PNL-SA-16927

VICKERS MICROINDENTATION TOUGHNESS OF A SINTERED SiC IN THE MEDIAN-CRACK REGIME

PNL-SA--16927

DE92 000225

by

Asish Ghosh, Zhuang Li*, Charles H. Henager, Jr.**,

Albert S. Kobayashi and Richard C. Bradt+++

College of Engineering

University of Washington

Seattle, WA 98195

Currently with

*Argonne National Laboratory, Argonne, IL 60439.

**~~Battelle~~ Pacific Northwest Laboratories, Richland, WA 99352

+++Mackay School of Mines, Univ. of Nevada-Reno, Reno, NV 89557-0047

ABSTRACT

The Vickers microindentation method for the determination of the fracture toughness of ceramics was investigated in the median crack regime for a sintered alpha SiC. The results are compared with fracture toughness measurements by conventional fracture mechanics techniques and also with the reported indentation toughness for the low-load Palmqvist crack regime. Indentation toughnesses in the median crack regime vary widely depending on the choice of the specific equation which is applied. The indentation toughnesses are also load (crack length) dependent. A decreasing R-curve trend results, in contradiction to the flat R-curve that has been observed with conventional fracture mechanics techniques. It is concluded that the Vickers microindentation method is not a reliable technique for the determination of the fracture toughness of ceramics in the median crack regime.

INTRODUCTION

The Vickers microindentation technique, where the lengths of the cracks emanating from the corners of a hardness indentation are utilized to determine the fracture toughness of a brittle material, was proposed about a decade ago[1]. In the ensuing time period, the method has attained considerable popularity because of its relative ease of application. Unlike many of the conventional fracture mechanics techniques, the Vickers microindentation method does not require extensive machining or sample preparation. Only a small specimen is required to determine the fracture toughness of the material. Furthermore, the "measured" crack lengths are often comparable in size to the strength-limiting critical flaws in structural ceramics, thus lending additional credibility to the toughness test results. Enthusiasm for the method has yielded a number of empirical equations that are applicable to the specific indentation crack patterns. The equations which have been proposed in the literature are based on either elastic or elastic/plastic analyses, and have been utilized by various researchers[2-13].

Figure 1 is a schematic of a Vickers impression and the accompanying surface crack pattern. There are two different types of underlying crack geometries which often form during indentation and appear just as that shown in Figure 1. For low test loads, the underlying crack geometry for many brittle materials is usually one of Palmqvist cracks. However, at higher indentation test loads the resulting geometry is one of median cracks. The two types are depicted schematically in Figure 2. The level of the test load for the Palmqvist crack to median crack transition has not been definitively specified, but it is dependent on the specific test material. One "rule of thumb" has been that Palmqvist cracks usually form when the (l/a) ratio is between 0.5 to 2.5 and that median cracks are generally formed when the (c/a) ratio is greater than about 2.5[2]. Since c, l and a are related, there obviously exists a transition region of some ambiguity.

Recent literature describing the use of the Vickers microindentation technique for fracture toughness measurements has created considerable concern relative to the application of the method. For example, Roberts and Warren[14] state that "great caution should be exercised when interpreting sizes of indentation cracks in terms of K_{IC}". Breval et al[15] have compared indentation toughness results for several sol-gel processed materials and observed peculiar inconsistencies. Using several of the various proposed equations for the same indentation test data, they report that the toughness values are dependent on the specific equation utilized for the calculations and are also ordered very differently from one test material to another, even for the same set of equations and test data. Miyoshi et al[16] have noted the same type of contradictions for testing silicon nitride ceramics, for which the fracture toughnesses were dependent on the indentation test load and also on the empirical equation utilized for calculations. For a ceria toughened zirconia, Matsumoto[17] reported that satisfactory results were obtained only with the indentation equation proposed by Anstis et al[8].

Indentation determined fracture toughnesses are seldom the same as those reported for measurements by conventional fracture mechanics techniques. The room temperature fracture toughness of dense, fine grain size sintered alpha SiC has been reported to be about 3 MPa $m^{1/2}$ as determined by conventional fracture mechanics techniques[18]. However, when the indentation technique has been applied to the same, or to very similar SiC materials, wide ranges of fracture toughness values have been obtained. Moussa et al[19] observed that the toughness is dependent on the test load and also on the specific equation used for calculations. Values determined by Moussa et al[19] ranged from 2.0 to 5.8 MPa $m^{1/2}$. Orange et al[20] similarly reported different values ranging from 2.6 to 3.3 MPa $m^{1/2}$ for two different equations at different indentation test loads. The indentation toughness value reported by Faber and Evans[21] for a sintered SiC was 2.3 MPa $m^{1/2}$, different from the value of 2.5 MPa $m^{1/2}$ that they obtained with the DCB technique. Srinivasan and Seshadri[22] measured 3.6 MPa $m^{1/2}$ for a similar sintered SiC using the indentation method, while Mohri et al[23] reported a toughness of only 2.1 MPa $m^{1/2}$. Similarly for an alumina, Lemaitre and Piller[24] have reported 4.18 MPa $m^{1/2}$ by the single edge precracked beam method and 3.44 MPa $m^{1/2}$ by the controlled microflaw method, but only 2.28 MPa $m^{1/2}$ by the Vickers microindentation method. These wide ranges of toughness are unacceptable for nearly all applications.

Applying the microindentation method to a dense sintered SiC in the Palmqvist crack regime, Li et al[25] have reported that the indentation toughnesses can vary by a factor of five and are load dependent as well as dependent on the specific equation which is used for calculations. They also observed that a number of the proposed indentation equations yielded a decreasing R-curve trend, contrary to the flat R-curve behaviour obtained by conventional techniques for the same SiC. Li et al[25] also noted that a substantial portion of the microhardness indentation impressions are simply not suitable for crack length measurements. They reported that the percentage of acceptable indentation crack patterns, those without chipping, extensive peripheral cracking, cracks emanating from the sides instead of the corners, or with obvious crack-branching decreased substantially with increasing indentation test load from 80% at 3N to only 64% at 10N.

As a consequence of these concerns regarding the Vickers microindentation toughness test method, this study, specifically addressing toughness measurements in the higher indentation load, median crack regime for a sintered dense alpha SiC, was undertaken. The goals were: (i) to monitor the frequency of occurrence of unacceptably flawed indentations; (ii) to evaluate each of the various proposed equations for indentation toughness calculations; (iii) to examine the effects of indentation test load and the resulting crack lengths; and (iv) to directly compare the toughness results with other similar fracture toughness measurements, including those previously published for the Palmqvist crack regime and those utilizing conventional fracture mechanics techniques for the same SiC material.

EXPERIMENTAL PROCEDURES

A commercial sintered alpha SiC* was chosen as the model material for study of the measurement of its fracture toughness by the Vickers microindentation technique in the high load, median crack regime. This particular SiC was chosen for several reasons, including: (i) extensive previous measurements by conventional fracture mechanics toughness test methods; (ii) the possession of a well documented, flat R-curve, indicative of a crack-length-independent fracture toughness; and (iii) the existence of the previously noted, extensive microindentation toughness study in the Palmqvist crack regime, that of Li et al[25]. The fracture toughness of this SiC was determined by conventional fracture mechanics methods utilizing large sharp cracks and has been reported to be 2.97 $\pm$ 0.54 MPa $m^{1/2}$[18]. The density is 3.16 g/cc and its Young's modulus is 430 GPa. The average grain size is about 10 µm, although an occasional larger grain can be observed within the microstructure. Figure 3 illustrates the SiC microstructure as a reflected light polished and etched section.

*Hexoloy,Carborundum Company, Niagara Falls, NY 14302

Samples of approximately 10mmX20mmX30mm bars were diamond polished through 1 μm to yield a mirror like surface finish for indentation. Individual Vickers indentations were made at 25, 35, 45 and 50 N loads for a loading time of 15 seconds each using a commercial microhardness testing machine**. The indentation and crack sizes were measured immediately after unloading, employing the microscope attached to the hardness tester. At each test load, 15 perfect Vickers indentations (i.e. those with an obviously symmetrical pyramidal impression and a well defined symmetrical crack pattern of four nearly equal length cracks originating from the indentation corners) were utilized to determine the hardness and to calculate the fracture toughness[26]. All indentations with the defects previously noted by Li et al[25] were considered unacceptable.

RESULTS AND DISCUSSION

The number of indentations actually required to achieve 15 perfect crack patterns was greater than 15 for each level of test load. Numerous indentations yielded unsymmetrical impressions and similarly unsatisfactory crack patterns with the presence of severe chipping or extensive crack branching. These flawed indentations are not considered to be acceptable for the toughness calculations. The indentation numbers as well as the number of perfect indentations for each test load are summarized in Table I. It is obvious that acceptable indentation crack patterns are only achieved for a portion of the indentations. A similar trend in this observation was

**Zwick 3212, East Windsor, CT.

previously reported for Palmqvist cracks by Li et al[25], as their percentage of unacceptable indentations also increased from 20% at 3N to 36% at 10N with increasing test loads, even though for much lower loads in the Palmqvist crack regime. In the present study the percentage of unacceptable indentations increased from 44% at the 25N test load to 57% at 50N. For test loads in excess of 50N, it was not possible to obtain any acceptable indentations, i.e. ones without one or more of the previously mentioned indentation-associated defects. The fact that a significant portion of the Vickers indentations yield unacceptable crack patterns is certainly not a mechanical test characteristic which inspires confidence in the indentation toughness measurement technique.

Average indentation sizes, as well as the measured crack lengths and their 95% confidence intervals are summarized in Table II. Figure 4, a plot of the crack lengths versus the indentation test loads, also includes the data of Li et al[25] for the low-test-load Palmqvist crack regime. For increasing test loads from 25 N to 50 N, the crack lengths increased as expected. This has also been reported for other similar SiC materials by other investigators, including Moussa et al[19] and Lankford[27]. Li et al[25] also observed an increase of crack lengths for test loads from 1N to 10N for the same SiC. When the P values of Li et al[25] are contrasted with the values in this study, the former are much lower, as might be expected when Palmqvist crack patterns and median crack patterns are compared.

The (c/a) ratios for the different test loads are also summarized in Table II. As the (c/a) ratio is always much greater than 2.5 for the test loads utilized in this study, the cracks emanating from the indentations are certain to be median cracks. In contrast, the Palmqvist cracks obtained by Li et al[25] had (l/a) ratios much less than 2.5 for the various loads. It is evident from Figure 4, that there exists a distinct difference in the c versus P relationships for cracks formed at test loads less than 10N and those formed at test loads greater than 25N. The transition from a Palmqvist crack system to one of median cracks occurs between the 10N and 25N test loads for this SiC.

Hardness values were obtained at all four test loads from the measurements of the indentation sizes using the standard Vickers hardness equation:

$$H_V = \frac{463.6\ P}{a^2} \qquad (1)$$

where H_V is the Vickers microhardness, P is the applied test load and a is the indentation diagonal length. The hardnesses summarized in Table II are in general agreement with the 27 GPa hardness value reported by Li et al[25]. However, these hardnesses are higher, by a factor of 1.5, than the true hardness value of 18.2 GPa for this SiC as determined by Li et al[25] using the Knoop microhardness and a related analysis. It is apparent that the formation of the extensive crack (median crack) patterns about these indentations results in an "artificially" high hardness result.

Fracture Toughnesses

Table III summarizes proposed equations which have been utilized to calculate the fracture toughness values using the Vickers microindentation technique. Li et al[25] have reviewed these equations and classified them into three categories: (i) those equations specifically for median cracks; (ii) those equations specifically for Palmqvist cracks; and (iii) curve-fitting expressions that have been applied to and are supposedly applicable to both Palmqvist and median crack systems. All of the equations are empirical in the sense that they require some form of constant adjustments.

Toughnesses for this SiC along with their 95% confidence intervals as calculated by using the equations summarized in Table III are listed in Table IV. These toughnesses vary from 1.50 to 5.69 MPa $m^{1/2}$, nearly a factor of four, depending on the test load and the equation. This wide range of toughnesses does, however, include the accepted fracture toughness value of 2.97 $\pm$ 0.54 MPa $m^{1/2}$ for this SiC as measured by conventional fracture mechanics techniques[18]. It is noteworthy, that for this same SiC, Li et al[25] also reported a similarly wide range of indentation toughnesses for the Palmqvist crack regime for the different test loads, as well as for the different equations, varying from 1.25 to 4.81 MPa $m^{1/2}$, also about a factor of four. This wide range of toughness values, depending on the test load and the specific equation, is also similar to the scatter reported by Moussa et al[19] for a SiC. Only for certain loads does the value of the toughness determined by the microindentation method agree with the fracture toughness as measured by conventional fracture mechanics techniques.

As some investigatiors may indiscriminately use the various equations for estimating the indentation toughness irrespective of the crack geometry, it is appropriate to consider these results for both the general curve fitting equations and also the Palmqvist crack equations. In Table IV, the fracture toughnesses obtained by using the general curve fitting equations and the Palmqvist crack equations are seen to vary from 4.25 to 5.69 MPa $m^{1/2}$ for the range of test loads and different proposed equations. These values are considerably higher than the accepted fracture toughness value. Considering that these are median cracks, it is not surprising that the Palmqvist crack equations do not yield reasonable toughness values. However, since the general curve fitting equations also yield toughness values that are much too high, it raises serious questions concerning their validity and applicability in the median crack regime for this SiC and other ceramic materials as well.

The seven median crack equations, M-1 through M-7, yield a wide range of toughnesses from 1.51 to 4.61 MPa $m^{1/2}$. For the results of the present study, only the equation proposed by Anstis et al (Eqn M-6)[8] yields fracture toughness values which are in satisfactory agreement with the accepted conventionally measured fracture toughness of 2.97 MPa $m^{1/2}$ for this SiC. However, that result is probably fortuitous, as even the Anstis et al[8] equation must be considered suspect as a consequence of its load (crack length) dependence of calculated toughness.

Crack Length Dependence of Toughness

Figure 5 illustrates the calculated toughnesses vs the applied test loads for all of the proposed median crack equations. This toughness versus test load plot is related to the crack length versus toughness, which is equivalent to an R-curve plot even though conventional R-curves are plotted as either K_{IR} or G_{IR} versus Δc. Except for equation M-1, all of the lines calculated using the median crack equations slope downwards, implying that this SiC exhibits a decreasing R-curve behaviour. This is not only impossible, but it is in obvious contradiction with the results from conventional fracture mechanics measurements, which have clearly revealed a flat R-curve for this SiC. These median crack equations do not only yield the incorrect fracture toughness values, but they also yield an incorrect R-curve trend. These equations must be considered highly questionable for the prediction of the toughness of brittle materials.

Figure 6 illustrates the toughness variation with test load (crack length) when attempting to apply the three curve-fitting equations and the two Palmqvist crack equations. The three curve fitting equations also indicate a decreasing R-curve trend, but the two Palmqvist crack equations suggest an increasing or rising R-curve trend. As this SiC has a flat R-curve, neither the three curve-fitting equations, nor the two

Palmqvist crack system equations correctly describe the R-curve trend for this material in the median crack regime.

Incorporating the True Hardness Concept

In their study of the Palmqvist crack regime for this SiC, Li et al[25] were able to salvage the experimental data and obtain reasonable fracture toughness results by incorporating the concept of the true hardness, H_o, into the Nihara-Morena-Hasselman Equation[2] in place of the measured hardness, H_v. The true hardness concept had a levelling effect on the rising R-curve trend observed in the Palmqvist crack regime to yield a load, or crack-length-independent toughness and thus a flat R-curve behaviour. The Niharra-Morena-Hasselman[2] equation then not only predicted the correct fracture toughness level, but also confirmed the flat R-curve behaviour observed experimentally by conventional fracture mechanics test methods.

Li et al[25] determined the true hardness for this SiC to be 18.2 GPa. When this hardness value is incorporated with the data for the median cracks, all but two of the results continue to deviate from the toughness value determined by conventional fracture mechanics methods. The toughness values still range from 1.32 to 4.73 MPa $m^{1/2}$ with only the Lawn and Fuller[5] and the Tanaka[9] equations yielding values in reasonable agreement with the conventionally-measured fracture toughness. Not only are the other fracture toughness values incorrect, but none of the R-curve trends are flat either. For these median crack systems, the two Palmqvist equations predict rising R-curves, while the three curve fitting equations and all of the median crack equations predict decreasing R-curves, including the Lawn and Fuller[5] and the Tanaka[9] equations. It is evident that incorporating the true hardness concept does not satisfactorily improve the experimental results for the median crack system as it has been demonstrated to do for the Palmqvist indentation crack system.

From these results it is evident for this SiC that the fracture toughness values which are calculated and the R-curve trends which are obtained using the microindentation toughness equations from the literature are not consistent with the results obtained by conventional fracture mechanics techniques. For those few instances when the values do agree with those from conventional fracture mechanics measurements, it seems to be fortuitous. These results clearly indicate that the Vickers microindentation toughness technique is not satisfactory for this SiC in the median crack regime. As this dense, fine grain size SiC is almost an ideal, single phase, non-phase-transforming, model, brittle, ceramic material, it should not be surprising, therefore, that the Vickers microindentation technique may not be satisfactory when applied to other types of ceramic materials either. This is probably why the numerous inconsistencies previously cited in the introduction have occurred.

Similarly extensive study is probably also required for other types of ceramic materials including multiphase microstructures and phase transforming ones to directly ascertain the validity of the Vickers microindentation technique for those ceramics. However, until those extensive studies are completed, any toughness values which are obtained and reported using the Vickers microindentation technique must be viewed with considerable skepticism and compared with measurements by the accepted, reliable conventional fracture mechanics methods before they are recognized to be a characteristic value.

SUMMARY AND CONCLUSIONS

The Vickers microindentation technique for the measurement of the fracture toughness was investigated in the median crack regime for a dense, fine grain size sintered alpha SiC. A large number of indentations were necessary to obtain a representative number of satisfactory crack patterns. About one-half of all of the indentations between the 25N-50N test loads were chipped, asymmetric or exhibited extensive peripheral cracking and crack branching and were therefore unacceptable for toughness measurements. The percentage of acceptable indentation crack patterns decreased with increasing test load, until for test loads above 50N, no satisfactory indentation crack patterns could be obtained.

Applying the various median crack equations yielded a wide range of fracture toughnesses, from 1.51 to 4.61 MPa $m^{1/2}$, depending on the applied test load and the specific equation which was utilized to calculate the fracture toughness for this SiC, which has a fracture toughness of 2.97

MPa $m^{1/2}$ as determined by conventional fracture mechanics test methods. The only equation which yielded the correct toughness value for this SiC was that of Anstis et al[8], but it also yielded a decreasing R-curve trend. It fact, all the median crack equations, except the one of Lawn and Swain[4], yield decreasing R-curves, while that one predicts a rising R-curve. When applying the median crack equations, not only are the fracture toughness values in error and widely scattered, but also the R-curve trends are incorrect for this SiC material.

The general curve-fitting equations and the Palmqvist crack equations yield fracture toughnesses which range from 4.25 to 5.69 MPa $m^{1/2}$, depending on the test load and the specific equation. These values are much higher than the conventionally determined fracture toughness value by a factor of 1.5 to 2. Not only are the microindentation fracture toughness values much too high, but the R-curve trends of the equations are different from the flat R-curve behaviour for this SiC.

Incorporation of the true hardness concept within the median crack toughess equations, as Li et al[25] did successfully for Palmqvist crack systems, does not improve the scatter of the fracture toughness values for median cracks. The results still depend on the test load and the specific equation. It is also significant that the incorrect R-curve trends are not corrected either.

Values of fracture toughness determined using the Vickers microindentation technique must be viewed only with substantial skepticism and should only be accepted with considerable reservation. The values determined by the technique should not be given any credibility and should be used only if they are validated by conventional fracture mechanics test methods

ACKNOWLEDGEMENTS

The authors would like to thank M.S. Srinivasan of Carborundum for providing the SiC material. The support of DOE under contract No.86A-00209C and NASA under Grant No. NAGW-199 are gratefully acknowledged.

REFERENCES

1) A.G. Evans and E.A. Charles, "Fracture Toughness Determinations by Indentation," J. Amer. Ceram. Soc., 59 [7-8] 371-372 (1976).

2) K. Niihara, R. Morena and D.P.H. Hasselman, "Evaluation of K_{Ic} of Brittle Solids by the Indentation Method with Low Crack-to-Indent Ratios," J. Mat. Sci. Lett., 1 13-16 (1982).

3) K. Niihara, "A Fracture Mechanics Analysis of Indentation-Induced Palmqvist Crack in Ceramics," J. Mat. Sci. Lett., 2 221-223 (1983).

4) B.R. Lawn and M.V. Swain, "Microfracture Beneath Point Indentations in Brittle Solids," J. Mat. Sci., 10 113-122 (1975).

5) B.R. Lawn and E.R. Fuller, "Equilibrium Penny-Like Cracks in Indentation Fracture," J. Mat. Sci., 10 2016-2024 (1975).

6) A.G. Evans and T.R. Wilshaw, "Quasi-Static Solid Particle Damage in Brittle Solids-I. Observations, Analysis and Implications," Acta Metal., 24 939-956 (1976).

7) B.R. Lawn, A.G. Evans, and D.B. Marshall, "Elastic/Plastic Indentation Damage in Ceramics: The Median/Radial Crack System," J. Amer. Ceram. Soc., 63 [9-10] 574-581 (1980).

8) G.R. Anstis, P. Chantikul, B.R. Lawn and D.B. Marshall, "A Critical Evaluation of Indentation Techniques for Measuring Fracture Toughness: I, Direct Crack Measurements," J. Amer. Ceram. Soc., 64 [9] 533-538 (1981).

9) K. Tanaka, "Elastic/Plastic Indentation Hardness and Indentation Fracture Toughness: The Inclusion Core Model," J. Mat. Sci., 22 1501-1508 (1987).

10) J.E. Blendell, "The Origins of Internal Stresses in Polycrystalline Alumina and Their Effects on Mechanical Properties," PhD Thesis, MIT, (1979).

11) A.G. Evans, "Fracture Toughness: The Role of Indentation Techniques," p 112-135 in. Fracture Mechanics Applied to Brittle Materials ASTM STP 678, Ed. S.W. Freiman, (1979).

12) J. Lankford, "Indentation Microfracture in the Palmqvist Crack Regime: Implications for Fracture Toughness Evaluation by the Indentation Method," J. Mater. Sci. Lett., 1 493-496 (1982).

13) D.K. Shetty, I.G. Wright, P.N. Mincer and A.H. Clauer, "Indentation Fracture of WC-Co Cermets," J. Mat. Sci., 20 1873-1882 (1985).

14)S.G. Roberts and P.D. Warren, "Flow and Fracture Patterns around Indentations in Brittle Single Crystals," pp 273-279 in Proc. Brit. Cer. Soc. 38 (1986).

15)E. Breval, G.C. Dodds and N.H. Macmillan, "The Hardness, Stiffness and Toughness of Diphasic Abrasive Materials Prepared by Sol-Gel Techniques," Mat. Res. Bull., 20 [4] 413-429 [1985].

16)T. Miyoshi, N. Sagawa and T. Sasa, "A Study on Evaluation of K_{IC} for Structural Ceramics," J. Jap. Soc. Mech. Eng., A, 51 [471] 2489-2497 (1985).

17)R.L.K. Matsumoto, "Evaluation of Fracture Toughness Determination Methods as Applied to Ceria Stabilized Tetragonal Zirconia Polycrystals," J. Amer. Ceram. Soc., 70 [12] C366-C368 (1987).

18)A. Ghosh, M.G. Jenkins, K.W. White, A.S. Kobayashi and R.C. Bradt, "Elevated Temperature Fracture Resistance of a Sintered Alpha Silicon Carbide," J. Amer. Ceram. Soc., 72 [2] 242-247 (1989)

19)R. Moussa, I. Coppolani and F. Osterstock, "Indentation Techniques Applied to Silicon Carbide," Proc. Brit. Ceram. Soc., 32 237-247 (1982).

20)G. Orange, H. Tanaka and G. Fantozzi, "Fracture Toughness of Pressureless Sintered SiC: A Comparison of K_{IC} Measurement Methods," Cer. Int., 13 (3) 159-165 (1987).

21)K.T. Faber and A.G. Evans, "Intergranular Crack Deflection Toughening of Silicon Carbide," J. Amer. Ceram. Soc., 66 (6) C94-C96 (1983).

22)M. Srinivasan and S.G. Seshadri, "Application of Single Edge Notched Beam and Indentation Techniques to Determine Fracture Toughness of Alpha Silicon Carbide," pg 46-68, Fracture Mechanics For Ceramics, Rocks and Concrete, ASTM STP 745 (1980).

23)M. Mohri, Y. Fujii and K. Yamada, "Effect of Microstructure on Mechanical Properties of SiC," paper # 214-B-388, presented at the annual meeting of the Amer. Cer. Soc., May 1-5, 1988, Cincinnati, Ohio.

24)P. Lemaitre and R. Piller, "Comparison of the Fracture Toughness of Alumina Measured by Three Different Methods," J. Mat. Sc. Lett., 7, 772-774 (1988).

25)Z. Li, A. Ghosh, A.S. Kobayashi and R.C. Bradt, "Indentation Fracture Toughness of Sintered SiC in the

Palmqvist Crack Regime," accepted for publication by the J. Amer. Ceram. Soc..

26) S. Palmqvist, "Metod Att Bestamma Segheten Hos Sproda Material, Sarskilt Hardmetaller," Jernlontorets Ann., 141 300-307 (1957).

27) J. Lankford, "Threshold Microfracture During Elastic-Plastic Indentation of Ceramics," J. Mater. Sci., 16 1177-1182 (1981).

LIST OF TABLES

TABLE I: SUMMARY OF NUMBER OF INDENTATIONS

TABLE II: SUMMARY OF RESULTS

TABLE III: EQUATIONS FOR CALCULATION OF K_{ic} VALUES FROM VICKERS INDENTATION CRACK SYSTEMS

TABLE IV: SUMMARY OF FRACTURE TOUGHNESS CALCULATIONS

TABLE V: SUMMARY OF INDENTATION FRACTURE TOUGHNESS VALUES FOR SiC

LIST OF FIGURES

FIGURE 1: SCHEMATIC OF VICKERS INDENT

FIGURE 2: SCHEMATIC OF A)PALMQVIST AND B)MEDIAN CRACK SYSTEM

FIGURE 3: ROOM TEMPERATURE MICROSTRUCTURE OF SINTERED ALPHA SILICON CARBIDE

FIGURE 4: CRACK LENGTH VERSUS TEST LOAD FOR PALMQVIST[22] AND MEDIAN CRACK SYSTEMS

FIGURE 5: FRACTURE TOUGHNESS VERSUS TEST LOAD USING MEDIAN CRACK EQUATIONS

FIGURE 6: FRACTURE TOUGHNESS VERSUS TEST LOAD USING PALMQVIST AND CURVE FITTING EQUATIONS

TABLE I

Summary of Numbers of Indentations

	25N	35N	45N	50N
No. Perfect Indents	15	15	15	15
Total No. of Indents	27	28	29	35
% Unacceptable	44%	46%	48%	57%

TABLE II

Summary of Results

	25N	35N	45N	50N
a (μm)	21.86 ± 0.26	25.06 ± 0.23	27.66 ± 0.72	29.84 ± 0.74
H_V (GPa)	23.82 ± 0.56	25.36 ± 0.41	26.95 ± 1.45	25.69 ± 1.30
c (μm)	65.46 ± 4.22	82.20 ± 3.00	100.56 ± 4.25	108.38 ± 4.29
(c/a) ratio	2.99	3.28	3.64	3.63

TABLE III

Equations for Calculation of K_{Ic} Values from Vickers Indentation Crack Systems

Median Crack Systems

M-1	Lawn and Swain [4]	$K_{Ic} = 0.0160\ (HP/C)^{1/2}$
M-2	Lawn and Fuller [5]	$K_{Ic} = 0.0726\ P/C^{3/2}$
M-3	Evans and Charles [1]	$K_{Ic} = 0.0742\ P/C^{3/2}$
M-4	Nihara et al [2]	$K_{Ic} = 0.0950\ P/C^{3/2}$
M-5	Lawn et al [7]	$K_{Ic} = 0.0558\ P/C^{3/2}$
M-6	Anstis et al [8]	$K_{Ic} = 0.0639\ P/C^{3/2}$
M-7	Tanaka [9]	$K_{Ic} = 0.0725\ P/C^{3/2}$

Palmqvist Crack Systems

P-1	Nihara et al [2]	$K_{Ic} = 0.0370\ (HP/l)^{1/2}$
P-2	Shetty et al [13]	$K_{Ic} = 0.0446\ (HP/l)^{1/2}$

Curve Fitting Equations

C-1	Blendell et al [10]	$K_{Ic} = 0.0285H^{0.6}E^{0.4}a^{0.5}\log(8.4a/c)$
C-2	Evans [11]	$K_{Ic} = H^{0.6}E^{0.4}a^{0.5}10^{y}$
C-3	Lankford [12]	$K_{Ic} = 0.0735H^{0.6}E^{0.4}a^{0.5}(c/a)^{-1.56}$

*The above equations are based on the material properties of the α-SiC in this study: E=430 GPa and ν= 0.22.

TABLE IV

Summary of Fracture Toughness Calculations

Equation	TEST LOAD 25N	35N	45N	50N
M-1	1.51 ± 0.04	1.65 ± 0.03	1.74 ± 0.05	1.73 ± 0.05
M-2	3.40 ± 0.26	3.37 ± 0.18	3.22 ± 0.20	3.19 ± 0.18
M-3	3.50 ± 0.26	3.48 ± 0.18	3.31 ± 0.20	3.29 ± 0.18
M-4	4.61 ± 0.37	4.46 ± 0.24	4.16 ± 0.29	4.20 ± 0.25
M-5	2.59 ± 0.21	2.48 ± 0.13	2.31 ± 0.17	2.34 ± 0.14
M-6	3.19 ± 0.26	3.06 ± 0.16	2.84 ± 0.21	2.88 ± 0.17
M-7	3.40 ± 0.26	3.37 ± 0.18	3.21 ± 0.20	3.18 ± 0.18
P-1	4.56 ± 0.18	4.74 ± 0.12	4.79 ± 0.14	4.84 ± 0.12
P-2	5.17 ± 0.19	5.51 ± 0.14	5.69 ± 0.17	5.64 ± 0.18
C-1	4.53 ± 0.25	4.59 ± 0.17	4.43 ± 0.23	4.48 ± 0.21
C-2	4.66 ± 0.32	4.60 ± 0.22	4.30 ± 0.29	4.35 ± 0.26
C-3	4.76 ± 0.39	4.58 ± 0.25	4.25 ± 0.31	4.29 ± 0.26

These fracture toughness values were calculated using hardness values measured by the Vicker's indenter at that specific load. Units are MPa $m^{1/2}$.

TABLE V

Summary of Indentation Fracture Toughness Values for SiC

Study	Fracture Toughness (MPa $m^{1/2}$)	Equation Used	Load Used
Moussa et al[19]	2.0 - 5.8	M-3, C-2, M-5, P-1, M-6	1-10N
Faber & Evans[21]	2.3	M-3	200-300N
Srinivasan and Seshadri[22]	3.6	C-2	-
Orange et al[20]	2.6 - 3.3	M-5, M-6	5N-300N
Mohri et al[23]	2.1	M-5	5N

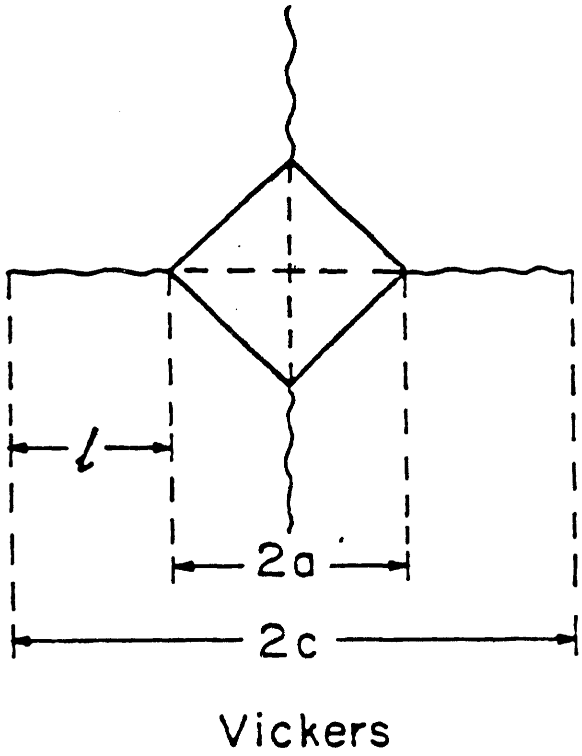

FIGURE 1: SCHEMATIC OF VICKERS INDENT

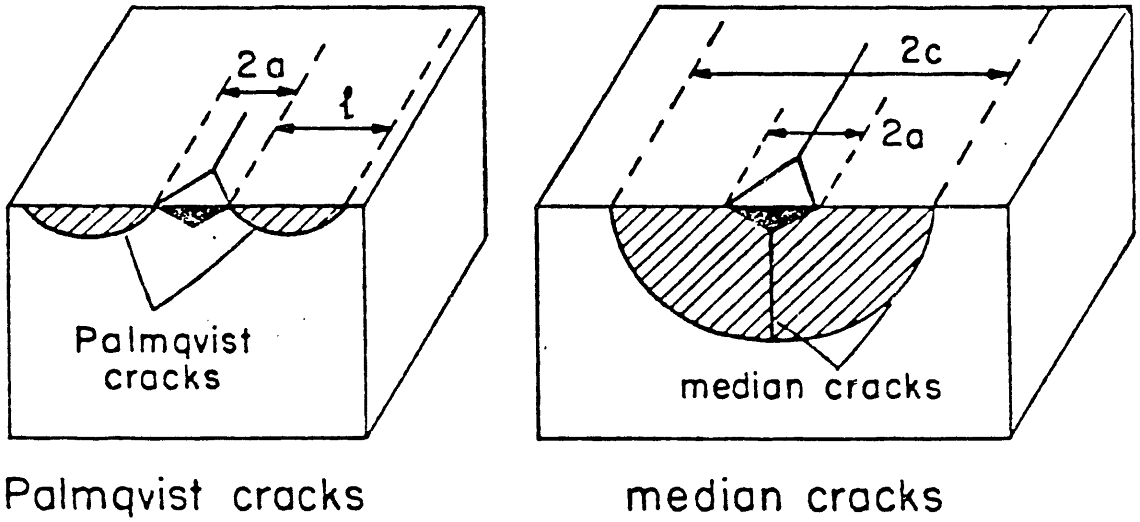

FIGURE 2: SCHEMATIC OF A)PALMQVIST AND B)MEDIAN CRACK SYSTEM

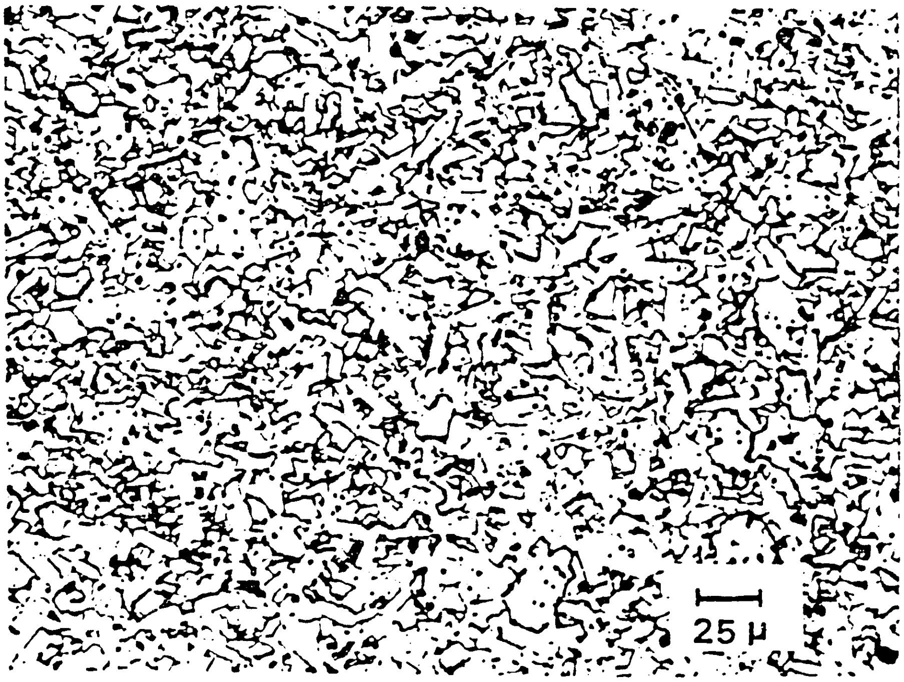

FIGURE 3: ROOM TEMPERATURE MICROSTRUCTURE OF SINTERED ALPHA SILICON CARBIDE

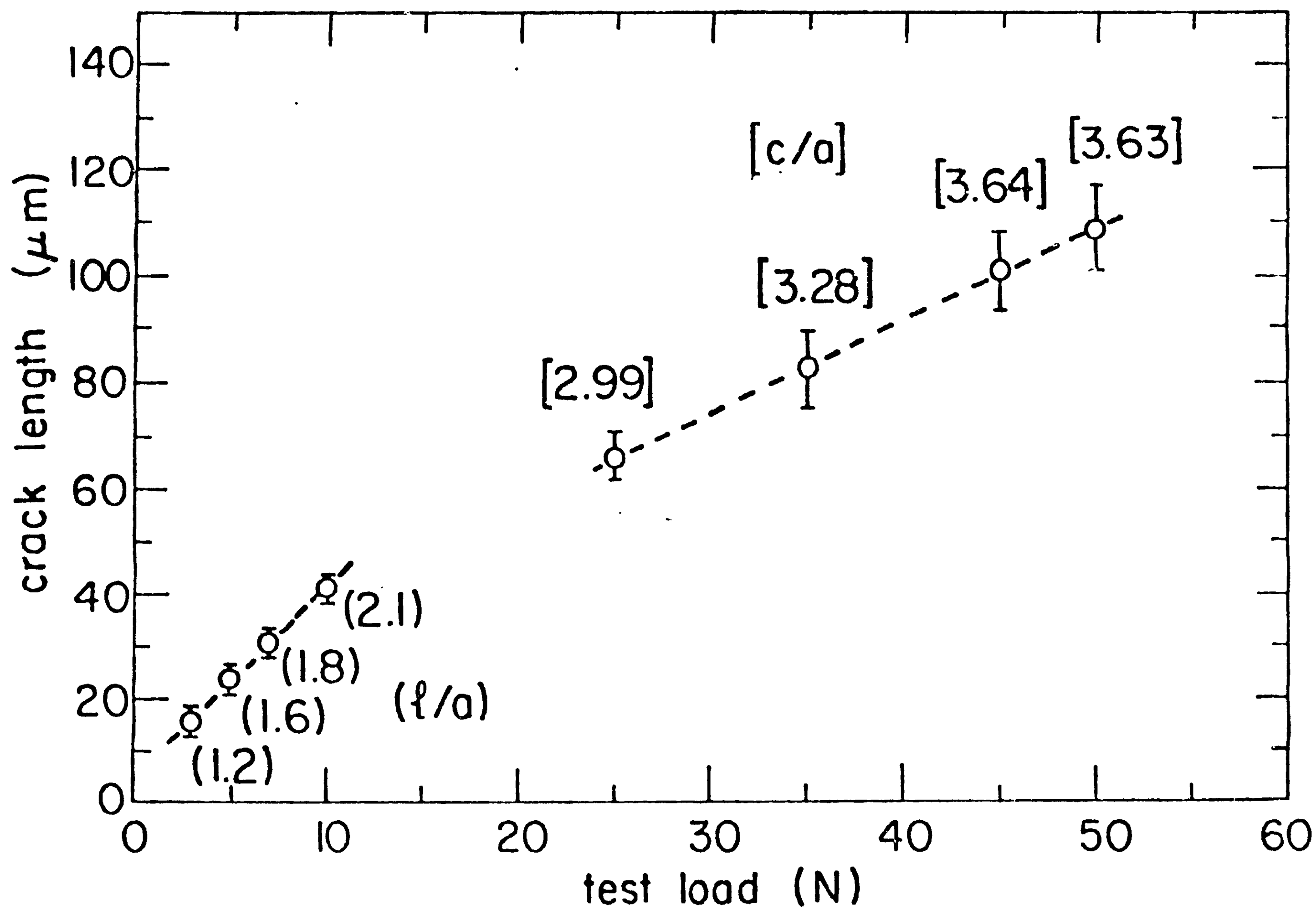

FIGURE 4: CRACK LENGTH VERSUS TEST LOAD FOR PALMQVIST[22] AND MEDIAN CRACK SYSTEMS

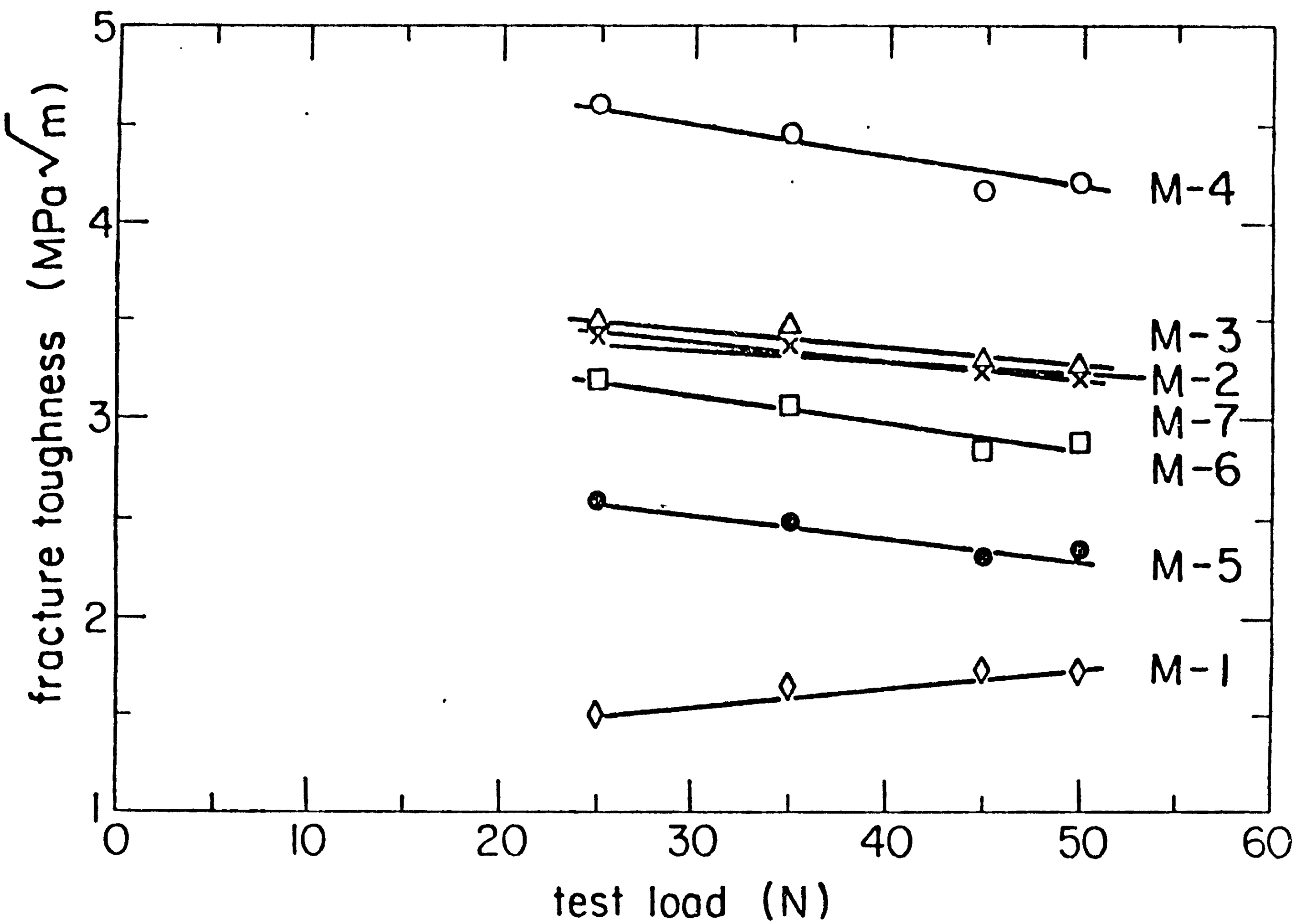

FIGURE 5: FRACTURE TOUGHNESS VERSUS TEST LOAD USING MEDIAN CRACK EQUATIONS

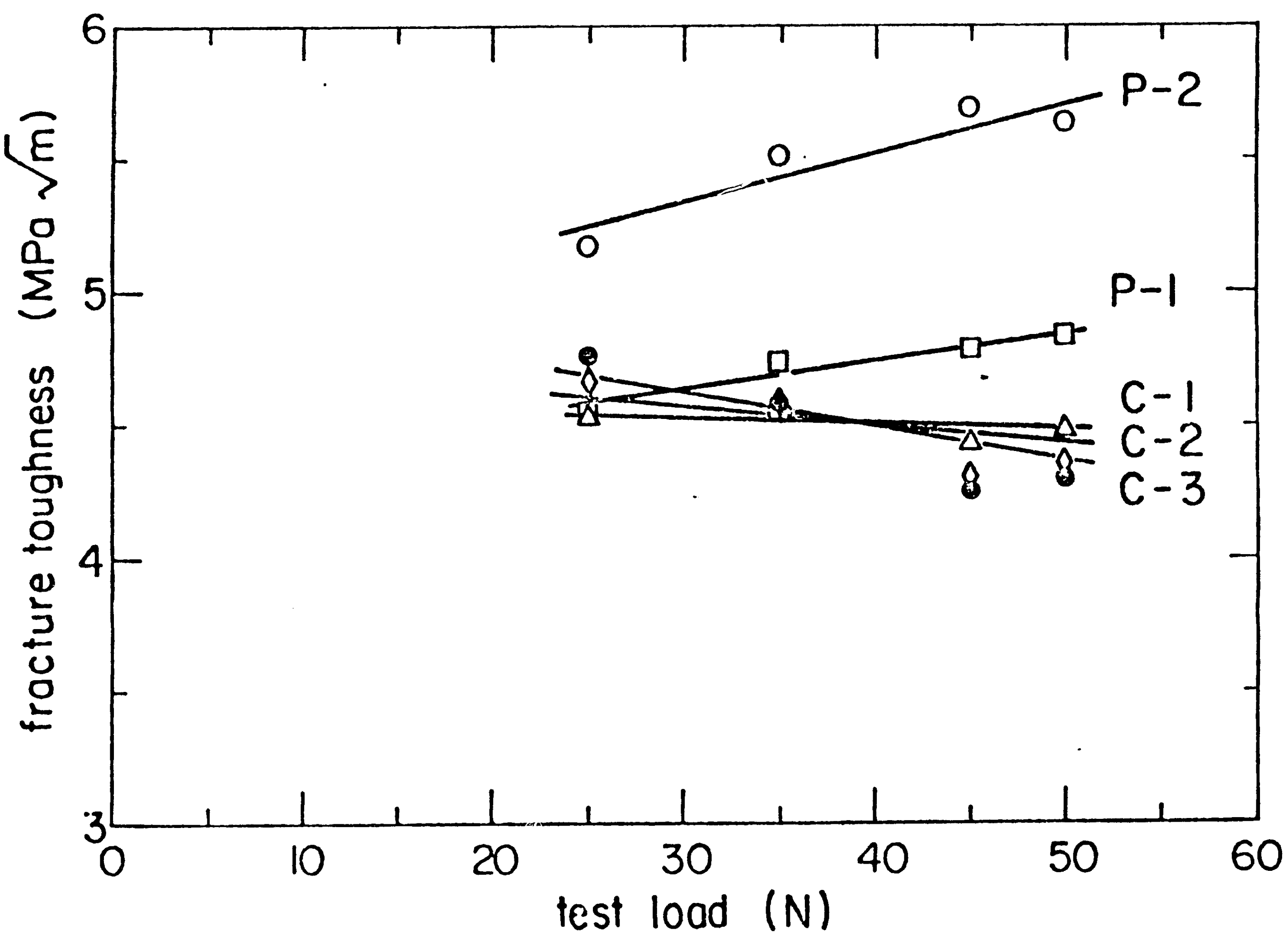

FIGURE 6: FRACTURE TOUGHNESS VERSUS TEST LOAD USING PALMQVIST AND CURVE FITTING EQUATIONS

END

DATE FILMED

10 / 29 / 91