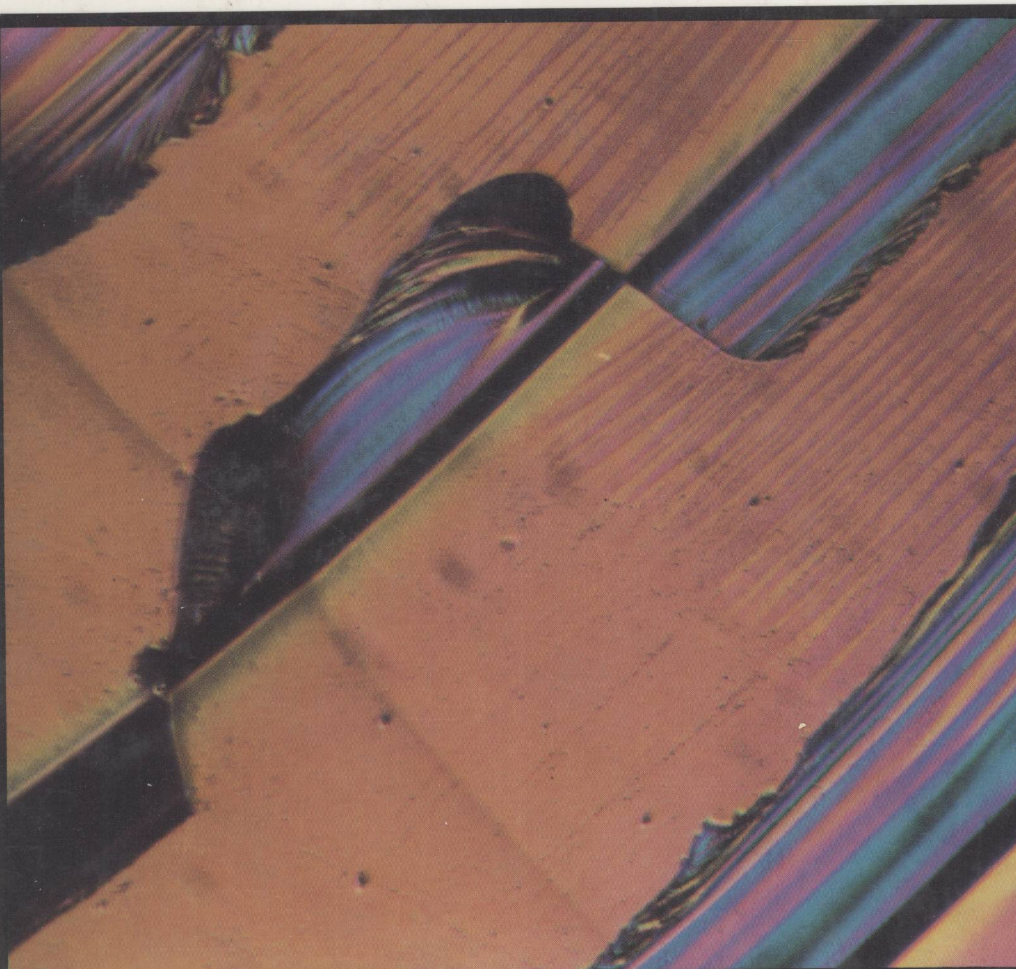
ADVANCES IN CERAMICS • VOLUME 22

FRACTOGRAPHY OF GLASSES AND CERAMICS

Edited by
J. R. Varner
V. D. Frechette



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FRACTOGRAPHY of GLASSES and CERAMICS

Edited by V. D. Fréchette and James R. Varner
Alfred University

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Westerville, Ohio

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On the cover: Optical photomicrograph of a glass fracture-exposed surface (original magnification 100 x), taken using differential interference contrast in reflected light. Visible fracture markings include twist hackle, subcritical cavitation hackle, and a Michalske scarp.

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Preface

The widespread dissemination of fractographic literature on metals and polymers has been notably useful to those concerned with the development and application of those materials. Glasses and ceramics have also seen application of fractographic principles to a point where it was felt to be valuable to convene a conference where exchange of relevant information could take place.

Papers read at the International Conference on the Fractography of Glasses and Ceramics, held at Alfred, NY, August, 1986, are grouped in sections representing the areas of interest in which they fall. Section I, Fundamental Phenomena, deals with crack profiles and markings on crack surfaces, including environmental and compositional effects. Section II, High-Temperature Effects, deals with cracking at elevated temperatures. Section III, Fractography and Fracture Mechanics, is concerned with interrelationships between fracture phenomena and fracture mechanics. Section IV, Fractography in Development and Test, gives examples of the use of fractography in guiding product development and in validating tests. Section V, Field Failures, shows how fractography can be used to identify the causes of failures in service.

The interest in crack behavior and in the markings on crack surfaces has matured to a point where efforts in pure science and in engineering have increasingly converged to complement one another. The exhibition of flint artifacts at the conference, and the demonstration by Mr. Are Tsirk of the flint-knapping techniques by which they were formed, show that the interest is by no means new.

The conference attendees were saddened by the untimely death of Dr. R.F. Pabst of the Institute for Materials Science, Max Planck Institute for Metals, Stuttgart, West Germany, who studied high-temperature crack growth in ceramics with such success. Dr. Pabst had been scheduled to deliver one of the invited papers.

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The assistance of the Convening Committee—Professors J. Bailey, J. Pentecost, J. Mackenzie, and J. Mecholsky; Drs. H. Richter, S. Freiman, J. Kepple, J. Lonergan, T. Michalske, and R. Rice—is also gratefully acknowledged.

Mr. W.T. Emrick, Mrs. D. Brewer, and many colleagues and graduate students of the New York State College of Ceramics deserve much credit for facilitating the conference arrangements.

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Section I

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Perspective on Fractography

Roy W. Rice

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A perspective on the fractography of ceramics having amorphous, single- or polycrystalline, and single- or multiphase character is presented. Techniques of directly observing fracture surfaces, as well as crack-path profiles, and opportunities for better exploiting these methods are first reviewed. Major aspects of fracture, such as path/mode (e.g., in polycrystalline bodies) and especially mirror, mist, hackle, and branching patterns, are then reviewed and briefly contrasted with features on other materials, such as metals and polymers. Next our understanding of the mechanisms associated with these features is assessed. The inadequacy of any single criterion to explain mirror, mist, hackle, and branching is noted, and the potential of combined criteria, such as stress intensity and strain-energy density, is discussed. The lack of a basic understanding of the actual mechanism for generating any of these features, especially nucleation is, however, pointed out. Finally, opportunities and needs for further study are noted, mostly from a generic point of view, along with a general discussion of the use of fractography. Here, both the encouraging use of fractography for advanced ceramics (e.g., in engine components) and the challenge of obtaining much broader use in mechanical-property studies are noted.

Fractography is both extremely challenging and important, because it provides a permanent record of many—and possibly all—key aspects of the fracture process. This importance, of course, in turn stems from the significance of understanding the fracture process, which is critical because of the central, pervasive role of avoiding mechanical failure in materials applications. Both the importance and complexity of fractography also derive from the nature of the fracture process itself.

Two fundamental aspects that greatly contribute to the complexity of fracture are its velocity and geometry. The velocity provides significant complexity, since fracture speeds on the order of 10^3 m/s are typically reached. The directional aspect of velocity is also important, because variations of the fracture path may occur along some, or all, of the fracture, and many of these paths often differ in basic parameters that affect the fracture process itself. Much early work on fractography was with glasses, not only due to their importance and advanced status of development, but also because their isotropy and homogeneity on both the macro- and microscale simplified fracture analysis. The geometric complexity of fractography arises because the fracture process, at least until it ends, generates internal surfaces within the body. The combination of fracture speed and the

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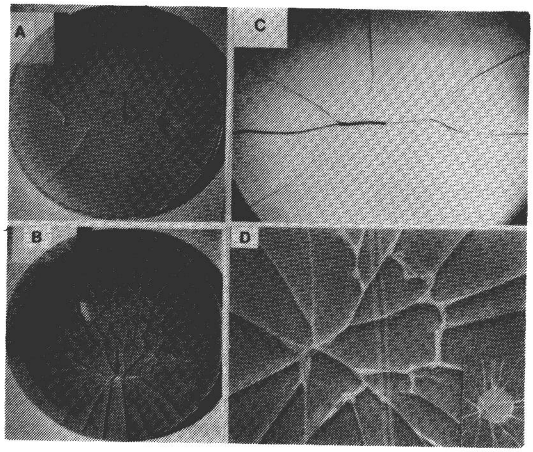

Fig. 1. Crack branching of ceramic specimens fractured at room temperature: *(a)* and *(b)* show, respectively, lower and higher stress failures in MgF_2 disks 8.5 cm in diameter, tested in ring-on-ring tests with diameters of 8.1 and 3.8 cm at failure stresses, respectively, of 47.0 MPa (6.83×10^3 psi) and 81.5 MPa (13.3×10^3 psi); *(c)* shows Al_2O_3 disk 5.05 cm in diameter, failed by ring-on-ring testing with 3.8- and 1.9-cm-diameter support-loading points at a stress of 269 MPa (38.8×10^3 psi) (specimen courtesy of Prof. G. Sines, UCLA); *(d)* is 10.2- by 8.3- by 0.125-cm glass plate, failed using ring-on-ring testing (loading diameters, 6.03 and 1.27 cm) at ≈70 MPa (10 000 psi) (specimen courtesy of K. McKinney, Naval Research Lab).

internal nature of the fracture process has made it extremely difficult to view directly, hence placing great importance on ''reading'' the fracture topography, as a basic record of fractures after the fact.

The methodology of observing fractures, our understanding of the relationship of the primary fracture characteristics—namely the mirror, mist, hackle, and crack-branching patterns—and the fracture path/mode are summarized in this paper. Some uses of fractography, along with needs and opportunities for further study are also discussed. Although a previous summary of fractography[1] is drawn upon, considerable updating, as well as a change in emphasis, is provided by this perspective.

Observational Techniques

Fractography is important, in addition to those reasons just cited, because it can accomplish so much with such simple tools, ranging from the unaided eye (Fig. 1) to the hand lens to inexpensive, low-power microscopes. Overall, optical microscopy (see Fig. 2) is still the single, most powerful/cost-effective tool for fractography. This method has been greatly aided by the development of stereo microscopes, high-magnification, long-working-distance lenses, and a much broader availability of various contrast systems, such as dark-field microscopy, interferometry, and, especially, interference contrast. Broader availability, sim-

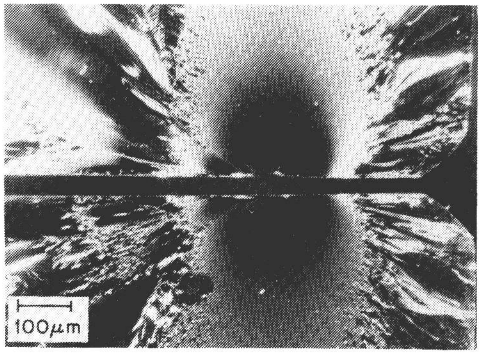

Fig. 2. Optical photomicrographs of fracture mirrors on matching halves of a silicate-based glass bar (1 by 2.5 cm) broken in normal room-temperature flexural testing at 62 MPa (8.9×10^3 psi). Note elongation of mirror into thickness of sample, reduced density of mist, and absence of hackle in the portions of fracture closer to the neutral axis, due to stress gradients. Also note much earlier onset of mist, from each of two features in left side of fracture mirror.

plicity of operation, and low cost have also made sputter-coating systems, to provide enhanced reflectivity, helpful, although these methods are not used nearly as extensively as they might be.

Certainly, the most significant advance in recent years has been the wide availability of scanning electron microscopes (SEMs) for observing fracture surfaces. These instruments not only solve almost all of the depth-of-field limitations of the optical microscope, but substantially surpass its magnification. Although the SEM still does not offer the quality of contrast available in an optical microscope, especially for differentiating more subtle topographic features, electronic-contrast systems and our understanding of them (Fig. 3) are clearly improving. Furthermore, the ability to do elemental analysis has greatly improved our opportunity to understand microstructural details of the fracture path. (Note that developments, especially of micro-Raman techniques, have also begun to provide some of these capabilities for optical microscopes.) Finally, the SEM offers opportunities for the quantitative analysis of topography (for instance, coupled into a computer, for even more sophistication[1]) that are now gaining acceptance.[3]

An important adjunct to these fracture-surface observations, especially for the optical microscope, with its high-contrast capabilities, is ultrasonic marking of the fracture surface. This procedure is accomplished by ultrasonically activating the specimen so that the crack path is actually modulated by the activation, hence leaving timing marks on the surface (Fig. 4). Whereas earlier systems used hundreds of watts of power[4–6] for such activation, more recent techniques require much less power.[7]

Two other techniques for characterizing fracture surfaces are X-ray topography and electron channeling. Although limited in magnification capabilities, X-ray topography can characterize not only slip, but several types of defects in the region somewhat below the fracture surface (see Fig. 5). This technique has only

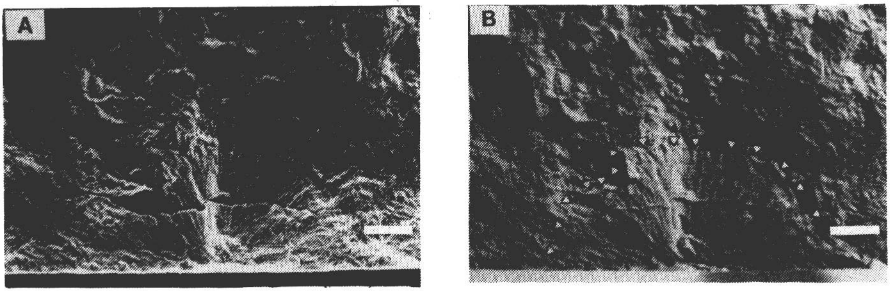

Fig. 3. Example of changing clarity of microstructural features with different contrast systems in scanning electron microscopy: *(a)* fracture origin of ceramic specimen using topographical backscatter image, clearly identifying failure-initiating flaws (triangular markers); *(b)* same location and magnification, taken using compositional backscatter, more clearly delineating microstructural features and less clearly delineating failure-causing flaw. (Photos courtesy of Dr. J. J. Mecholsky, Jr. (Ref. 2). Copyright ASTM. Reprinted with permission.)

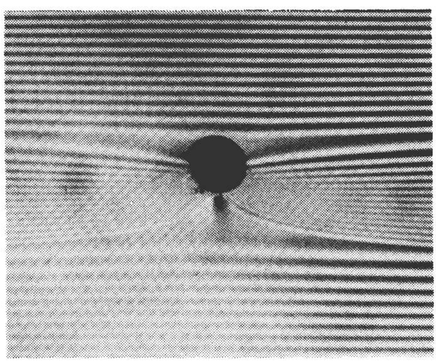

Fig. 4. Ultrasonic timing marks on a glass fracture. Crack propagated from photo top toward bottom, intersecting a pore ≈65 μm in diameter at center of photo (actually a capillary tube perpendicular to the fracture in the specimen). Ultrasonic markings clearly show high (≈150 m/s) initial velocity, slight acceleration of crack as it first contacts void, then significant retardation of crack propagation as it prepares to leave void, and lower (≈20 m/s) subsequent velocity. (Photograph courtesy of Dr. E. Sommer, Fraunhofer Institut für Werkstoffmechanick.)

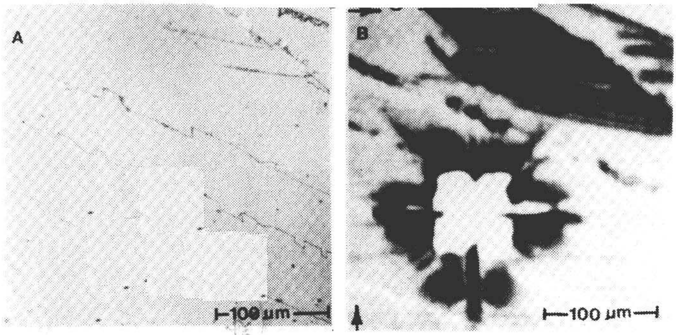

Fig. 5. Illustration of X-ray topographic method of examining ceramic surfaces: *(a)* optical micrograph of cleaved MgO crystal surface with diamond pyramid indent, showing only surface features; *(b)* X-ray topographic photo of same specimen and area, showing substantial additional slip and cracking immediately beneath surface around indent and along some cleavage steps (<010> is vertical and <100> horizontal). (Photos courtesy of Dr. Carl Wu, U. S. Naval Research Lab (Ref. 8). Copyright by The American Ceramic Society. Reprinted with permission.)

recently been applied to fractography, but it is expected to identify defects such as microcracks and foreign particles, as well as slip and twinning. Electron channeling apparently has two applications: First, it may be feasible for identifying the specific crystallographic orientation of individual grains, which could be of significant value; second, the technique seems capable of identifying plastic deformation in the near-surface region. Langford and Davidson[9] thus reported some evidence of plastic deformation in the fracture surfaces of large-grained, dense, polycrystalline Al_2O_3 (Lucalox).

Although the techniques just discussed for directly observing the fracture surface are the most general and most important, there are also ways to observe fracture profiles. The simplest of these techniques, of course, are visual and optical examination. Observing the intersection of the fracture with the surface of the specimen during incremental loading of specimens with controlled crack growth (for example, constant-K double-cantilever-beam (DCB) specimens) is one important technique. Such profiling provides valuable insight into microcracking and crack branching above or below the fracture surface and the choice of crack paths, that is, whether a crack changes course to follow a grain boundary or cleave a grain (Fig. 6). High-speed photography, as pioneered by Schardin,[10] is another technique that essentially provides stop-action photos of a catastrophic crack during its various stages of propagation. This method is, however, limited to modest magnifications because of the high light intensities that it demands.

The crack-profile technique with incremental loading has been extended to the SEM[11] and TEM (transmission electron microscope) (Figs. 6 and 7). The development of high voltage—one-million-volt—microscopes, to allow transmission through much thicker specimens, and in situ loading techniques has made possible crack-propagation profiles in situ in the TEM and, hence, at high

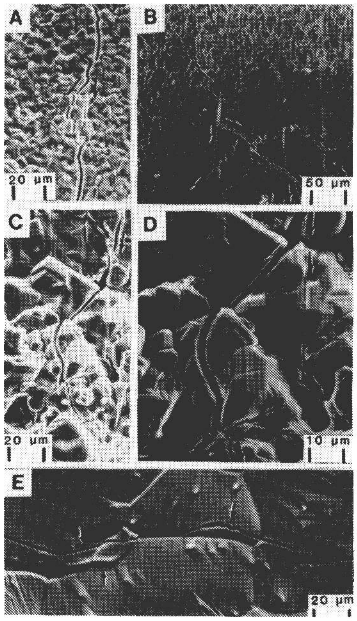

Fig. 6. SEM of crack profiles: *(a)* and *(b)* MgO; *(c)* and *(d)* Al_2O_3; and *(e)* Y_2O_3 (arrows indicate direction of crack propagation). Note: (1) crack branching by ≈1 grain width and rejoining *(a)* and *(c);* (2) more branching and a wider separation of branches in larger grain regions of *(d)* and especially *(b);* and (3) absence and apparent avoidance of grain-boundary fracture in *(e),* which may correlate with low stress corrosion in Y_2O_3.

magnifications. This technique has greatly clarified the mechanisms of toughening in alumina-zirconia and partially stabilized zirconia, as demonstrated by Rühle et al.[12] (Fig. 7).

A similar, but importantly different, profile technique was developed by Wu et al.[11,13] using X-ray microradiography (Fig. 8). Although this technique, as most profile techniques, is limited to observing static cracks, much can again be learned by following the path resulting from the incremental loading of specimens with stable crack propagation, e.g., constant-*K* DCB specimens. Slightly rotating the specimen about the crack axis relative to the X-ray beam allows some mapping of internal crack wandering and branching. The technique is also broadly applicable for internal observation, since it is not dependent on optical transparency.

The availability of synchrotron radiation sources should significantly extend this X-ray technique, with a possibly revolutionary impact on our observation of the fracture process: The intensity of synchrotron sources should allow stop-action X-ray microradiographs of catastrophic fracture. Developing the mechanics of a "camera" that allows film to be advanced at the necessary rates and subsequent registry of sequential frames for later analysis, and possibly making an actual

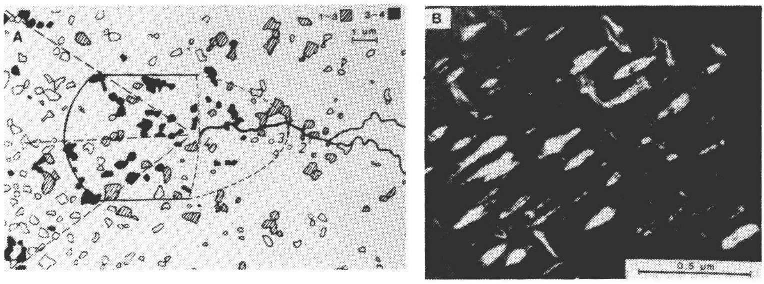

Fig. 7. High-voltage TEM profile of crack propagation in ZrO_2-toughened material: *(a)* Al_2O_3–ZrO_2 specimen evaluated for four subsequent in situ stages of crack development. Hatched areas are ZrO_2 grains transformed during crack propagation 1–3, solid areas grains transformed during fourth crack propagation, and blank enclosed areas untransformed ZrO_2 grains; *(b)* Mg-PSZ in dark field, showing transformed precipitates (white) around crack across center. (Photos courtesy of Dr. M. Rühle, U. of California, Santa Barbara (Ref. 12). Copyright by The American Ceramic Society. Reprinted with permission.)

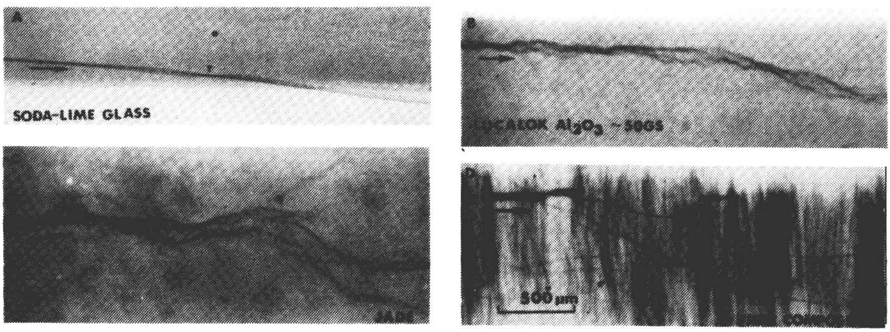

Fig. 8. X-ray microradiographic profiling of cracks. These photos illustrate type of crack profiles seen using X-ray microradiography per the technique of Wu et al. (Ref. 13). Note corresponding increase in crack complexity and diffuseness as fracture toughness increases from (*a*) through (*d*).

movie, would be a major step. Retention of registry is a challenge, because this technique derives from magnifying the X-ray image of the crack on the film in an optical microscope. High-magnification movies of high-speed crack propagation will thus require accurate registry of the magnifications of the crack image from frame to frame and the reassemblement of this sequence to make a motion picture of the actual crack. This process is clearly feasible, but will require significant engineering; the payoff, on the other hand, should be extensive.

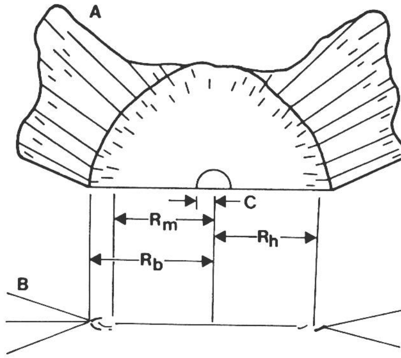

Fig. 9. *(a)* Schematic of fracture-mirror, mist, hackle, and branching patterns in typical ceramic specimen: *C* represents radius of failure causing flaw, shown here as an idealized half-penny, i.e., semicircular, flaw; R_m is radius from center of origin to onset of mist and R_h radius from center of origin to onset of hackle; R_b is similar radius to onset of macroscopic crack branching. Note that different distances between R_m-R_b have been significantly exaggerated. *(b)* Cross-sectional schematic of above fracture surface, illustrating typical branching orientations and options, i.e., two branches only (right) or two branches plus continuation of original crack (left).

Mirror, Mist, Hackle, and Crack-Branching Fracture Patterns

Overall Behavior and Trends

The most important and widely observed features of ceramic fracture are the normally sequentially formed mirror, mist, hackle, and crack-branching patterns (Fig. 9). Overall, these features are well known. Failure originates from a flaw, which may or may not be a sharp crack, but from which, in any case, a sharp crack develops and propagates. The velocity increases rapidly as the crack propagates, approaching terminal velocities of $\approx 10^3$ m/s within 3 to 6 times the length of the original crack.[1] Over, and generally substantially beyond, the region of acceleration, the crack generates a rather smooth fracture surface, which, because of its high mirrorlike reflectivity on glass samples, is called the fracture mirror. This flat, smooth mirror region is bounded by small, radially shaped ridges, or flakes, called mist; shortly beyond this first region, larger, radially shaped ridges, called hackle, generally begin. The onset of hackle is, in turn, frequently followed by macroscopic crack branching (Fig. 9), with possible subsequent sequences of crack branching (Fig. 1).

These patterns are important for identifying the region, if not the specific source, from which fracture initiates, because they are usually symmetric about the fracture origin and the mist and hackle are generally radially oriented. In addition, the radii (R_i) from the center of the origin to each subsequent feature tend to be inversely related to the fracture stress (σ_f),[12–14] i.e.,