

THE COMPELLING CASE FOR INDENTATION AS A FUNCTIONAL EXPLORATORY AND CHARACTERIZATION TOOL

David B. Marshall,¹ Robert F. Cook,² Nitin P. Padture,³ Michelle L. Oyen,⁴
Antonia Pajares,⁵ Jodie E. Bradby,⁶ Ivar E. Reimanis,⁷ Rajan Tandon,⁸
Trevor F. Page,⁹ George M. Pharr,¹⁰ Brian R Lawn^{2,*}

¹ *Teledyne Scientific Co, Thousand Oaks CA 91360, USA*

² *Materials Measurement Laboratory, National Institute of Standards and Technology,
Gaithersburg MD 20899, USA*

³ *School of Engineering, Brown University, Providence RI 02912, USA*

⁴ *Department of Engineering, Cambridge University, Cambridge CB2 1PZ, UK*

⁵ *Departamento de Ingeniería Mecánica, Energética y de los Materiales, Universidad de
Extremadura, 06006 Badajoz, Spain*

⁶ *Research School of Physics and Engineering, Australian National University, Canberra ACT
2601, Australia*

⁷ *Department of Metallurgical and Materials Engineering, Colorado School of Mines, Golden
CO 80401, USA*

⁸ *Analytical Technologies, Sandia National Laboratories, Albuquerque NM 87185, USA*

⁹ *School of Chemical Engineering and Advanced Materials, Newcastle University, Newcastle
Upon Tyne NE1 7RU, UK*

¹⁰ *Department of Materials Science and Engineering, University of Tennessee, Knoxville TN
37996, USA; and Oak Ridge National Laboratories, Oak Ridge, TN 37831, USA*

The utility of indentation testing for characterizing a wide range of mechanical properties of brittle materials is highlighted in light of recent articles questioning its validity, specifically in relation to the measurement of toughness. Contrary to assertion by some critics, indentation fracture theory is fundamentally founded in Griffith–Irwin fracture mechanics, based on model crack systems evolving within inhomogeneous but well-documented elastic and elastic–plastic contact stress fields. Notwithstanding some numerical uncertainty in associated stress intensity factor relations, the technique remains an unrivalled quick, convenient and economical means for comparative, site-specific toughness evaluation. Most importantly, indentation patterns are unique fingerprints of mechanical behavior and thereby afford a powerful functional tool for exploring the richness of material diversity. At the same time, it is cautioned that unconditional usage without due attention to the

conformation of the indentation patterns can lead to overstated toughness values. Limitations of an alternative, more engineering approach to fracture evaluation, that of propagating a pre-crack through a 'standard' machined specimen, are also outlined. Misconceptions in the critical literature concerning the fundamental nature of crack equilibrium and stability within contact and other inhomogeneous stress fields are discussed.

* Corresponding author: brianlawn@gmail.com

I. Introduction

Since Hertz,¹ indentation testing has assumed a preeminent place as an exploratory and characterization research tool for the mechanical evaluation of ceramics and other brittle materials, particularly in the context of fracture. It has served as a model system for analyzing contact-induced cracks and other strength-degrading damage in a wide array of practical engineering applications—bearings, semiconductor devices and panels, windscreens and laminates, small devices and microelectromechanical systems, scratch-resistant films and high-temperature coatings, layer structures and composites, teeth and bone, implants and other biomaterials, and even the fashioning of ancient tools. The history of crack evolution in inhomogeneous but generally well-documented contact stress fields, surveyed in several articles extending back almost half a century,²⁻⁸ is firmly rooted in fundamental fracture mechanics principles. The methodology includes testing with 'blunt' (sphere, cylinder) and 'sharp' (Vickers, Knoop, Berkovich, cube corner) indenters. It is demonstrably the simplest, most economical and versatile of all mechanical testing protocols. It provides a powerful basis for investigating many materials topic areas, of which the following are just some examples:

- (i) Simple, rapid method of toughness measurement
- (ii) Critical contact force analyses, soundly based in Griffith energy-balance concept
- (iii) Elucidation of intrinsic mechanisms of brittle crack initiation and propagation
- (iv) *In situ* monitoring, affording rare insight into flaw evolution processes
- (v) Evaluation of local and macroscopic residual stresses
- (vi) Quantification of environmental effects and crack kinetics
- (vii) Role of plasticity (quasiplasticity) in ceramics, quantification of brittleness
- (viii) Large range of indentation loads, providing a bridge between short and long cracks

- (ix) Probing crack evolution at the microstructural level
- (x) Nanoindentation, automation of hardness and modulus evaluation
- (xi) Indentation creep, viscoelastic properties
- (xii) Phase transformations at ultra-high pressures
- (xiii) Point-to-point property mapping
- (xiv) Insight into strength degradation in relation to microcontact flaws
- (xv) Contact fatigue mechanisms
- (xvi) Framework for theories of wear and erosion
- (xvii) Nanomechanics, simulation of contacts and property evaluation in small-scale samples and devices
- (xviii) Mechanical properties of thin films, coatings and layer structures
- (xix) Properties of grain boundaries and interfaces in composites
- (xx) Rigorous fracture mechanics analysis of edge chipping
- (xxi) Biomechanics, fracture in shells, implants, teeth, bone
- (xxii) Site-specific evaluation of local in-service damage in engineering components

An article by Quinn & Bradt ⁹ has openly questioned the veracity of the indentation methodology, specifically as a measure of toughness or any like crack resistance parameter, and advocates discontinued usage. Others have sounded a similar call. ¹⁰⁻¹² The core of the claim is that indentation is limited by uncertainty in numerical coefficients and exponents in representative toughness equations. It is implied that contact stress fields are too complex and insufficiently well defined for accurate solutions to fracture evolution, and that the physics of the underlying fracture processes are not rigorously modeled. There is the assertion that toughness is defined by a critical condition where cracks begin to undergo catastrophic propagation and, by association, that crack resistance is fundamentally different in unstable and stable propagation states. It is also argued that different materials behave in widely different ways, with attendant

variations in crack pattern, implying a lack of universality in the indentation methodology. The disapproval is underpinned by a quest to measure a single engineering toughness parameter, K_{IC} , using reliable and traceable 'standard' test specimens with pre-cracks in machined specimens.^{9,10} The danger is that the value of indentation testing as a broad-based diagnostic materials characterization tool be derailed by questionable concerns about numerical accuracy.

We submit that the above perceived issues are misleading in at least three major aspects. First, it is widely overlooked that all these issues are in fact clearly outlined and discussed in depth in the original studies, especially in the article by Anstis *et al.*¹³ Indentation, as with all testing methods, has its caveats and limitations, often neglected by the casual user, leading to overstated claims concerning toughness properties. But this in no way detracts from the general usefulness of the methodology. Second, the assertion regarding the lack of rigor in the indentation fracture analyses is incorrect. Elastic and elastic-plastic contact fields beneath blunt and sharp indenters are in fact well defined and documented in classic texts and articles,¹⁴⁻¹⁶ and analyses of the evolution of cracks within these fields are based on rigorous Griffith-Irwin equilibrium fracture mechanics, in accord with the first law of thermodynamics (Panel A).^{6,17} Third, specification of a single toughness number from a standardized pre-crack test is restrictive. For instance, it precludes measurement at the microstructural scale where crack initiation and growth are determined. Further, many brittle materials exhibit toughness properties that are dependent on crack length and history (*R*-curves), where a unique toughness value is meaningless. Proper characterization of mechanical properties demands test protocols that address the questions at issue, not just provide a number.

The present article is presented as a case for usage of indentation as an indispensable tool for exploring material behavior, and for providing a firm basis for modeling a range of practical properties. In many instances indentation is the most practical way to explore material behavior at the microstructural level. It is often the *only*

way to probe small-scale specimens and components, e.g. modern microelectronic and micromechanical systems, where 'bulk' properties may no longer apply. We maintain that the variations in indentation responses alluded to above provide uniquely visual and quantitative 'fingerprints' of a rich material diversity, over a wide range of crack dimensions. Select examples are given in Figs. 1 and 2. Accordingly, we critique the methodologies used to measure toughness and other material properties, both by indentation and from 'standard' specimens, and argue that any limitations of indentation testing are greatly outweighed by its countless virtues. We point out several misconceptions and misleading assertions in some of the detracting articles, primarily the rejection of fundamental mechanics principles in favor of empirical fracture criteria.

Figures 1 and 2 here

Panel A. Fundamental basis of fracture mechanics

Modern fracture mechanics begins with the energy-balance concept of Griffith, with subsequent expression in terms of stress intensity factor terminology by Irwin.^{6,17-19} The Griffith energy balance condition for fracture is anchored in the first law of thermodynamics. According to Griffith, a crack in equilibrium is on the verge of extension when $G = R$, where G is mechanical energy release rate and R is crack resistance. In ideally brittle materials, R is twice the reversible surface energy. In terms of Irwin stress intensity factor terminology, equilibrium is stated as $K_I = K_{IC}$, with subscript I denoting mode I. Again in ideal brittle materials, K_{IC} identifies with a single-valued toughness T . These two terminologies are equivalent, linked by relations of the form $G = K_I^2/E$, $R = T^2/E$, with E Young's modulus.

Equilibrium can be unstable or stable, depending on whether G or K increases or decreases with crack length c , i.e. by the sign of dK/dc .^{6,20} The empirical pre-Griffith notion of fracture as attainment of some critical stress, either applied externally or operative at a crack tip,^{10,21} is oversimplistic and restrictive. Kinetic states, manifested as a crack velocity function $v(G)$ or $v(K)$ in the domain $G < R$, $K < T$, ensue when moisture or some other reactive environment diminishes the effective crack resistance, leading to subcritical crack extension at a specific rate.²²

For non-ideal brittle materials, toughness can be a function of crack length and history, so-called R -curve behavior.⁶ This applies to ceramics with large-grain, heterogeneous microstructures, especially those with weak internal interfaces and inbuilt local residual stresses. In that case the crack extension condition is generalized to $G = R_0 + R_S$, $K_I = T_0 + T_S$, where R_0 and T_0 are short-crack crack resistance quantities and R_S and T_S are crack shielding quantities from microstructural sources (bridging, phase transformation, microcracking, etc).

II. Brief History

(1) *Blunt indenters*

Cracks from concentrated loading beneath hard spherical indenters are the longest studied examples of fracture in inhomogeneous stress fields.^{1,2,23,33} The prototypical case is the growth of a cone crack within the Hertzian elastic stress field from contact at load P on a flat surface with a sphere of radius r (Fig. 1a). The Hertzian stress field solutions are explicit and exact for elastically isotropic solids.¹ Interest in the Hertzian fracture problem was aroused over a century ago by Auerbach, who observed experimentally that the critical load for cone pop-in satisfies $P_C \propto r$ (Auerbach's law).³³ Such a relation is at odds with $P_C \propto r^2$ derived from the notion that fracture should initiate from a critical flaw when the maximum tensile stress outside the Hertzian contact equals the bulk strength. This seemingly paradoxical discrepancy highlighted the inadequacy of simplistic critical stress criteria for predicting the onset of fracture in non-uniform stress fields. Subsequent analysis of crack growth within the Hertzian field using Griffith–Irwin mechanics showed that a shallow ring crack first forms from a surface flaw and then grows stably downward within a rapidly diminishing tensile field before popping into a full cone at the critical load.^{2,7} That analysis produces a rigorous validation of Auerbach's law

$$P_C = ArR = ArT^2/E \quad (1)$$

with A a dimensionless constant, R crack resistance, T toughness and E Young's modulus. On loading beyond the critical point, the fully developed cone crack first arrests and then propagates stably at $P > P_C$ according to the relation of the form^{23,34}

$$P/c^{3/2} = B(RE)^{1/2} = BT \quad (2)$$

with c a characteristic crack size and B another dimensionless constant. Note the appearance of toughness terms in eqns. 1 and 2, foreshadowing later relations for sharp indenters.

Despite a wealth of compelling evidence supporting the formal Griffith–Irwin derivation of eqn. 1, the simplistic notion that unstable fracture always occurs at some maximum stress has proved hard to shake. Evaluations of the maximum tensile stresses at cone crack initiation at the circumference of the Hertzian contact can be more than an order of magnitude greater than independently measured flexural strengths.^{25,35} Moreover, these maximum tensile stresses increase as the sphere size diminishes, i.e. there is an intrinsic size effect. Original attempts to account for this size effect invoked flaw statistics, using an argument that smaller indenters sample a smaller surface area and therefore stand a reduced chance of locating a critical flaw, with corresponding increase in stress level. While that explanation may apply to pristine surfaces with widely dispersed ultra-small (submicrometer) flaws,³⁶ where stress gradients are minimal, its unconditional use was discredited almost half a century ago by cone-crack tests on glass surfaces with controlled flaw populations.³⁵ Nevertheless, recent studies have chosen to revert to such empirical explanations, without attempt to identify the underlying mechanics of ring–cone cracking,¹² thereby ignoring a long history of formal indentation theory.

(2) *Sharp indenters*

As indicated above, Hertzian cone fracture is an important forerunner to more widely adopted sharp indenter tests with fixed-profile Vickers, cube-corner and Berkovich geometries (Fig. 2a).⁸ A major advantage of sharp indenters is that they enable straightforward measurement of radially extending cracks on the specimen surface. Sharp indenters are favored because of their simplicity, economy and versatility in routine laboratory testing. The stress field is elastic–plastic,³⁷ with cracks initiating

within and propagating from a near-hemispherical plastic zone immediately beneath the contact. The critical load to initiate radial cracks has the form ^{8,38,39}

$$P_C = CH(H/E)^2(T/H)^4 \quad (3)$$

where H is hardness and C is another dimensionless coefficient. The use of sharp indenters to measure toughness was foreshadowed by Palmqvist ⁴⁰ and Evans & Charles ⁴¹ and subsequently developed more rigorously using an 'expanding cavity' model for the elastic-plastic field.³⁹ A formal solution for the size of well-developed radial crack traces at $P > P_C$ is given by Anstis *et al.*¹³

$$P/c^{3/2} = (1/\xi)(H/E)^{1/2}T \quad (4)$$

This last equation is the most extensively used of indentation toughness relations, and is the one that has evoked the bulk of the criticism. It has several variants,⁴²⁻⁴⁵ principally in the value of coefficient ξ but also in the H/E exponent. Another variant employs direct measurement of crack-opening displacements.⁴⁶ In addition to radial-median cracks, shallow Palmqvist, subsurface lateral and (incomplete) cone or ring cracks add to the fracture multiplicity (Fig. 2a). Potential complications from non-ideal crack geometries and interactions are subsumed into the coefficient ξ .⁴⁷

A feature of eqn. 4 is that it can cover a wide range of contact loads, over 4 orders of magnitude in well-behaved materials, providing a bridge between short-crack and long-crack behavior.^{6,17} The range can be extended downward at the low-load end by using indenters with greater acuity, including cube-corners.⁴⁸⁻⁵² This takes us into the domain of nanoindentation, with all the benefits of automation ⁵³ and property mapping.⁵⁴ Small-scale indentation is unique in the way it facilitates elucidation of crack interactions with microstructural features, such as grain boundaries, interfaces, and second

phases.^{31,55,56} It is also being extended to viscoelastic materials, including biological tissue.⁵⁷ Such information has aided enormously in the design and synthesis of more fracture resistant materials.

III. Indentations as Fingerprints—Exploring Material Diversity

It has been argued that different materials have a spectrum of mechanical behaviors, and that consequent variations in indentation response conspire against an all-encompassing closed-form toughness equation.⁹ The challenges presented by this diversity in behavior, not only *between* different classes of brittle solids but also *within* a given class, are part of what gives materials science its charm. Generally, materials have to be selected and tailored individually for specific applications, and testing protocols need to be chosen to reflect each application. This diversity is nowhere better revealed than in indentation damage patterns, such as those in Figs. 1 and 2. Indentations are valuable 'fingerprints', elucidating a rich tapestry of material behavior.^{8,58}

Consider blunt indenters first. A near-axisymmetric surface ring crack can immediately confirm that a material is isotropic, as in silica glass (Fig. 1b),^{2,23} or anisotropic with preferred cleavage planes, as in monocrystalline diamond (Fig. 1c).⁵⁹ However, the classic Hertzian fracture analysis, predicated on a fully elastic contact field, is satisfied only in a select range of highly brittle solids. Softer and more heterogeneous ceramics, those with *R*-curve behavior (Panel A), may deform irreversibly beneath the indenter before fracture occurs: compare the (half-) surface traces in a fine-grain silicon nitride with its coarse-grain counterpart (Fig. 1d).²⁵ The residual impression in the latter case is due to local shear-driven breakdown of weak internal interfaces within the microstructure (quasiplasticity). The condition for exceeding the yield stress is $P_Y \propto r^2$ which, in relation to the Auerbach condition $P_C \propto r$ for cone crack initiation, means that plasticity is favored by small spheres,⁶⁰ a size effect again incommensurate with a critical

stress condition for fracture. In cyclic loading, such microstructural breakdown in heterogeneous ceramics can cumulate rapidly, resulting in severe contact fatigue (Fig. 1e).^{25,26,61-63} In aqueous environments the deformation can be augmented by deep penetrating inner cone cracks, driven by hydraulic pumping.⁶⁴ Finally, the test is readily extendable to brittle layer structures,^{7,65,66} including teeth⁶⁷⁻⁷⁰ and other biological structures, with consequent revelation of undersurface cracking modes (Fig. 1f).

Likewise with patterns from sharp indenters. The quintessential brittle materials, such as normal silicate glasses, exhibit well-defined cross-shaped radial crack patterns over a wide range of loads (Fig. 2b).⁷¹ Some materials depart from this ideal: 'anomalous' glasses and porous ceramics which deform by densification rather than shear;^{46,72,73} coarse-grain ceramics;¹³ phase-transforming ceramics;^{74,75} viscoelastic materials.⁷⁶ But even there indentation patterns provide valuable visual clues to the mechanical complexion. There is also an intrinsic indentation size effect, whereby radial cracks are suppressed below a threshold load (Fig. 2c). This size effect is a manifestation of the different load dependence of the crack dimension c in eqn. 2, $P/c^{3/2} = \text{constant}$, relative to the hardness dimension a , $P/a^2 = \text{constant}$.^{28,77-79} The threshold load diminishes as the acuity of the indenter tip becomes greater.^{49,79} Even in the subthreshold region, strength-degrading flaws can evolve from shear bands within the plastic zone.⁸⁰⁻⁸⁵ In materials like silicon, the deformation occurs in part from crystallographic slip^{86,87} and part from phase transformation (Fig. 2d).^{30,79,88-92} These elements of flaw character and evolution are not readily ascertained by any other experimental approach. Indentations can be conducted at elevated temperatures, enabling one to track the changing competition between slip and cleavage, i.e. brittle-to-ductile transitions.⁹³ Indentations can also be used to probe the properties of internal interfaces in thin films, coatings and composites (Fig. 2e).^{31,55,56,94-97} Finally, they can be used to evaluate residual stresses,⁹⁸⁻¹⁰² as well as provide a vivid demonstration of the intensity of these stresses from observation of spontaneously ejected material after load release (Fig. 2f).^{32,103}

IV. Indentation Toughness

(1) *Critique*

The chief objection to the indentation methodology, specifically by Quinn & Bradt⁹ but also by others,¹⁰⁻¹² centers around the first listed item in the Introduction, i.e. measurement of toughness. Most criticism is directed toward the use of eqn. 4 in conjunction with Vickers indenters, although other variants of this equation are swept up by the broad brush of disapproval. The objectivity is belied by the rhetoric and misconceptions.⁹ It is argued that the indentation fracture mechanics relations in Sect. II do not have applicable fracture mechanics solutions and are instead products of dimensional analyses modified by experimentally derived calibration factors, with 'occasional vague allusions to a theoretical basis'. It is also argued that these calibration factors render the technique suspect in any absolute toughness evaluation. Based on these claims, they unilaterally advocate usage of indentation testing be discontinued. An unfortunate consequence is that this kind of critique spills beyond toughness and casts a pall on all the other applied research areas enumerated in the Introduction.

(2) *Dimensionality of indentation relations—a certain universality*

We assert that the suggestion that the indentation relations in Sect. II do not have a strong foundation in applicable fracture mechanics is baseless. These relations are derived rigorously from first principles, for model crack geometries in well-defined contact fields, with all the important material variables, toughness primarily, expressed in explicit form. They are *not*, as claimed,⁹ derived simply from dimensional analysis. At the same time, there is a commonality in the dimensionality of these relations that speaks to a certain universality in soundly-based fracture mechanics solutions:

(i) *Auerbach's law and JKR.* The condition for cone crack initiation in eqn. 1 is

expressed as a proportionality between the critical load quantity P_C/r and crack resistance R . An identical proportionality is observed in the celebrated JKR relation obtained by Johnson, Kendall & Roberts in their analysis of pulloff force for adherent spheres,¹⁰⁴ with the 'crack resistance' R replaced by an interfacial adhesion energy. This identical form is attributable to the fact that both Hertzian contact configurations essentially involve stable precursor crack growth prior to criticality, in the latter case as the inward running of a crack along the adhesion interface. These relations can *only* be accounted for using rigorous energy-balance principles.

(ii) *Contact far-field solutions.* A key feature of the indentation fracture mechanics relations for fully propagating cracks in both blunt and sharp contact fields is proportionality of the quantity $P/c^{3/2}$ to toughness T in eqns. 2 and 4. The dimensionality is consistent with solutions for center-loaded cracks propagating with circular, penny-like fronts in the far field.^{6,105} This constancy of $P/c^{3/2}$ is in fact remarkably well satisfied in experimental data for glasses over a large range of loads and indenter geometries, blunt (flattened spheres) and sharp (cones and pyramids with different apical angles).^{16,34} It is also confirmed in data using pyramidal indenters in several fine-grain ceramics^{102,106-108}, including data for smaller, less well-developed (Palmqvist) cracks.¹⁰⁹ This resilience in data behavior is testament to the broad reach of a sound fracture mechanics approach. It is true that the presence of macroscopic residual stresses in a body can cause deviations from constancy in $P/c^{3/2}$, but even there such deviations can be usefully employed to quantify the magnitude of such stresses.^{98,102,110-112}

An interesting adjunct to indentation fracture is edge chipping, when point-contacts are placed at a distance h close to an orthogonal side wall. A critical spallation load P is attained only after a contact-initiated crack propagates stably to a critical depth. A basic Griffith–Irwin analysis of the critical condition yields a proportionality between the quantity $P/h^{3/2}$ and toughness T , i.e. of the same form as eqns. 2 and 4.¹¹³ Prior to this

analysis, $P(h)$ data were simply subjected to statistical regression procedures without any consideration of stability in the crack growth, resulting in empirical power laws with no physically or dimensionally correct relation to toughness.

(3) *Assumptions and accuracy—use and misuse*

Notwithstanding the fundamental underpinning in the indentation formulations, eqn. 4 in particular, there are caveats as to accuracy and applicability that should be considered in any usage. Where the analysis is most vulnerable is in the dimensionless coefficients in the toughness relations, especially the quantity ξ in eqn. 4.^{9,11} The indentation stress fields are highly inhomogeneous, and there are acknowledged assumptions in the modeling of inelastic components, so that absolute values deriving from the fracture mechanics analyses are indeed subject to numerical uncertainty, even for materials with well-behaved crack patterns. It is for this reason that the coefficient ξ in the original study was calibrated against independently measured toughness values for select ceramics with single-value toughness.¹³ In that study the absolute numerical accuracy was estimated at 30% to 40% over a wide range of materials, and considerably better for comparative measurements within a given material class. (If these bounds are taken into account, the perceived disparity between toughness values for a selected 'standard reference material' measured by indentation and an independent method vanishes.⁹) In this context it must be reiterated that indentation testing was never proposed by the original authors as a standard for toughness measurement. It has always been advocated as an exploratory test—an incomparably quick, convenient and versatile method for probing fracture susceptibility, especially in a point-to-point capacity and in small-scale specimens, provided due recognition is given to the limits of accuracy.

Other objections to the indentation toughness methodology have been cited, especially in Vickers tests where radial crack patterns depart from the ideal.⁹⁻¹² These include: the tendency for cracks to become disrupted in coarse-grain ceramics;

departures from ideal penny-like crack geometries; softer ceramics where crack extension is not well developed ($c < 2a$); the existence of densification or dilatation in the contact deformation of anomalous glasses, phase-transforming ceramics (zirconia) and porous materials; complications from multiple crack formation (lateral cracking); and the presence of residual stresses. Another criticism cites the need for exacting microscopic examination to locate crack tips in non-reflecting specimen surfaces and to test in inert environments. These are legitimate issues, but all are acknowledged in the original paper by Anstis *et al.*¹³ and in subsequent review articles.^{5,7,8} There are further questions concerning the use of the expanding cavity model for the elastic-plastic field, but this model has been validated experimentally and theoretically for wide ranges of indenter shapes, materials, and crack sizes.^{5,7,8,16,34,106,107,114}

In summary, failure to exercise due diligence when using eqn. 4 for Vickers indentation toughness tests can certainly lead to suspect toughness numbers. The very simplicity of the indentation technique can lead to misuse by the unwary user. In anomalous glasses for instance, the crack patterns tend to be relatively complex, with stunted radial arms.^{46,72} Unconditional measurements can then lead to overstated values. This despite the fact that long-crack toughnesses of anomalous glasses are comparable to their normal glass counterparts.¹¹⁵ Exaggerated toughness values have been reported from Vickers indentations, in some cases with barely visible or even no radial cracks at all.¹¹⁶⁻¹¹⁸ The use of Vickers indentation testing in bone tissue has aroused similar controversy.¹¹⁹⁻¹²¹ In that instance the application of any analysis based on elastic-plastic theory, indentation or otherwise, is problematic because bone exhibits pronounced time dependence and anisotropy in its deformation, important elements missing from the modeling leading to eqns. 3 and 4.⁵⁷ It is interesting that some of the co-authors critical of Vickers toughness evaluation¹¹ have employed this very same technique to map out toughness variations in tooth tissue.⁶⁷

The same cautionary warnings extend to automated nanoindentation testing,

where due allowance needs to be made for potential artifacts from instrument calibration, thermal drift, pile-up or sink-in, surface roughness, tip rounding, tip adhesion, and so on.⁸ Nanoindentation is more than a black box, and misuse can lead to serious errors in property evaluation.⁵⁸ As with all measurement techniques, it is a case of user beware.

(4) Crack equilibrium and stability

A common thread in the indentation fracture mechanics is stability in various initiation and propagation phases of crack evolution. The existence of stable equilibrium states in brittle fracture is in fact the norm.²⁰ Indentation cracks, such as those illustrated in Figs. 1 and 2, simply comprise the most widely documented examples. Crack stability is arguably the least well appreciated element of fracture mechanics. It is suggested by some that indentation tests pertain to an 'arrest' stress intensity factor K_{IA} , which differs from K_{IC} measured at the point of unstable failure.^{9,10} This mindset contends that arrested cracks satisfy some alternative fracture condition, implying that cracks in stable and unstable equilibrium are fundamentally different in nature. That is tantamount to rejecting the Griffith energy-balance concept of fracture (Panel A), which makes no physical distinction between equilibrium crack states. The contention that toughness K_{IC} is specifically a measure of resistance to catastrophic fast fracture is highly restrictive. Perhaps all this is an unfortunate outcome of engineering stress intensity factor terminology, with subscript C interpreted as signifying only instability instead of a more broadly based equilibrium state.

V. Limitations of Pre-crack Test Specimens

If not indentation, what then the alternative? Some advocate the use of engineered test specimens with machined pre-cracks as standards for toughness measurement.⁹ Such tests may be useful to a materials processor or manufacturer who

seeks some form of reliable number to quantify the virtue (or otherwise) of a specific material. However, those specimens require accurate and costly machining with reproducibly sharp pre-cracks to avoid erroneously high values.^{11,12} Specimens involving crack propagation from a sawn notch are particularly suspect. Quoting independently obtained toughness numbers from standardized pre-crack tests alone can provide little or no insight as to how a given material is likely to respond to many practical stress states, especially in intense, inhomogeneous stress fields and in small-scale bodies.

And if, as argued,⁹ the 'arrest' K_A for stable cracks is indeed fundamentally different to 'critical' K_C for unstable cracks, then how can data from pre-crack tests provide any information on any of the applications (other than the first) listed in the Introduction? With regard to the example of anomalous versus normal glasses cited in Sect. IV(3), it is unclear how pre-crack toughness data could predict the different fracture behavior of these two material classes in concentrated fields. Nor is it apparent how pre-crack data might be used to predict strength degradation from microcontacts, or to quantify wear and scratch resistance in service environments, phenomena governed by behavior of small-scale stable cracks.^{3,7,8,122} Long-crack toughness numbers are unlikely to shed any insight into the way flaws evolve at the microstructural level,^{123,124} or on the interactive role of local residual stresses from highly concentrated loads.^{71,123} Such numbers are also unlikely to be useful for materials used in the nanomechanical domain, where responses can undergo marked changes due to size effects and differences in microstructure.¹²⁵ Long-crack specimens are totally ill-equipped to explore point-by-point property variations in a given material component, or distributions of any residual stresses in such a component. It is not the test protocols that are at issue here, but the limited information that can be obtained from them.

Toughness is a nebulous quantity. Any measurement, in either pre-crack or indentation tests, is sensitive to material fabrication (heat treatment, grain size, additives and impurities, second-phase particles, porosity), presence of residual stresses (local and

macroscopic) and exposure to moisture (slow crack growth). In heterogeneous structural materials it depends on crack size and history, in which case long-crack measurement of K_I at instability corresponds to some location along an R -curve.⁶ Toughness *per se* does not rank highly up the ladder of fundamental material properties. Its measurement is best made under conditions that closely represent specific applications, especially in those applications subject to inhomogeneous contact stress states.

VI. Conclusion

This article has sought to make the case that any perceived limitations of the indentation technique are greatly outweighed by an overwhelming abundance of advantages. Some of the critiques contain misconceptions of fracture mechanics. They are based on the restrictive notion that toughness represents only a critical instability condition. That assertion disregards the basis of fundamental Griffith–Irwin fracture mechanics, with misplaced distinctions between stable and unstable equilibrium states. Recent attempts to recast indentation mechanics in term of simplistic critical stress notions reflect a tendency to ignore these fundamentals. Those attempts focus on standardized toughness measurements, a role for which indentations were never proposed in the original papers. The dangers arising from fixation on accurate toughness numbers are twofold: that refutation of indentation analysis as a standard measurement tool should derail the broader range of applications listed in the Introduction; and in so doing, that a wealth of rigorous indentation analysis over more than half a century should be bypassed.

Indentation is a versatile tool for exploring a rich diversity of material responses, in a uniquely visual and quantitative way. Indentation toughness relations are based on rigorous fracture mechanics analyses of crack growth in inhomogeneous but well-documented stress fields. The principal limitation of the toughness equations lies in the

values of the coefficients, although that hardly detracts from the wider utility of the method. Apart from enabling evaluation of material properties, indentation offers rare insight into the way damage evolves in brittle materials—the competition and interaction between cracking and various forms of deformation, and the mechanisms of crack nucleation and initiation at the microstructural level. It quantifies intrinsic size effects and the associated concept of brittleness. Indentation also establishes a physical basis for modeling strength and wear properties. Even departures from ideal behavior can tell us a great deal about the material complexion, including residual stress states. At the same time, application of the technique demands due caution, with full awareness of caveats, as outlined in the original articles.

Acknowledgements

The authors wish to acknowledge contributions over many decades from enumerable students, postdoctoral fellows and guest researchers. Funding for this work was provided by the following sources: (DBM) U.S. Office of Naval Research (N00014-09-C-0288); (NPP) the II-VI Foundation and U.S. Department of energy (DoE-BES #DE-FG02-10ER4677); (JEB) Australian Research Council Future Fellowship; (IER) U.S. Department of Energy (DoE-BES #DE-FG02-07ER46397); (RT) Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000; (TFP) U.K. Engineering and Physical Sciences Research Council; (GMP) National Science Foundation (CMMI 1069165); (BRL) NIST, via a contract administered by Dakota Consulting Inc.

References

1. H. Hertz, *Hertz's Miscellaneous Papers*. Macmillan, London, Chs. 5,6, 1896.
2. F.C. Frank and B.R. Lawn, "On the Theory of Hertzian Fracture," *Proc. Roy. Soc. Lond.*, A299[1458] 291-306 (1967).
3. B.R. Lawn and T.R. Wilshaw, "Indentation Fracture: Principles and Applications," *J. Mater. Sci.*, 10[6] 1049-81 (1975).
4. P. Ostojic and R. McPherson, "A Review of Indentation Fracture Theory: Its Development, Principles and Limitations," *Int. J. Fract.*, 33 297-312 (1987).
5. R.F. Cook and G.M. Pharr, "Direct Observation and Analysis of Indentation Cracking in Glasses and Ceramics," *J. Amer. Ceram. Soc.*, 73[4] 787-817 (1990).
6. B.R. Lawn, *Fracture of Brittle Solids*. Cambridge University Press, Cambridge, Second ed, 1993.
7. B.R. Lawn, "Indentation of Ceramics With Spheres: A Century After Hertz," *J. Amer. Ceram. Soc.*, 81[8] 1977-94 (1998).
8. B.R. Lawn and R.F. Cook, "Probing Material Properties With Sharp Indenters: A Retrospective," *J. Mater. Sci.*, 47[1] 1-22 (2012).
9. G.D. Quinn and R.C. Bradt, "On the Vickers Indentation Fracture Test," *J. Amer. Ceram. Soc.*, 90[3] 673-80 (2007).
10. R. Morrell, "Fracture Toughness Testing for Advanced Technical Ceramics: Internationally Agreed Good Practice," *Advances in Applied Ceramics*, 105[2] 88-98 (2006).
11. J.J. Kruzic, D.K. Kim, K.J. Koester and R.O. Ritchie, "Indentation Techniques for Evaluating the Fracture Toughness of Biomaterials and Hard Tissues," *J. Mech. Behav. Biomed. Mat.*, 2 384-95 (2009).
12. J. Wade, S. Ghosh, P. Claydon and H. Wu, "Contact Damage of Silicon Carbide Ceramics With Different Grain Structures Measured by Hertzian and Vickers Indentation," *J. Europ. Ceram. Soc.*, 35 1725-36 (2015).
13. G.R. Anstis, P. Chantikul, D.B. Marshall and B.R. Lawn, "A Critical Evaluation of Indentation Techniques for Measuring Fracture Toughness: I. Direct Crack Measurements," *J. Amer. Ceram. Soc.*, 64[9] 533-38 (1981).
14. D. Tabor, *Hardness of Metals*. Clarendon, Oxford, 1951.
15. K.L. Johnson, *Contact Mechanics*. Cambridge University Press, London, 1985.
16. S.S. Chiang, D.B. Marshall and A.G. Evans, "The Response of Solids to Elastic/Plastic Indentation. I. Stresses and Residual Stresses," *J. Appl. Phys.*, 53[1] 298-311 (1982).
17. D.J. Green, *Introduction to Mechanical Properties of Ceramics*. Cambridge University Press, Cambridge, 1998.
18. A.A. Griffith, "The Phenomena of Rupture and Flow in Solids," *Phil. Trans. Roy. Soc. Lond.*, A221 163-98 (1920).
19. G.R. Irwin, "Fracture"; pp. 557-94 in *Handbuch der Physik*. Vol. 6. Edited Springer-Verlag, Berlin, 1958.
20. G.I. Barenblatt, "The Mathematical Theory of Equilibrium Cracks in Brittle Fracture," *Adv. Appl. Mech.*, 7 55-129 (1962).
21. J.B. Quinn, S.S. Scherrer and G.D. Quinn, "The Increasing Role of Fractography in the Dental Community"; pp. in *Fractography of Glasses and Ceramics V*:

- Ceramic Transactions, Vol 199*. Edited by J.R. Varner, G.D. Quinn and M. Wightman. John Wiley, Hoboken, NJ, USA, 2011.
22. S.M. Wiederhorn, "Influence of Water Vapor on Crack Propagation in Soda-Lime Glass," *J. Amer. Ceram. Soc.*, 50[8] 407-14 (1967).
 23. F.C. Roesler, "Brittle Fractures Near Equilibrium," *Proc. Phys. Soc. Lond.*, B69 981-92 (1956).
 24. B.R. Lawn and H. Komatsu, "The Nature of Deformation Around Pressure Cracks on Diamond," *Philos. Mag.*, 14[130] 689-99 (1966).
 25. S.K. Lee, S. Wuttiphon and B.R. Lawn, "Role of Microstructure in Hertzian Contact Damage in Silicon Nitride: I. Mechanical Characterization," *J. Amer. Ceram. Soc.*, 80[9] 2367-81 (1997).
 26. F. Guiberteau, N.P. Padture, H. Cai and B.R. Lawn, "Indentation Fatigue: A Simple Cyclic Hertzian Test for Measuring Damage Accumulation in Polycrystalline Ceramics," *Philos. Mag.*, A 68[5] 1003-16 (1993).
 27. D.B. Marshall and B.R. Lawn, "Indentation of Brittle Materials"; pp. 26-46 in *Micro Indentation Hardness Testing*. Edited by P.J. Blau and B.R. Lawn. A.S.T.M. Special Technical Publication 899, Philadelphia, PA, 1986.
 28. Y.-G. Jung, A. Pajares, R. Banerjee and B.R. Lawn, "Strength of Silicon, Sapphire and Glass in the Subthreshold Flaw Region," *Acta Mater.*, 52[12] 3459-66 (2004).
 29. T.F. Page, L. Reister and S.V. Hainsworth, "The Plasticity Response of 6H-SiC and Related Isostructural Materials to Nanoindentation: Slip vs Densification"; pp. 113-18 in *Fundamentals of Nanoindentation and Nanotribology*. Vol. 522. Edited by N.R. Moody, W.W. Gerberich, S.P. Baker and N. Burnham. Materials Research Society, 1998.
 30. I. Zarudi, L.C. Zhang and M.V. Swain, "Microstructure Evolution in Monocrystalline Silicon in Cyclic Microindentations," *J. Mater. Res.*, 18[4] 758-61 (2003).
 31. P.E.D. Morgan and D.B. Marshall, "Ceramic Composites of Monazite and Alumina," *J. Amer. Ceram. Soc.*, 78[11] 1553-63 (1995).
 32. I.E. Reimanis, C. Seick, K. Fitzpatrick, E.R. Fuller and S. Landin, "Spontaneous Ejecta from β -Eucryptite Composites," *J. Amer. Ceram. Soc.*, 90[8] 2497-2501 (2010).
 33. F. Auerbach, "Measurement of Hardness," *Ann. Phys. Chem.*, 43 61 (1891).
 34. B.R. Lawn and E.R. Fuller, "Equilibrium Penny-like Cracks in Indentation Fracture," *J. Mater. Sci.*, 10[12] 2016-24 (1975).
 35. F.B. Langitan and B.R. Lawn, "Hertzian Fracture Experiments on Abraded Glass Surfaces as Definitive Evidence for an Energy Balance Explanation of Auerbach's Law," *J. Appl. Phys.*, 40[10] 4009-17 (1969).
 36. J.D. Poloniecki and T.R. Wilshaw, "Determination of Surface Crack Size Densities in Glass," *Nature*, 229 226-7 (1971).
 37. D.M. Marsh, "Plastic Flow and Fracture in Glass," *Proc. Roy. Soc. Lond.*, A282[1388] 33-43 (1964).
 38. B.R. Lawn and A.G. Evans, "A Model for Crack Initiation in Elastic/Plastic Indentation Fields," *J. Mater. Sci.*, 12[11] 2195-99 (1977).

39. 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] 574-81 (1980).
40. S. Palmqvist, "Method att Bestamma Segheten hos Spread Material, Sarskit Hardmettaler," *Jernkontorets Ann*, 141 300-07 (1957).
41. A.G. Evans and E.A. Charles, "Fracture Toughness Determinations by Indentation," *J. Amer. Ceram. Soc.*, 59[7-8] 371-72 (1976).
42. K. Nihara, "Indentation Fracture Toughness of Brittle Materials for Palmqvist Cracks," *J. Mater. Sci. Lett.*, 2 221-23 (1983).
43. M.T. Laugier, "Palmqvist Crack Extension and the Center-Loaded Penny Crack Analogy," *J. Amer. Ceram. Soc.*, 68[2] C51-C52 (1985).
44. C.B. Ponton and R.D. Rawlings, "Vickers Indentation Fracture Toughness Test: Part I. Review of Literature and Formulation of Standardised Indentation Toughness Equations," *Mater. Sci. Tech.*, 5[9] 865-72 (1989).
45. C.B. Ponton and R.D. Rawlings, "Vickers Indentation Fracture Toughness Test: Part II. Application and Critical Evaluation of Standardised Indentation Toughness Equations," *Mater. Sci. Tech.*, 5[10] 961-76 (1989).
46. Z. Burghard, A. Zimmermann, J. Rödel, F. Aldinger and B.R. Lawn, "Crack Opening Profiles of Indentation Cracks in Normal and Anomalous Glasses," *Acta Mater.*, 52[2] 293-97 (2004).
47. D.B. Marshall, B.R. Lawn and A.G. Evans, "Elastic/Plastic Indentation Damage in Ceramics: The Lateral Crack System," *J. Amer. Ceram. Soc.*, 65[11] 561-66 (1982).
48. G.M. Pharr, D.S. Harding and W.C. Oliver, "Measurement of Fracture Toughness in Thin Films and Small Volumes Using Nanoindentation Methods"; pp. 449-61 in *Mechanical Properties and Deformation Behavior of Materials Having Ultra-Fine Microstructures*. Edited by M. Nastasi, D.M. Parkin and H. Gleiter. Kluwer Academic Publishers, Dordrecht, 1993.
49. G.M. Pharr, "Measurement of Mechanical Properties by Ultra-Low Load Indentation," *Mater. Sci. Eng.*, A253[1-2] 151-59 (1998).
50. D.J. Morris and R.F. Cook, "In Situ Cube-Corner Indentation of Soda-Lime Glass and Fused Silica," *J. Amer. Ceram. Soc.*, 87[8] 1494-1501 (2004).
51. D.J. Morris and R.F. Cook, "Radial Fracture During Indentation by Acute Probes: I. Description by and Indentation Wedging Model," *Int. J. Fract.*, 136[1-4] 237-64 (2004).
52. D.J. Morris, A.M. Vodnik and R.F. Cook, "Radial Fracture During Indentation by Acute Probes: II. Experimental Observations of Cube-Corner and Vickers Indentation," *Int. J. Fract.*, 136[1-4] 265-84 (2004).
53. W.C. Oliver and G.M. Pharr, "An Improved Technique for Determining Hardness and Elastic-Modulus Using Load and Displacement Sensing Indentation Experiments," *J. Mater. Res.*, 7[6] 1564-83 (1992).
54. J.L. Cuy, A.B. Mann, K.J. Livi, M.F. Teaford and T.P. Weihs, "Nanoindentation Mapping of the Mechanical Properties of Human Molar Tooth Enamel," *Arch. Oral Biol.*, 7[4] 281-91 (2002).
55. D.B. Marshall, "An Indentation Method for Measuring Matrix-Fiber Frictional Stresses in Ceramic Composites," *J. Amer. Ceram. Soc.*, 67[12] C259-60 (1984).

56. P.F. Becher, E.Y. Sun, C.H. Hseuh, K.B. Alexander, S.L. Waters and C.G. Westmoreland, "Debonding of Interfaces Between Beta-Silicon Nitride Whiskers and Si-Al-Y Oxynitride Glasses," *Acta Mater.*, 44[10] 3881-93 (1996).
57. A.K. Bembey, M.L. Oyen, A.J. Bushby and A. Boyde, "Viscoelastic Properties of Bone as a Function of Hydration State Determined by Nanoindentation," *Philos. Mag.*, 86[33-35] 5691-5703 (2006).
58. M.L. Oyen and R.F. Cook, "A Practical Guide for Analysis of Nanoindentation Data," *J. Mech. Behav. Biomed. Mater.*, 2 396-407 (2009).
59. B.R. Lawn, "Hertzian Fracture in Single Crystals with the Diamond Structure," *J. Appl. Phys.*, 39[10] 4828-36 (1968).
60. Y.-W. Rhee, H.-W. Kim, Y. Deng and B.R. Lawn, "Brittle Fracture Versus Quasiplasticity In Ceramics: A Simple Predictive Index," *J. Amer. Ceram. Soc.*, 84[3] 561-65 (2001).
61. H. Cai, M.A. Stevens Kalceff and B.R. Lawn, "Deformation and Fracture of Mica-Containing Glass–Ceramics In Hertzian Contacts," *J. Mater. Res.*, 9[3] 762-70 (1994).
62. B.R. Lawn, N.P. Padture, H. Cai and F. Guiberteau, "Making Ceramics 'Ductile'," *Science*, 263 1114-16 (1994).
63. N.P. Padture and B.R. Lawn, "Toughness Properties of a Silicon Carbide With an In-Situ-Induced Heterogeneous Grain Structure," *J. Amer. Ceram. Soc.*, 77[10] 2518-22 (1994).
64. Y. Zhang, J.-K. Song and B.R. Lawn, "Deep Penetrating Conical Cracks in Brittle Layers from Hydraulic Cyclic Contact," *Journal of Biomedical Materials Research*, 73B[1] 186-93 (2005).
65. A. Pajares, L. Wei, B.R. Lawn, N.P. Padture and C.C. Berndt, "Mechanical Characterization of Plasma-Sprayed Ceramic Coatings on Metal Substrates by Contact Testing," *Mater. Sci. Eng.*, A208[2] 158-65 (1996).
66. H. Chai, B.R. Lawn and S. Wuttiphon, "Fracture Modes In Brittle Coatings With Large Interlayer Modulus Mismatch," *J. Mater. Res.*, 14[9] 3805-17 (1999).
67. V. Imbeni, J.J. Kruzic, G.W. Marshall, S.J. Marshall and R.O. Ritchie, "The Dentin–Enamel Junction and the Fracture of Human Teeth," *Nature Mater.*, 4 229-32 (2005).
68. H. Chai, J.J.-W. Lee, P.J. Constantino, P.W. Lucas and B.R. Lawn, "Remarkable Resilience of Teeth," *Proceedings of the National Academy of Sciences of the United States of America*, 106 7289-93 (2009).
69. A. Barani, A.J. Keown, M.B. Bush, J.J.-W. Lee, H. Chai and B.R. Lawn, "Mechanics of Longitudinal Cracks in Tooth Enamel," *Acta Biomater.*, 7 2285-92 (2011).
70. A. Barani, H. Chai, B.R. Lawn and M.B. Bush, "Mechanics Analysis of Molar Tooth Splitting," *Acta Biomater.*, 15 237-43 (2015).
71. D.B. Marshall and B.R. Lawn, "Residual Stress Effects in Sharp-Contact Cracking: I. Indentation Fracture Mechanics," *J. Mater. Sci.*, 14[8] 2001-12 (1979).
72. A. Arora, D.B. Marshall, B.R. Lawn and M.V. Swain, "Indentation Deformation/Fracture of Normal and Anomalous Glasses," *J. Non-Cryst. Solids*, 31[3] 415-28 (1979).

73. T.M. Gross, "Deformation and Cracking Behavior of Glasses Indented With Diamond Tips of Various Sharpness," *J. Non-Cryst. Solids*, 358 3445-52 (2012).
74. R.F. Cook, L.M. Braun and W.R. Cannon, "Trapped Cracks at Indentations: I. Experiments on Yttria-Tetragonal Zirconia Polycrystals," *J. Mater. Sci.*, 29 2133-42 (1994).
75. M.S. Kaliszewski, G. Behrens, A.H. Heuer, M.C. Shaw, D.B. Marshall, G.W. Dransman, R.W. Steinbrech, A. Pajares, F. Guiberteau, F.L. Cumbreira and A. Dominguez-Rodriguez, "Indentation Studies on Y₂O₃-Stabilized ZrO₂: I Development of Indentation-Induced Cracks," *J. Amer. Ceram. Soc.*, 77[5] 1185-93 (1994).
76. M.L. Oyen and R.F. Cook, "Load-Displacement Behaviour During Sharp Indentation of Viscous–Elastic–Plastic Materials," *J. Mater. Res.*, 18[1] 139-50 (2003).
77. B.R. Lawn, T. Jensen and A. Arora, "Brittleness as an Indentation Size Effect," *J. Mater. Sci.*, 11[3] 573-75 (1976).
78. B.R. Lawn and D.B. Marshall, "Hardness, Toughness, and Brittleness: An Indentation Analysis," *J. Amer. Ceram. Soc.*, 62[7-8] 347-50 (1979).
79. J. Jang and G.M. Pharr, "Influence of Indenter Angle on Cracking in Si and Ge During Nanoindentation," *Acta Mater.*, 56 4458-69 (2008).
80. M.V. Swain and J.T. Hagan, "Indentation Plasticity and the Ensuing Fracture of Glass," *J. Phys. D: Appl. Phys.*, 9 2201-14 (1976).
81. J.T. Hagan and M.V. Swain, "The Origin of Median and Lateral Cracks at Plastic Indents in Brittle Materials," *Journal of Physics: D*, 11[15] 2091-102 (1978).
82. J.T. Hagan, "Micromechanics of Crack Nucleation During Indentations," *J. Mater. Sci.*, 14 2975-80 (1979).
83. J.T. Hagan, "Shear Deformation Under Pyramidal Indenters in Soda-Lime Glass," *J. Mater. Sci.*, 15 1417-24 (1980).
84. B.R. Lawn, T.P. Dabbs and C.J. Fairbanks, "Kinetics of Shear-Activated Indentation Crack Initiation in Soda-Lime Glass," *J. Mater. Sci.*, 18[9] 2785-97 (1983).
85. T.F. Page, W.C. Oliver and C.J. McHargue, "The Deformation Behavior of Ceramic Crystals Subjected to Very Low Load (Nano)Indentations," *J. Mater. Res.*, 7[2] 450-73 (1992).
86. M.J. Hill and D.J. Rowcliffe, "Deformation of Silicon at Low Temperatures," *J. Mater. Sci.*, 9[10] 1569-76 (1974).
87. B.J. Hockey and B.R. Lawn, "Electron Microscopy of Microcracking About Indentations in Aluminum Oxide and Silicon Carbide," *J. Mater. Sci.*, 10[8] 1275-84 (1975).
88. D.R. Clarke, M.C. Kroll, P.D. Kirchner, R.F. Cook and B.J. Hockey, "Amorphization and Conductivity of Silicon and Germanium Induced by Indentation," *Phys. Rev. Lett.*, 60[21] 2156-59 (1988).
89. J.E. Bradby, J.S. Williams, J. Wong-Leung, M.V. Swain and P. Munroe, "Mechanical Deformation in Silicon by Micro-Indentation," *J. Mater. Res.*, 16[5] 1500-07 (2001).

90. J.E. Bradby, J.S. Williams, J. Wong-Leung, S.O. Kucheyev, M.V. Swain and P. Munroe, "Spherical Indentation of Compound Semiconductors," *Philos. Mag. A*, 82[10] 1931-39 (2002).
91. V. Domnich and Y. Gogotsi, "Phase Transformations in Silicon Under Contact Loading," *Rev. Adv. Mater. Sci.*, 3 1-36 (2002).
92. Y.B. Gerbig, C.A. Michaels and R.F. Cook, "In Situ Observation of the Spatial Distribution of Crystalline Phases During Pressure-Induced Transformations of Indented Silicon Thin Films," *J. Mater. Res.*, 30[1] 390-406 (2015).
93. N.G.F. Naylor and T.F. Page, "Microstructural Studies Of The Temperature Dependence Of Deformation Structures Around Hardness Indentations In Ceramics," *Journal of Microscopy*, 130[3] 345-60 (1983).
94. P.J. Burnett and T.F. Page, "An Investigation of Ion-Implantation-Induced Near-Surface Stresses and Their Effects in Sapphire and Glass " *J. Mater. Sci.*, 20 4624-46 (1985).
95. T.F. Page and S.J. Bull, "Measuring and Modelling the Instrumented Indentation (Nanoindentation) Response of Coated Systems," *Philos. Mag. A*, 86[33-35] 5331-46 (2006).
96. S.J. Bull and E.G. Berasetegui, "An Overview of the Potential of Quantitative Coating Adhesion Measurement by Scratch Testing," *Tribol. Int.*, 39[2] 99-114 (2006).
97. J. Chen and S.J. Bull, "Indentation Fracture and Toughness Assessment for Thin Optical Coatings on Glass," *J. Phys. D: Appl. Phys.*, 40[18] 5401-17 (2007).
98. D.B. Marshall and B.R. Lawn, "An Indentation Technique for Measuring Stresses in Tempered Glass Surfaces," *J. Amer. Ceram. Soc.*, 60[1-2] 86-87 (1977).
99. T.Y. Tsui, W.C. Oliver and G.M. Pharr, "Influences of Stress on the Measurement of Mechanical Properties Using Nanoindentation: I. Experimental Studies in an Aluminum Alloy," *J. Mater. Res.*, 11 752-59 (1996).
100. T.Y. Tsui, W.C. Oliver and G.M. Pharr, "Influences of Stress on the Measurement of Mechanical Properties Using Nanoindentation: II. Finite Element Simulations," *J. Mater. Res.*, 11 760-68 (1996).
101. D.J. Green, P.Z. Cai and G.L. Messing, "Residual Stresses in Alumina–Zirconia Laminates," *J. Europ. Ceram. Soc.*, 19[13] 2511-17 (1999).
102. R.F. Cook, "Toughening of a Cordierite Glass-Ceramic by Compressive Surface Layers," *J. Amer. Ceram. Soc.*, 88[10] 2798-2808 (2005).
103. S. Ramalingam, I.E. Reimanis and C. Packard, "Determining Activation Volume for the Pressure-Induced Phase Transformation in β -eucryptite Through Nanoindentation," *J. Amer. Ceram. Soc.*, 95[6] 2051-58 (2012).
104. K.L. Johnson, K. Kendall and A.D. Roberts, "Surface Energy and the Contact of Elastic Solids," *Proc. Roy. Soc. Lond.*, A324[1558] 301–13 (1971).
105. Y. Murakami, S. Aoki, N. Hasebe, Y. Itoh, H. Miyata, N. Miyazaki, H. Terada, K. Tohgo, M. Toya and R. Yuuki, eds, "Stress Intensity Factors Handbook." Pergamon Press, Oxford, 1987, p. 669.
106. D.B. Marshall, "Controlled Flaws in Ceramics: A Comparison of Knoop and Vickers Indentation," *J. Amer. Ceram. Soc.*, 66[2] 127-31 (1983).
107. D.B. Marshall, "Geometrical Effects in Elastic/Plastic Indentation," *J. Amer. Ceram. Soc.*, 67[1] 57-60 (1984).

108. R.F. Cook, "Strength and Sharp Contact Fracture of Silicon," *J. Mater. Sci.*, 41 841-72 (2006).
109. J. Lankford, "Indentation Microfracture in the Palmqvist Crack Regime: Implication for Fracture Toughness Evaluation by the Indentation Method," *J. Mater. Sci. Lett.*, 1 493-95 (1982).
110. R. Tandon, D.J. Green and R.F. Cook, "Surface Stress Effects on Indentation Fracture Sequences," *J. Amer. Ceram. Soc.*, 73[9] 2619-27 (1990).
111. R. Tandon and T.E. Buchheit, "Use of Cube-Corner Nano-Indentation Crack Length Measurements to Estimate Residual Stresses Over Small Spatial Dimensions," *J. Amer. Ceram. Soc.*, 90[2] 502-08 (2007).
112. R. Tandon and T.E. Buchheit, "A Technique for Measuring Stresses in Small Spatial Regions Using Cube-Corner Indentation: Application to Tempered Glass Plates," *J. Eur. Ceram. Soc.*, 27 2407-14 (2007).
113. H. Chai and B.R. Lawn, "A Universal Relation for Edge Chipping From Sharp Contacts in Brittle Materials: A Simple Means of Toughness Evaluation," *Acta Mater.*, 55 2555-61 (2007).
114. A.C. Fischer-Cripps and B.R. Lawn, "Stress Analysis of Contact Deformation in Quasi-Plastic Ceramics," *J. Amer. Ceram. Soc.*, 79[10] 2609-18 (1996).
115. S.M. Wiederhorn, "Fracture Surface Energy of Glass," *J. Amer. Ceram. Soc.*, 52[2] 99-105 (1969).
116. A.A. Kaminskii, M.S. Akchurin, V.I. Alshits, K. Ueda, K. Takaichi, J. Lu, T. Uematsu, M. Musha, A. Shirikawa, V. Gabler, H.J. Eichler, H. Yagi, T. Yanagitani, S.N. Bagayev, J. Fernandez and R. Balda, "New Data on the Physical Properties of $Y_3Al_5O_{12}$ -Based Nanocrystalline Laser Ceramics1," *Nanomaterials*, 48[3] 562-66 (2003).
117. G.-D. Zhan, J.D. Kunz, J. Wan and A.K. Mukherjee, "Single-Wall Carbon Nanotubes as Attractive Toughening Agents in Alumina-Based Nanocomposites.," *Nature Mater.*, 2 38-42 (2003).
118. X. Wang, N.P. Padture and H. Tanaka, "Contact-Damage-Resistant Ceramic/Single-Wall Carbon Nanotubes and Ceramic/Graphite Composites," *Nature Mater.*, 3 539-44 (2004).
119. L.P. Mullins, M.S. Bruzzi and P. McHugh, "Authors' Response to 'Comments on Measurement of the Microstructural Fracture Toughness of Cortical Bone Using Indentation Fracture'," *J. Biomech.*, 41[14] 2602-03 (2008).
120. J.J. Kruzic and R.O. Ritchie, "Comments on 'Measurement of the Microstructural Fracture Toughness of Cortical Bone Using Indentation Fracture'," *J. Biomech.*, 41 1379-80 (2008).
121. L.P. Mullins, M.S. Bruzzi and P. McHugh, "Measurement of the Microstructural Fracture Toughness of Cortical Bone Using Indentation Fracture," *J. Biomech.*, 40[14] 3185-88 (2007).
122. S.M. Wiederhorn and B.J. Hockey, "Effect of Material Parameters on the Erosion Resistance of Brittle Materials," *J. Mater. Sci.*, 18[3] 766-80 (1983).
123. D.B. Marshall and B.R. Lawn, "Flaw Characteristics in Dynamic Fatigue: The Influence of Residual Contact Stresses," *J. Amer. Ceram. Soc.*, 63[9-10] 532-36 (1980).

124. B.R. Lawn, "The Indentation Crack as a Model Indentation Flaw"; pp. 1-25 in *Fracture Mechanics of Ceramics*. Vol. 5. Edited by R.C. Bradt, A.G. Evans, D.P.H. Hasselman and F.F. Lange. Plenum, New York, 1983.
125. B.R. Lawn, "Fracture and Deformation in Brittle Solids: A Perspective on the Issue of Scale," *J. Mater. Res.*, 19[1] 22-29 (2004).

Figures

1. Blunt-indenter patterns formed by contact with hard spheres. (a) Schematic, showing formation of cone crack in flat specimen. (b) Underside view of near axisymmetric cone crack formation in silica glass slab, in Hertzian elastic field.²³ (c) Surface view of indentation on (111) diamond surface in elastic contact, showing modifying effect of crystallographic cleavage.²⁴ (d) Half-surface views of indentation in silicon nitride: upper—fine-grain, showing cone cracks in elastic contact; lower—coarse grain, showing quasiplastic impression.²⁵ (e) Surface view of indentation in coarse grain alumina after cyclic loading, showing local grain deformation and dislodgement.²⁶ (f) Section view of indentation in fine/coarse grain silicon nitride bilayer, showing surface and subsurface crack modes.⁷
2. Sharp-indenter patterns formed by contact with fixed-profile diamond indenters. (a) Schematic, showing radial–median (R–M), lateral (L) and cone (or ring) cracks (C). (b) Surface view of well-formed radial cracks from Vickers indentation in soda-lime glass.²⁷ (c) Surface view of subthreshold and postthreshold Berkovich indentations in silicon.²⁸ (d) TEM views of nanoindentations in single crystals: upper—Berkovich, surface view of hexagonal silicon carbide, showing radial cracks and interacting dislocation slip;²⁹ lower—small sphere, side view of silicon, showing slip bands and phase transformations.³⁰ (e) Surface view of Vickers-induced debonding of monazite-coated sapphire fiber in fine-grain alumina matrix.³¹ (f) Side view of spontaneous post-indentation ejection of particulates from release of intense internal residual stresses in β -eucryptite.³²

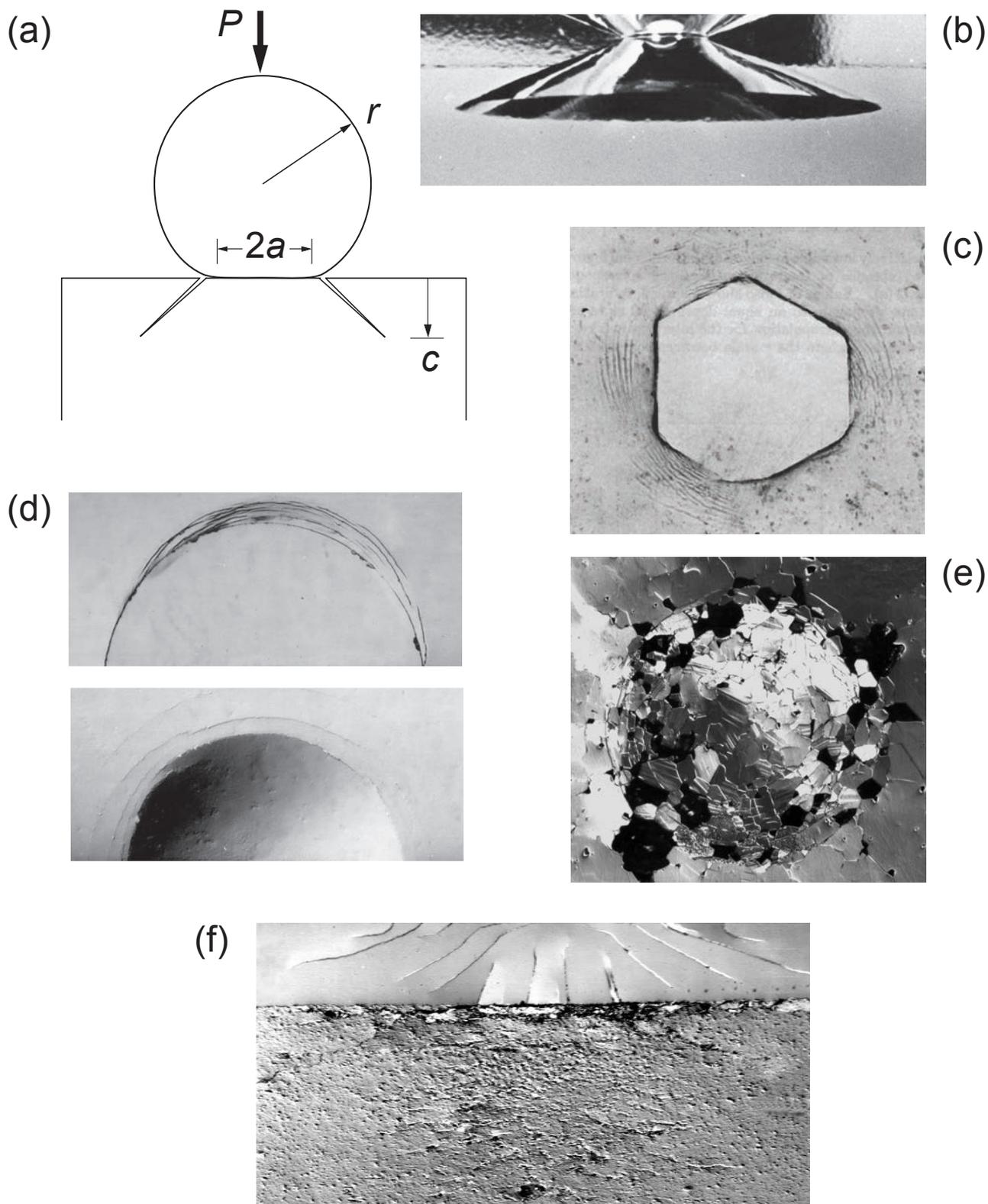


Fig. 1

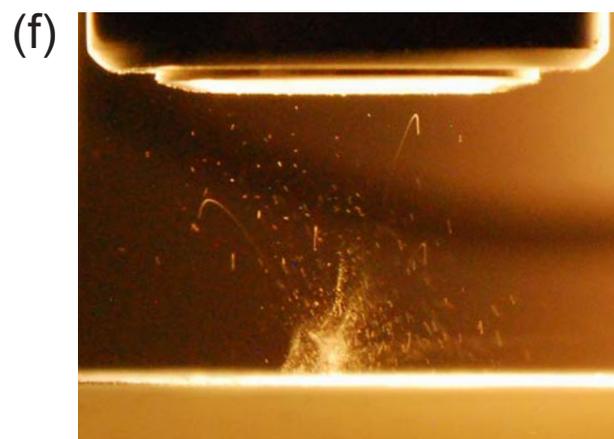
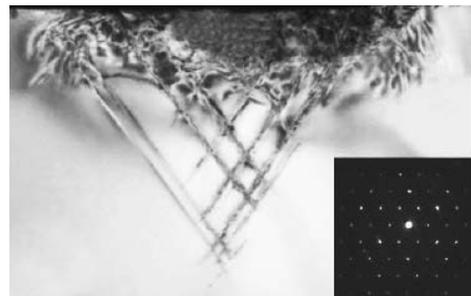
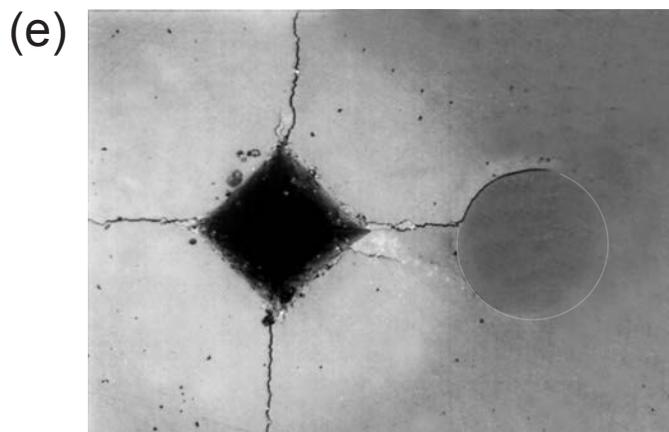
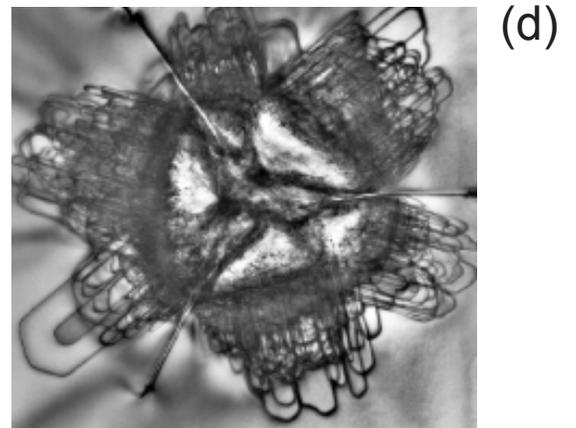
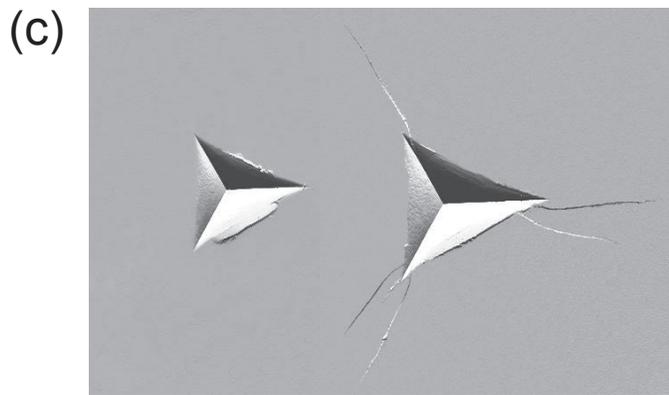
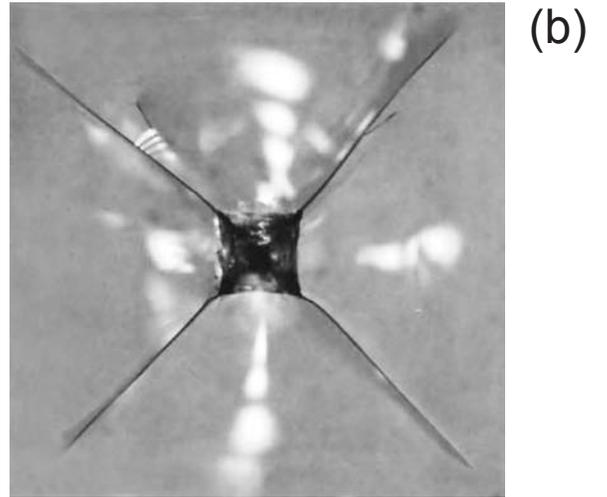
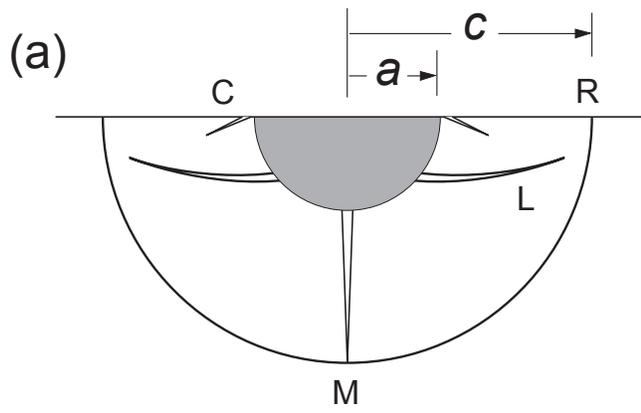


Fig. 2