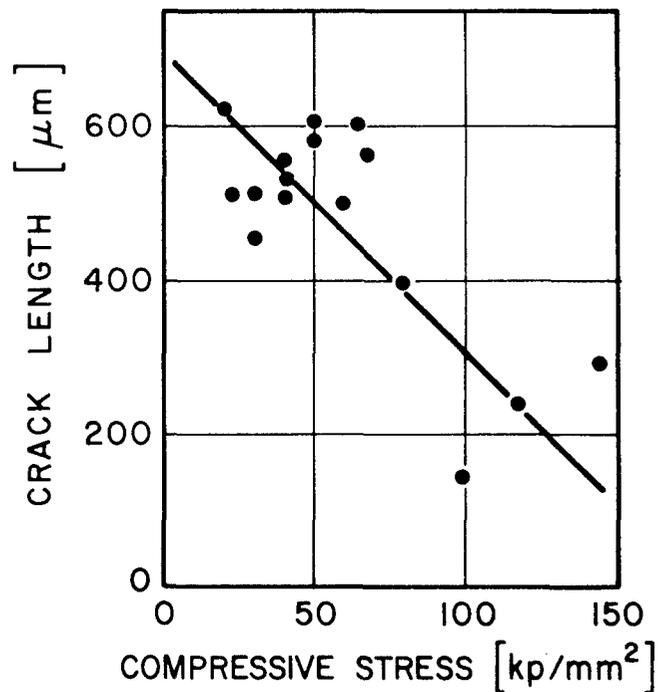


FIG. 8 CRACK LENGTH AS A FUNCTION OF COMPRESSIVE STRESS IN TUNGSTEN CARBIDE (AFTER ÖHMAN, PÄRNAMA AND PALMQVIST, (8))