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Modified indentation methods for fracture toughness determination of alumina ceramics

M. Szutkowska

The Institute of Metal Cutting, 37a Wrocławska Str., 30-011 Cracow, Poland

Abstract: In situ measurement of Vickers crack size as a function of applied stresses by means of indentation induced controlled Vickers crack growth in bending (ICVC) method for determination of fracture toughness is presented. For comparison purposes, both an indentation strength in bending (ISB) method and a direct crack measurement (DCM) method are also performed. A relation between a critical stress intensity factor K_{IC} and a ratio of cracks to indent size, is the basis for determination of the fracture toughness according to Lankford formula.

Keywords: Fracture toughness, Controlled crack growth, Alumina ceramics

1. INTRODUCTION

About ninety percent of the advanced ceramics today are used for electronic or related applications. The rest constitutes the structural ceramics in which the mechanical properties such as strength, fracture toughness, wear resistance, hardness etc. are of primary interest. Amongst the oxide ceramics, alumina is currently one of the most widely investigated structural ceramics. Due to the outstanding properties, such as: resistance to abrasion, wear and corrosion, high thermal stability, high electrical resistance, high hardness and relatively moderate price, alumina is already being used in significant amounts and applications as e.g. cutting tools. In spite of many attractive properties of alumina ceramics one of the primary drawback is their brittle nature, characterized by low fracture toughness. A vast number of experimental methods with specimen geometry is presented and adopted to determine the fracture toughness of alumina ceramics. These methods base on the three-point (3PB) or four-point bending (4PB) of the beam with notch such as: single edge notched beam SENB, single edge precracked beam SEPB, double torsion DT, double notched beam DCB and chevron notched beam CVNB. More popular methods deals with the extension of small Vickers indentation cracks under externally applied load in combination with the residual stress intensity factor. In general, two main crack systems i.e. median/radial and Palmqvist cracks derived from (irreversible) deformation zone, can be identified in alumina ceramics as a basis for determination of the fracture toughness, see Fig.1. In this paper the modified indentation techniques for measurement of fracture toughness by indentation induced controlled Vickers crack growth in bending (ICVC) is proposed. For comparison purposes, both an indentation strength in bending method (ISB) and direct crack measurement (DCM) method are also performed.

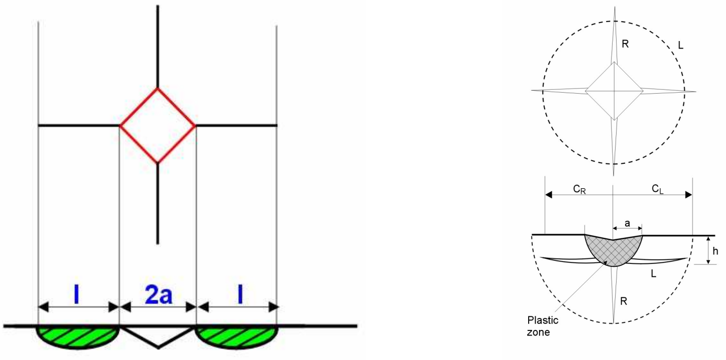

Figure1.Cracks formed during an indentation toughness test: a) Palmqvist crack; b) radial (R) and lateral (L) cracks

2. EXPERIMENTAL PROCEDURE

Commercial alumina powder type A16SG produced by the Alcoa is used to prepare the samples. The alumina powder contains the 90% of α-phase with 99.8% purity with mean particle size smaller than 0.5 μm. Green plates are uniaxially pressed at 50 MPa and then cold isostatically pressed at 250 MPa. The alumina ceramics are sintered in the high temperature electric furnace at the temperature 1923 K. The samples with Vickers indentation at 200 N load are subjected to three-point bending test. The universal testing machine Zwick 1446 with a rate v = 0.005 mm/min is used. After reloading of the specimen the crack length is measured with microscopical objective heads in horizontal configuration and then it is repeated up to failure. If the external stress field σ is applied to the indented specimen, the total stress intensity factor K_T is characterized by the sum of the residual stress intensity factor K_{RD} and the applied stress intensity factor K_A. The K_T can be expressed by equilibrium equation (2) [1]:

$$K_T = K_{RD} + K_A = \chi P c^{-3/2} + \psi \sigma c^{1/2} \quad (2)$$

where: P is the indentation load, c is the surface crack length, $\chi = \xi(E/H)^{1/2}$ is the dimensionless parameter characterizing the integrated effect of the residual indentation stress field over the half-penny crack, E is the Young's modulus, H is the hardness, ψ is the dimensionless crack geometry factor, σ is the applied stress.

The regime of stable crack extension between the initial crack length c_o and instability crack length c_m allows to obtain an experimental crack extension curve and to get the χ and the K_{IC} values. Equation (4) can be rearranged into (3):

$$\psi \sigma c^2 / P = K_{IC} c^{3/2} / P - \chi \quad (3)$$

The terms $X = c^{3/2} / P$ and $Y = \psi \sigma c^2 / P$ can be determined experimentally. Thus the K_{IC} and residual stress factor χ are obtained from the slope and the intercept of the interpolating function respectively. The coefficient ψ can be calculated from equation given in [1]. In case of the ISB method the specimen with Vickers cracks is loaded at the higher speed (1 mm/min) and K_{IC} is evaluated during unstable crack growth. Fracture toughness is calculated from simple formula (4) proposed by Chantikul [2] for the median/radial shape of cracks:

$$K_{IC} = 0.59(E/H)^{1/8} (\sigma P^{1/3})^{3/4} \quad (4)$$

where: σ is the failure stress, P is the loading force of Vickers hardness indenter

A correlation between the fracture toughness and ratio of cracks to indent size is expressed according to selected equations proposed by Lankford [4]. In this case, relation between the fracture toughness and the ratio of cracks to indent size is expressed by the equation (5):

$$K_{IC} = 0.142\,(Ha^{0.5})\,(E\phi/H)^{0.4}\,(c/a)^{-1.56}/\phi \qquad (5)$$

where: K_{IC} is the critical stress intensity factor, ϕ is the constrain factor, H is the Vickers hardness, E is the Young's modulus, c is the sum of half of indent diagonal and length of crack, a-half of indent diagonal.

3. RESULTS AND DISCUSSION

Data from more than fifteen measurement points determined by indentation induced controlled Vickers crack growth in bending methods (ICVC) allow to obtain plots of parameter $Y= \psi\,\sigma\,c^2/P$ versus $X=c^{3/2}/P$ for alumina ceramics, see Fig.5. The K_{IC} is evaluated from the slope of strain line. Residual stress factor χ is estimated from the intersection of the interpolating function with (Y) axis.

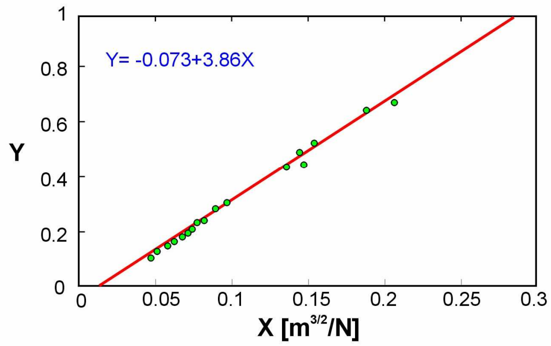

Figure2. Relation of $X=c^{3/2}/P$ and $Y= \psi\,\sigma\,c^2/P$ for alumina ceramics Al_2O_3

Results of the fracture toughness K_{IC} measured by means of indentation induced controlled Vickers crack growth in bending (ICVC) and indentation strength in bending (ISB) are presented in Table 1.

Table 1.
The fracture toughness K_{IC} obtained by following methods: the indentation induced controlled Vickers crack growth in bending (ICVC), the indentation strength in bending (ISB), direct crack measurement (DCM) and residual stress factor χ

Material	Fracture toughness K_{Ic} (ICVC) $[MPam^{1/2}]$	Fracture toughness K_{Ic} (ISB) $[MPam^{1/2}]$	Fracture toughness K_{Ic} (DCM) $[MPam^{1/2}]$	Residual stress factor χ
Al_2O_3	3.86±0.07	3.87±0.30	3.98±0.30	0.073± 0.008

The fracture toughness K_{IC} obtained by three point bending of the specimen with Vickers crack during its unstable growth (ISB method) and indentation induced controlled Vickers crack growth in bending (ICVC method) don't indicate difference. Difference (about 3%) of fracture toughness K_{IC} evaluated by direct crack measurement (DCM) method is not significant in comparison with the K_{IC} determined according to ICVC and ISB methods. The examples of crack's profiles in the alumina ceramics Al_2O_3 are illustrated in Figure 3. Corresponding SEM micrograph of the alumina ceramics Al_2O_3 confirms the both intergranular and transgranular character of crack path (Fig.3ab).

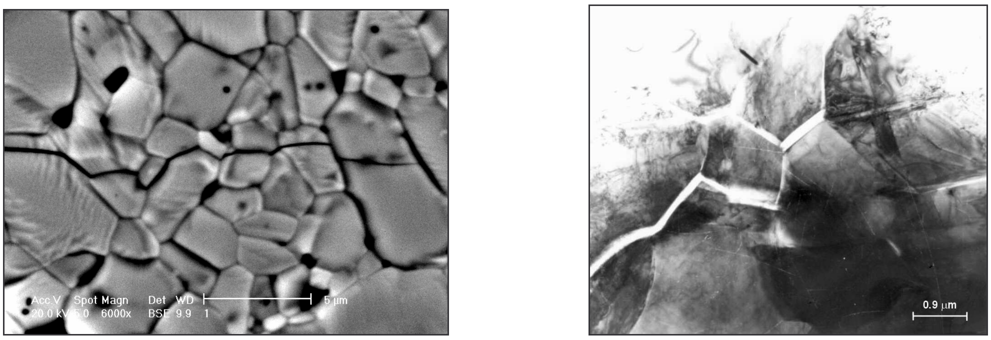

Figure 3. Microstructure of alumina ceramics Al_2O_3 with different Vickers crack profile: a) SEM image of intergranular and transgranular character of crack path, b) TEM image of bridging crack

The nearest to conventional fracture toughness (SENB specimen) is K_{IC} determined by Lankford's equation (5) [3]. Structure of the alumina ceramics in a zone adjacent to the Vickers indentation produced at 98.1 N loading force beneath the indentation tip was observed on the cross-section by means of transmission electron microscopy (TEM). Effect of such loading force generates deformation by dislocation slip and microtwinnings. Examples of TEM micrographs in the zone beneath the Vickers indenter tip, with effect of bridging crack are presented in Fig.3b.

4. CONCLUSIONS

1. Fracture toughness determined according to different methods: indentation induced controlled Vickers crack growth in bending (ICVC), indentation strength in bending (ISB) and direct crack measurement (DCM) does not indicate significant difference.
2. The calculations and own measurements allow verification and evaluation of the usefulness of the proposed methods in determining the K_{IC} for the most representative cutting tool ceramics.
3. The indentation technique has certain advantages compared with the conventional method because the experimental procedure is straightforward, involving minimal specimen preparation and small amount of material.

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