Designation: C 1678 – 07

Standard Practice for
Fractographic Analysis of Fracture Mirror Sizes in Ceramics and Glasses[1]

This standard is issued under the fixed designation C 1678; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice pertains to the analysis and interpretation of fracture mirror sizes in brittle materials. Fracture mirrors (Fig. 1) are telltale fractographic markings that surround a fracture origin in brittle materials. The fracture mirror size may be used with known fracture mirror constants to estimate the stress in a fractured component. Alternatively, the fracture mirror size may be used in conjunction with known stresses in test specimens to calculate fracture mirror constants. The practice is applicable to glasses and polycrystalline ceramic laboratory test specimens as well as fractured components. The analysis and interpretation procedures for glasses and ceramics are similar, but they are not identical. Different optical microscopy examination techniques are listed and described, including observation angles, illumination methods, appropriate magnification, and measurement protocols. Guidance is given for calculating a fracture mirror constant and for interpreting the fracture mirror size and shape for both circular and noncircular mirrors including stress gradients, geometrical effects, and/or residual stresses. The practice provides figures and micrographs illustrating the different types of features commonly observed in and measurement techniques used for the fracture mirrors of glasses and polycrystalline ceramics.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C 1145 Terminology of Advanced Ceramics

C 1256 Practice for Interpreting Glass Fracture Surface Features

C 1322 Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics

3. Terminology

3.1 *Definitions*: (See Fig. 1)

3.1.1 *fracture mirror*, *n*—as used in fractography of brittle materials, a relatively smooth region in the immediate vicinity of and surrounding the fracture origin **C 1145, C 1322**

3.1.2 *fracture origin*, *n*—the source from which brittle fracture commences. **C 1145, C 1322**

3.1.3 *hackle*, *n*—as used in fractography of brittle materials, a line or lines on the crack surface running in the local direction of cracking, separating parallel but noncoplanar portions of the crack surface. **C 1145, C 1322**

3.1.4 *mist*, *n*—as used in fractography of brittle materials, markings on the surface of an accelerating crack close to its effective terminal velocity, observable first as a misty appearance and with increasing velocity reveals a fibrous texture, elongated in the direction of crack propagation. **C 1145, C 1322**

3.2 *Definitions of Terms Specific to This Standard:* (See Fig. 1)

3.2.1 *mirror-mist boundary in glasses*, *n*—the periphery where one can discern the onset of mist around a glass fracture mirror. This boundary corresponds to A_i, the inner mirror constant.

3.2.2 *mist-hackle boundary in glasses*, *n*—the periphery where one can discern the onset of systematic hackle around a glass fracture mirror. This boundary corresponds to A_o, the outer mirror constant.

3.2.3 *mirror-hackle boundary in polycrystalline ceramics,*, *n*—the periphery where one can discern the onset of systematic new hackle and there is an obvious roughness change relative to that inside a ceramic fracture mirror region. This boundary corresponds to A_o, the outer mirror constant. Ignore premature hackle and/or isolated steps from microstructural irregularities in the mirror or irregularities at the origin.

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[1] This practice is under the jurisdiction of ASTM Committee C28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.03 on Physical Properties and Non-Destructive Evaluation.

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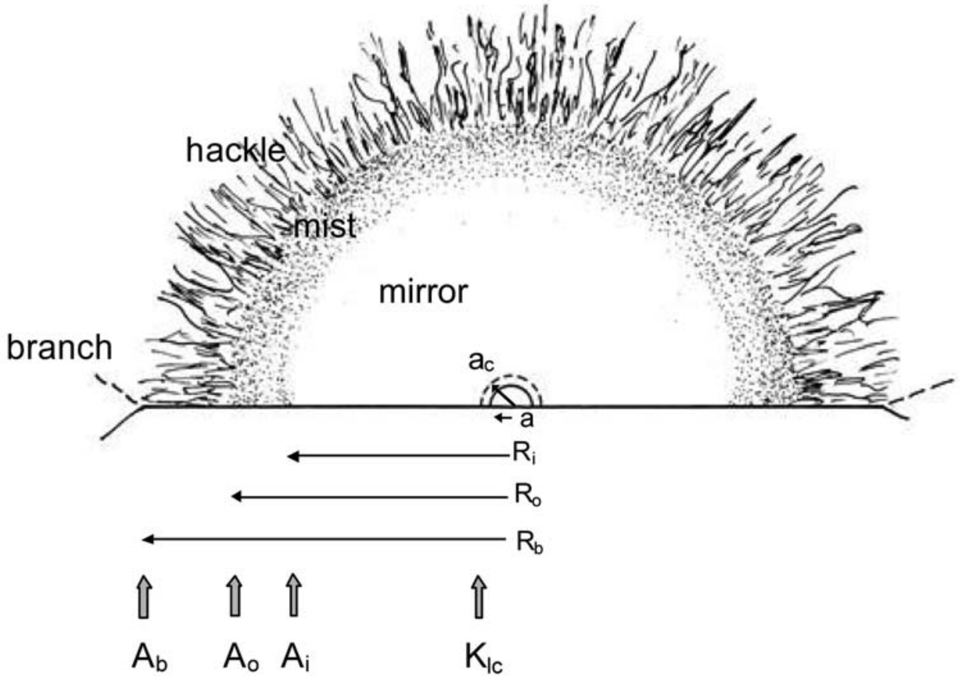
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



NOTE—The initial flaw may grow stably to size a_c prior to unstable fracture when the stress intensity reaches K_{Ic}. The mirror-mist radius is R_i, the mist-hackle radius is R_o, and the branching distance is R_b. These transitions correspond to the mirror constants, A_i, A_o, and A_b, respectively.

FIG. 1 Schematic of a Fracture Mirror Centered on a Surface Flaw of Initial Size (a).

3.2.4 *fracture mirror constant*, *n*—($Fl^{-3/2}$) an empirical material constant that relates the fracture stress to the mirror radius in glasses and ceramics.

4. Summary of Practice

4.1 This practice provides guidance on the measurement and interpretation of fracture mirror sizes in laboratory test specimens as well as in fractured components. Microscopy examination techniques are listed. The procedures for glasses and ceramics are similar, but they are not identical. Guidance is given for interpreting the fracture mirror size and shape. Guidance is given on how to interpret noncircular mirrors due to stress gradients, geometrical effects, or residual stresses.

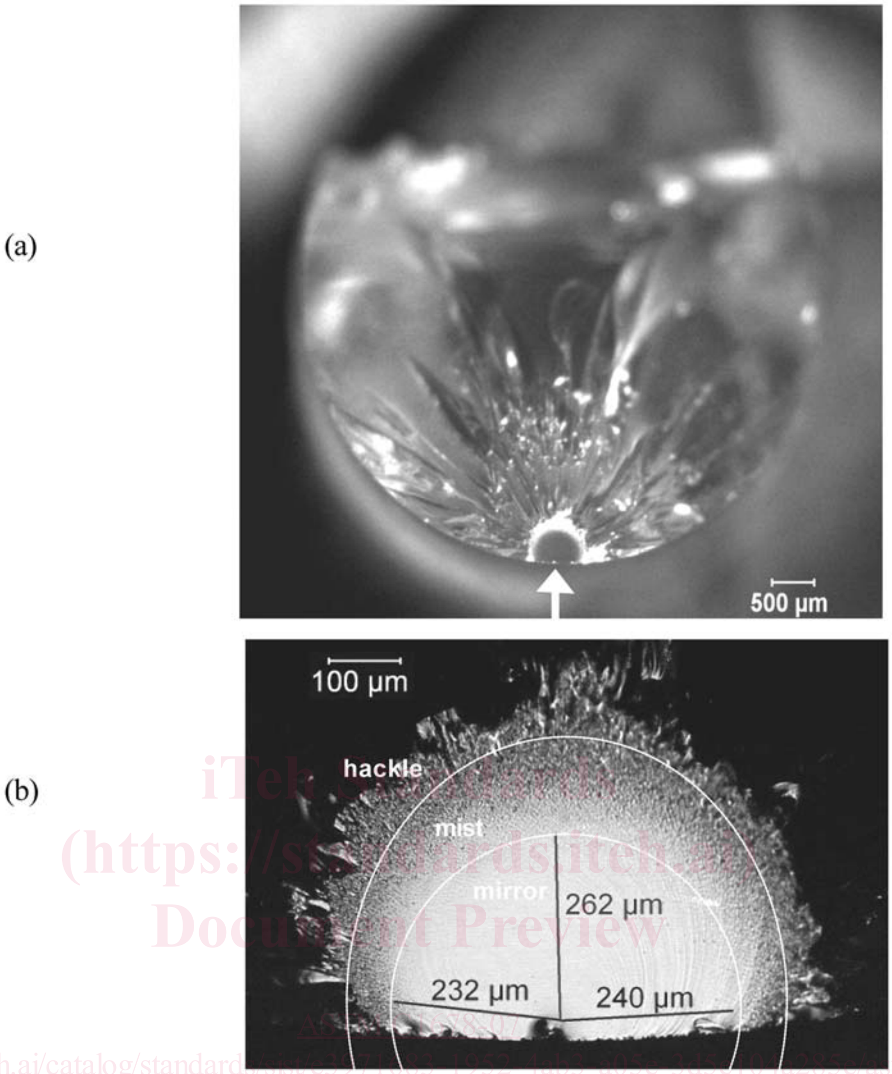
4.2 The stress at the origin in a component may be estimated from the mirror size.

4.3 Fracture mirror constants may be estimated from matched sets of fracture stresses and mirror sizes.

5. Significance and Use

5.1 Fracture mirror size analysis is a powerful tool for analyzing glass and ceramic fractures. Fracture mirrors are telltale fractographic markings in brittle materials that surround a fracture origin as discussed in Practices C 1256 and C 1322. Fig. 1 shows a schematic with key features identified. Fig. 2 shows an example in glass. The fracture mirror region is very smooth and highly reflective in glasses, hence the name "fracture mirror." In fact, high magnification microscopy reveals that, even within the mirror region in glasses, there are very fine features and escalating roughness as the crack advances away from the origin. These are submicrometer in size and hence are not discernable with an optical microscope. Early investigators interpreted fracture mirrors as having discrete boundaries including a "mirror-mist" boundary and also a "mist-hackle" boundary in glasses. These were also termed "inner mirror" or "outer mirror" boundaries, respectively. It is now known that there are no discrete boundaries corresponding to specific changes in the fractographic features. Surface roughness increases gradually from well within the fracture mirror to beyond the apparent boundaries. The boundaries were a matter of interpretation, the resolving power of the microscope, and the mode of viewing. In very weak specimens, the mirror may be larger than the specimen or component and the boundaries will not be present.

5.2 Figs. 3-5 show examples in ceramics. In polycrystalline ceramics, the qualifier "relatively" as in "relatively smooth" must be used, since there is an inherent roughness from the microstructure even in the area immediately surrounding the origin. In coarse-grained or porous ceramics, it may be impossible to identify a mirror boundary. In polycrystalline ceramics, it is highly unlikely that a mirror-mist boundary can be detected due to the inherent roughness created by the crack-microstructure interactions, even within the mirror. The word "systematic" in the definition for "mirror-hackle boundary in polycrystalline ceramics" requires some elaboration. Mirror boundary hackle lines are velocity hackle lines created after the radiating crack reaches terminal velocity. However, premature, isolated hackle can in some instances be generated well within a ceramic fracture mirror. It should be disregarded when judging the mirror boundary. Wake hackle from an isolated obstacle inside the mirror (such as a large grain or agglomerate) can trigger early "premature" hackle lines. Steps in scratches or grinding flaws can trigger hackle lines that



NOTE—(a) shows the whole fracture surface and the fracture mirror (arrow) which is centered on a surface flaw. (b) is a close-up of the fracture mirror which is elongated slightly into the interior due to the flexural stress gradient.

FIG. 2 Optical Micrographs of a Fracture Mirror in a Fused Silica Glass Rod Broken in Flexure at 122 MPa Maximum Stress on the Bottom.

emanate from the origin itself. Sometimes the microstructure of polycrystalline ceramics creates severe judgment problems in ceramic matrix composites (particulate, whisker, or platelet) or self-reinforced ceramics whereby elongated and interlocking grains impart greater fracture resistance. Mirrors may be plainly evident at low magnifications, but accurate assessment of their size can be difficult. The mirror region itself may be somewhat bumpy; therefore, some judgment as to what is a mirror boundary is necessary.

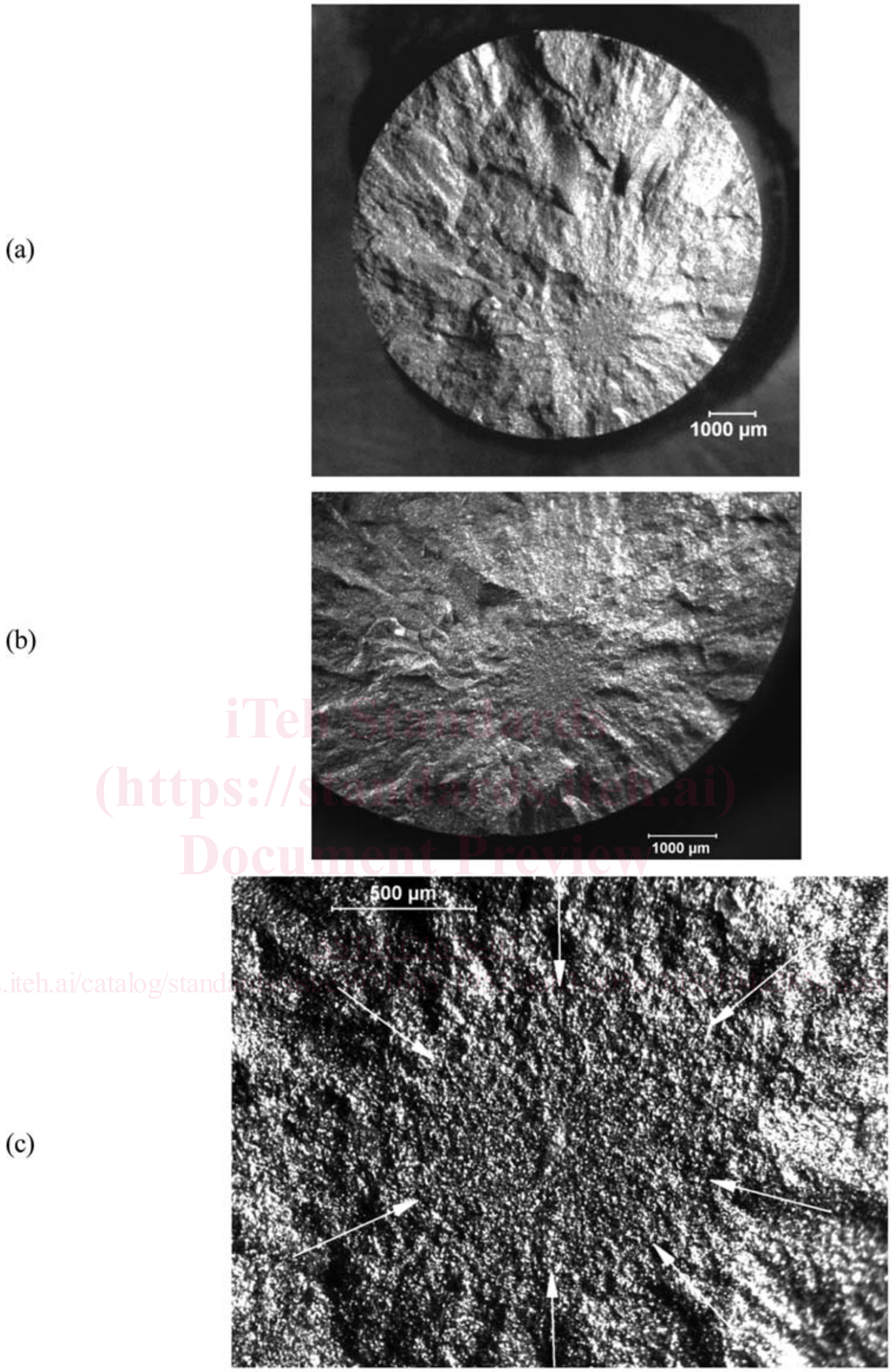
5.3 Fracture mirrors are circular in some loading conditions such as tension specimens with internal origins, or they are nearly semicircular for surface origins in tensile specimens, or if the mirrors are small in bend specimens. Their shapes can vary and be elongated or even incomplete in some directions if the fracture mirrors are in stress gradients. Fracture mirrors may be quarter circles if they form from corner origins in a specimen or component. Fracture mirrors only form in moderate to high local stress conditions. Weak specimens may not exhibit full or even partial mirror boundaries, since the crack may not achieve sufficient velocity within the confines of the specimen.

5.4 Fracture mirrors not only bring one's attention to an origin, but also give information about the magnitude of the stress at the origin that caused fracture and their distribution. The fracture mirror size and the stress at fracture are empirically correlated by Eq 1:

$$\sigma\sqrt{R} = A \qquad (1)$$

where:
σ = stress at the origin (MPa or ksi),
R = fracture mirror radius (m or in),
A = fracture mirror constant (MPa$\sqrt{m}$ or ksi$\sqrt{in}$).

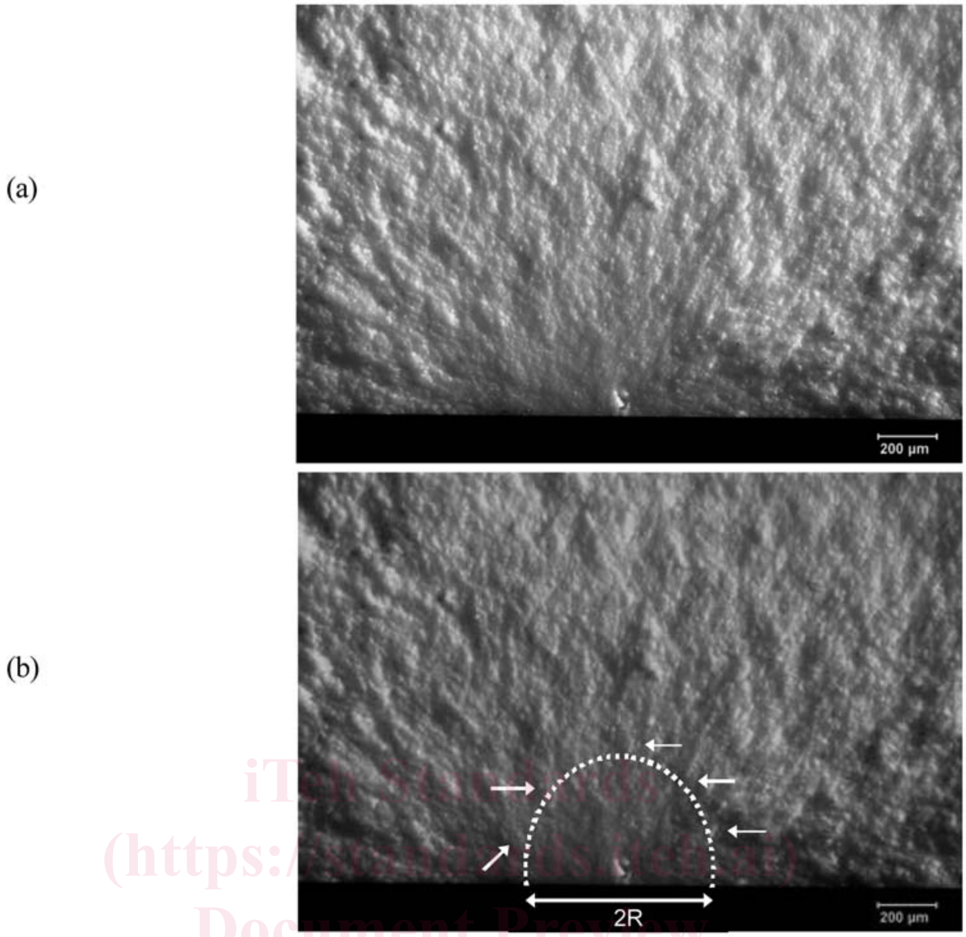
Equation 1 is hereafter referred to as the "empirical stress – fracture mirror size relationship," or "stress-mirror size relationship" for short. A review of the history of Eq 1, and fracture mirror analysis in general, may be found in Refs 1 and 2.



NOTE—Notice how clear the mirror is in the low power images in (a) and (b). The mirror boundary (arrows in c) is where systematic new hackle forms and there is an obvious roughness difference compared to the roughness inside the mirror region.

FIG. 3 Silicon Carbide Tension Strength Specimen (371 MPa) with a Mirror Centered on a Compositional Inhomogeneity Flaw.

5.5 A, the "fracture mirror constant" (sometimes also known as the "mirror constant") has units of stress intensity (MPa$\sqrt{m}$ or ksi$\sqrt{in}$) and is considered by many to be a material property. As shown in Figs. 1 and 2, it is possible to discern separate mist and hackle regions and the apparent boundaries between them in glasses. Each has a corresponding mirror constant, A. The most common notation is to refer to the mirror-mist boundary as the inner mirror boundary, and its mirror constant is designated A_i. The mist-hackle boundary is referred to as the outer mirror boundary, and its mirror constant



NOTE— The mirror boundary is difficult to delineate in this material. (a) shows the uncoated fracture surface of a 2.8 mm thick flexural strength specimen that fractured at 486 MPa. Vicinal illumination brings out the markings. (b) shows a mirror-hackle boundary where systematic new hackle is detected (small white arrows) as compared to the roughness inside the mirror. The marked circle is elongated somewhat into the depth due to the stress gradient. The radius in the direction along the bottom surface (a region of constant stress) was 345 mm.

FIG. 4 A Fracture Mirror in a Fine-Grained 3 Mol % Yttria-Stabilized Tetragonal Zirconia Polycrystal (3Y-TZP).

is designated A_o. The mirror-mist boundary is usually not perceivable in polycrystalline ceramics. Usually, only the mirror-hackle boundary is measured and only an A_o for the mirror-hackle boundary is calculated. A more fundamental relationship than Eq 1 may be based on the stress intensity factors (K_I) at the mirror-mist or mist-hackle boundaries, but Eq 1 is more practical and simpler to use.

5.6 The size predictions based on Eq 1 and the A values, or alternatively stress intensity factors, match very closely for the limiting cases of small mirrors in tension specimens. This is also true for small semicircular mirrors centered on surface flaws in strong flexure specimens. So, at least for some special mirror cases, A should be directly related to a more fundamental parameter based on stress intensity factors.

5.7 The size of the fracture mirrors in laboratory test specimen fractures may be used in conjunction with known fracture mirror constants to verify the stress at fracture was as expected. The fracture mirror sizes and known stresses from laboratory test specimens may also be used to compute fracture mirror constants, A.

5.8 The size of the fracture mirrors in components may be used in conjunction with known fracture mirror constants to estimate the stress in the component at the origin. Practice C 1322 has a comprehensive list of fracture mirror constants for a variety of ceramics and glasses.

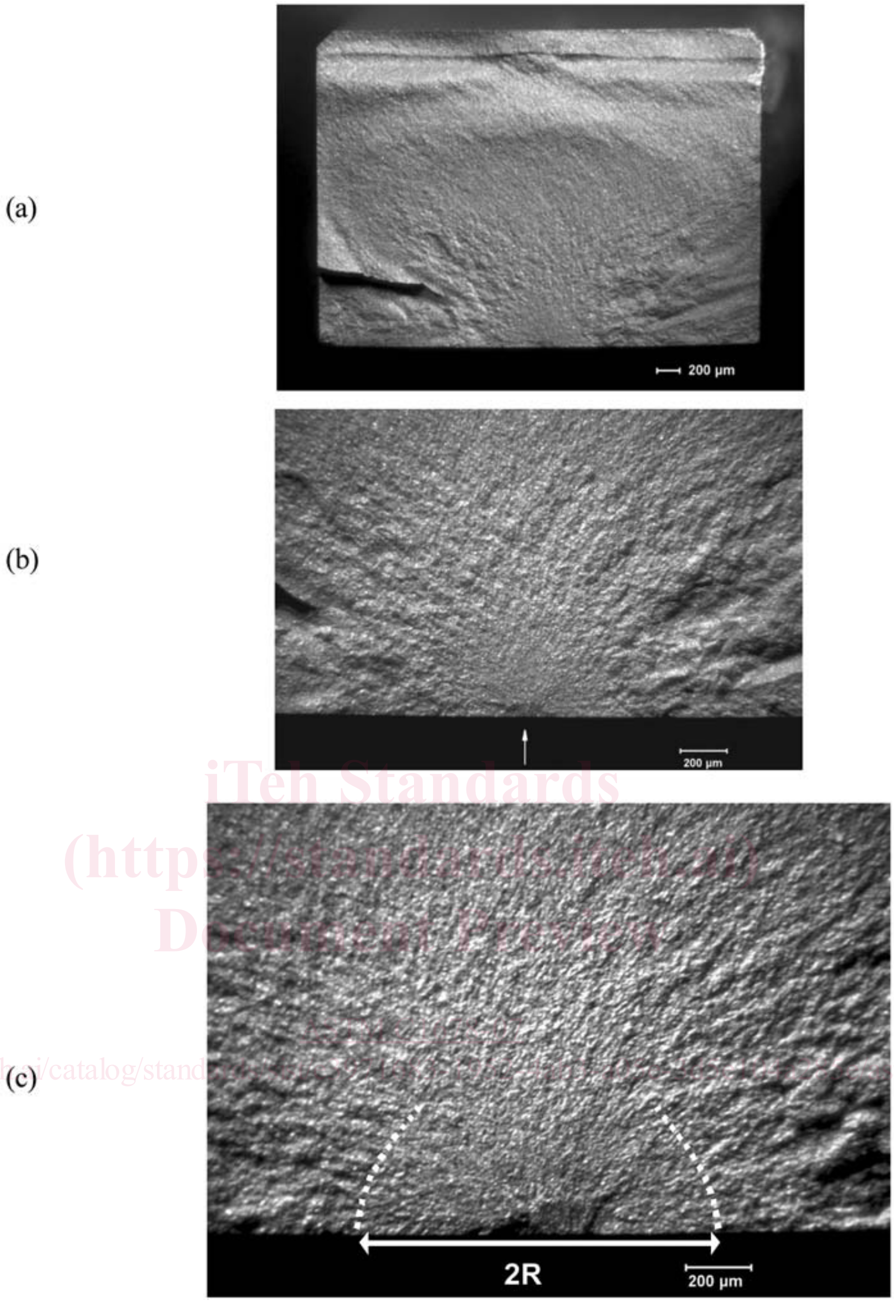
6. Procedure

6.1 Use an optical microscope whenever possible.

6.1.1 For glasses, use a compound optical microscope in bright field mode with reflected light illumination. A scanning electron microscope may be used if optical microscopy is not feasible.

6.1.2 For ceramics, use a stereo optical microscope with low angle grazing (vicinal) illumination. A scanning electron microscope may be used if optical microscopy is not feasible

6.1.3 Differential interference contrast (DIC, also known as Nomarski) mode viewing with a research compound microscope is not recommended for either glasses or ceramics. It is not suitable for rough ceramic fracture surfaces. It also creates complications with glass fracture surfaces. There is no question that DIC mode viewing can discern very subtle mist features in glasses, but the threshold of mist detectability is highly dependent upon how the polarizing sliders are positioned. Hence, DIC measured radii are quite variable. DIC measured



NOTE—The mirror is incomplete into the bend stress gradient, but the mirror sides can be used to construct boundary arcs in (c) [(b) and (c) are close-ups of (a)]. Radii are measured in the direction of constant stress along the bottom.

FIG. 5 Silicon Nitride Bend Bar with a Knoop Surface Crack in a Silicon Nitride (449 MPa).

radii can be substantially smaller than those obtained with conventional viewing modes. It also must be borne in mind that not all users have access to interference contrast microscopes.

6.1.4 Dark-field illumination may be used for glasses, but some resolution may be lost with glasses and radii may be slightly larger as a result. Dark field is very effective with highly-reflective mirror surfaces of ceramic single crystals.

6.1.5 Scanning electron microscope images of mirrors are not recommended for glasses, since the mirror-mist boundary is usually indiscernible. SEM images often appear flat and do not have adequate contrast to see the fine mist detail at the ordinary magnifications used to frame the whole mirror. SEM images may be used with very small mirrors that would be difficult to see with optical microscopy, e.g., high-strength optical fibers. Scanning electron microscope images may be used for ceramics if necessary, but contrast and shadowing should be enhanced.

6.2 The fracture surface should be approximately perpendicular to the microscope optical path or camera.

6.2.1 This requirement poses a small problem if the mirrors in ceramics are examined with a stereo binocular microscope. This microscope has two different tilted optical paths. If