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MILITARY HANDBOOK

FRACTOGRAPHY AND CHARACTERIZATION OF FRACTURE ORIGINS IN ADVANCED STRUCTURAL CERAMICS

AMSC N/A FSC 9350

DEPARTMENT OF DEFENSE

Structural Ceramics

1. This Military Handbook is approved for use by all Departments and Agencies of the Department of Defense.

2. This handbook provides information on the fractography of advanced structural ceramics as it pertains to their fracture origins. This handbook is not intended to be referenced in purchase specifications except for informational purposes, nor shall it supersede any specification requirements.

3. Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: U.S. Army Research Laboratory, Materials Directorate, ATTN: AMSRL-MA-S, Watertown, MA 02172-0001 by using the Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

FOREWORD

The objective of this handbook is to provide an efficient and consistent methodology to locate and characterize fracture origins in advanced structural ceramics. It is applicable to advanced structural ceramics which are brittle; that is, the material adheres to Hooke's law. In such materials, fracture commences from a single location which is termed the fracture origin. The fracture origin in brittle ceramics normally consists of some irregularity or singularity in the material which acts as a stress concentrator. In the parlance of the engineer or scientist, these irregularities are termed "flaws" or "defects". The latter should not be construed to mean that the material has been prepared improperly or is somehow faulty. This usage is consistent with the definition of a fracture origin given by Frechette: "The fracture origin is that flaw (discontinuity) from which cracking begins.". (See Failure Analysis of Brittle Materials by Frechette (1990) in the Bibliography.)

The handbook will address monolithic and some composite ceramics, e.g. particulate- and whisker-reinforced and continuous-grain-boundary phase ceramics. (Long- or continuous-fiber reinforced ceramics are excluded.) For some materials, the location and identification of fracture origins may not be possible due to the specific microstructure.

The handbook is principally oriented towards characterization of fracture origins in specimens loaded in so called "fast fracture" testing, but the approach can be extended to include other modes of loading as well.

The procedures described within are primarily applicable to mechanical test specimens, although the same procedures may be relevant to component failure analyses as well. It is customary practice to test a number of specimens (constituting a "sample") in order to permit statistical analysis of the variability of the material's strength. It is usually not difficult to test the specimens in a manner that will facilitate subsequent fractographic analysis. This may not be the case with component failure analyses.

This handbook is applicable to quality control, materials research and development and design. It will also serve as a bridge to mechanical testing standards and statistical analysis practices to permit comprehensive interpretation of data for design. An important feature of this handbook is the adoption of a consistent manner of fracture origin characterization including nomenclature. This will further enable the construction of efficient computer data bases.

The irregularities or flaws which act as fracture origins in advanced structural ceramics can develop during or after fabrication of the material. Large flaws (relative to the average size of the microstructural features) such as pores, agglomerates and inclusions are typically introduced during processing and can (in one sense) be considered intrinsic to the manufacture. Other flaws can be introduced after processing as a result of machining, handling, impact, wear, oxidation, corrosion and high temperature exposure. These flaws can be considered extrinsic flaws. However, machining damage will be considered intrinsic to the manufacture to the extent that machining is a natural consequence of producing a finished specimen or component. It is beyond the scope of this handbook to discuss the origin of flaws or their behavior from a fracture mechanics viewpoint. A comprehensive bibliography is provided as an appendix to this handbook for additional information. It behooves the fractographer to avail him or herself of all available processing or subsequent usage information in order to make as complete a fractographic analysis as possible. Regardless of how flaws develop they are either inherently volume-distributed throughout the bulk of the material (e.g. agglomerates, large grains or pores) or inherently surface-distributed "on"

the material (e.g. handling damage, pits from oxidation or corrosion). The volume-distributed origins in a ceramic material can, in any test specimen, be located in the bulk, at the surface or near-to-the-surface, or at an edge of a specimen. The variety of locations for a volume-distributed flaw is a consequence of the random sampling procedure incurred in preparing test specimens (e.g. machining).

As fabricators improve materials by careful process control thus eliminating large, "abnormal" microstructural features, ceramics will become strength-limited by "flaws" that come from the large-sized end distribution of the "normal" microstructural features. Rice has considered these origins-of-failure and refers to them as "mainstream microstructural features". (See Bibliography: R. Rice, "Failure Initiation in Ceramics: Challenge to NDE and Processing" 1988). In other instances, regions of slightly different microstructure (locally higher microporosity) or microcracks between grains (possibly introduced by thermoelastic strains) may act as failure origins. Literature examples of these are discussed in the Bibliography (see MTL TR 90-57, "Fractographic Analysis of Monolithic Advanced Structural Ceramics"). These origins will blend in well with the background microstructure and will be extremely difficult or impossible to discern even with careful scanning electron microscopy (SEM). This handbook can still be used to analyze such failure origins, but specific flaw definitions may have to be devised.

CONTENTS

1. GENERAL

1.1 Summary of practice. The following procedure should be followed in characterizing fracture origins in advanced ceramics:

(a) Test the specimen to failure in a fashion that preserves the primary fracture surface and the specimen for further reference.
(b) Carefully handle and store the specimen to minimize additional damage and/or contamination of the fracture surfaces.
(c) Visually (1-10X) inspect the fractured specimen in order to determine crack branching patterns, any evidence of abnormal failure patterns (indicative of testing misalignments), the primary fracture surface and the location of the mirror and, if possible, the fracture origin.
(d) Optically (10-100X) examine the fracture surfaces in order to locate and, if possible, characterize the flaw. If necessary, inspect the specimen surfaces near the fracture origin to check for handling or machining damage. If definite characterization of the fracture origin cannot be made, then conduct an optical examination with the purpose of expediting subsequent SEM examination.
(e) Prepare and clean the specimen for scanning electron microscope (SEM) examination.
(f) Carry out SEM examination (10-2000X) of fracture surfaces. Characterize the strength-limiting flaw by its IDENTITY, LOCATION and SIZE (optional). When appropriate, use the chemical analysis capability of the SEM to help characterize the flaw.
(g) At each step, keep appropriate records and photographs in order to characterize the flaw, show its location, and show the general features of the fracture specimen.
(h) For a new material, or a new set of processing or exposure conditions, it is highly recommended that a representative polished section of the microstructure be photographed to show the normal microstructural features such as grain size and porosity.

1.2 Equipment. The following equipment is essential for a comprehensive characterization of the fracture origins in advanced structural ceramics:

(a) Binocular stereomicroscope with adjustable magnification between 10-100X and directional light source, Figure 1. A camera or video monitor system used with this microscope is a useful option, Figure 2.
(b) Cleaning and preparation equipment such as an ultrasonic bath and a diamond cut-off wheel.
(c) Scanning electron microscope with energy or wavelength dispersive spectroscopy, Figure 3.
(d) Various peripheral equipment such as tweezers, grips, compressed air, etc., Figure 4.
(e) A macrophotography camera stand, Figure 5, if a camera system is not available on the stereomicroscope.

1.3 Safety. This practice may involve hazardous materials, operations, and equipment. The handbook does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this handbook to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1.4 Referenced Documents.

(a) ASTM Standard F109-73 (Reapproved 1991), "Standard Terminology Relating to Surface Imperfections on Ceramics", ASTM Annual Book of

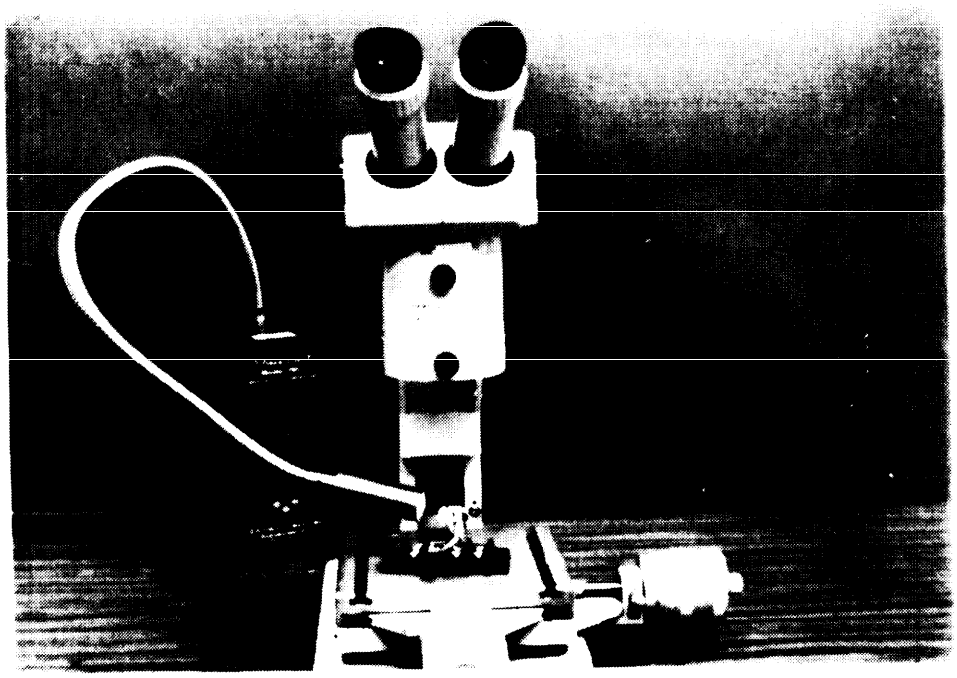

FIGURE 1. Binocular stereomicroscope with directionally adjustable fiber-optical light source and variable magnification between 5x and 80x.

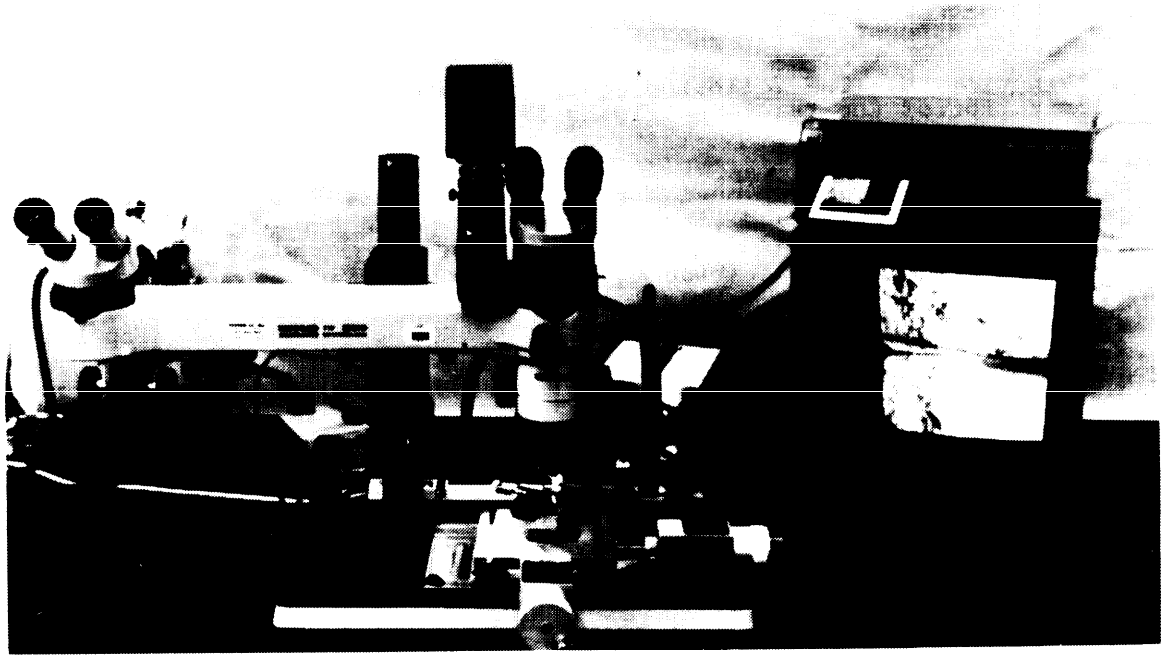

FIGURE 2. Dual station, binocular stereomicroscope with two directionally adjustable light sources, video camera, monitor and instant photographic capability. This type of system is excellent for instructional purposes.

FIGURE 3. Scanning electron microscope with energy dispersive spectroscopic capabilities, low-energy operation and magnification between 20x and 20,000x.

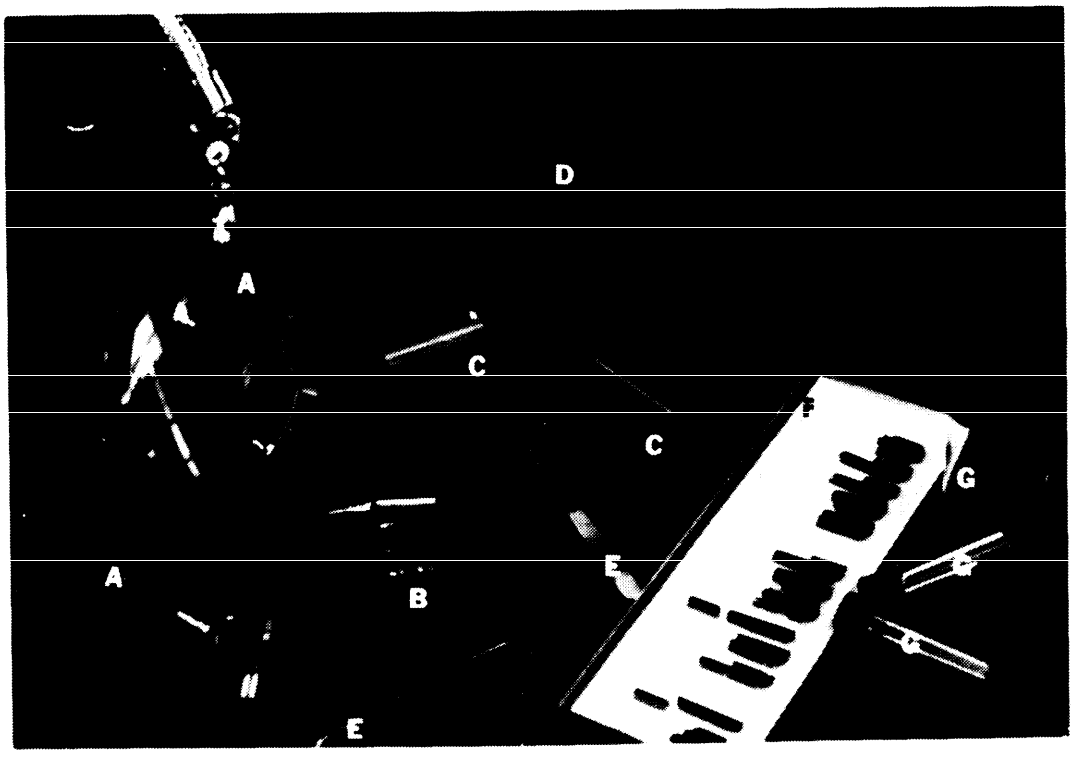

FIGURE 4. Peripheral equipment to assist in fractography and storage of fractured specimens and components. (A) Hand-held and tabletop magnifying glass; (B) Variable-angle grips with compliant surface; (C) Fixtures to support specimens to view machined surfaces; (D) Compressed air; (E) Tweezers or specimen manipulation; (F) Plastic storage trays; (G) Glass vials for storage of fractured specimens prior to SEM analysis.

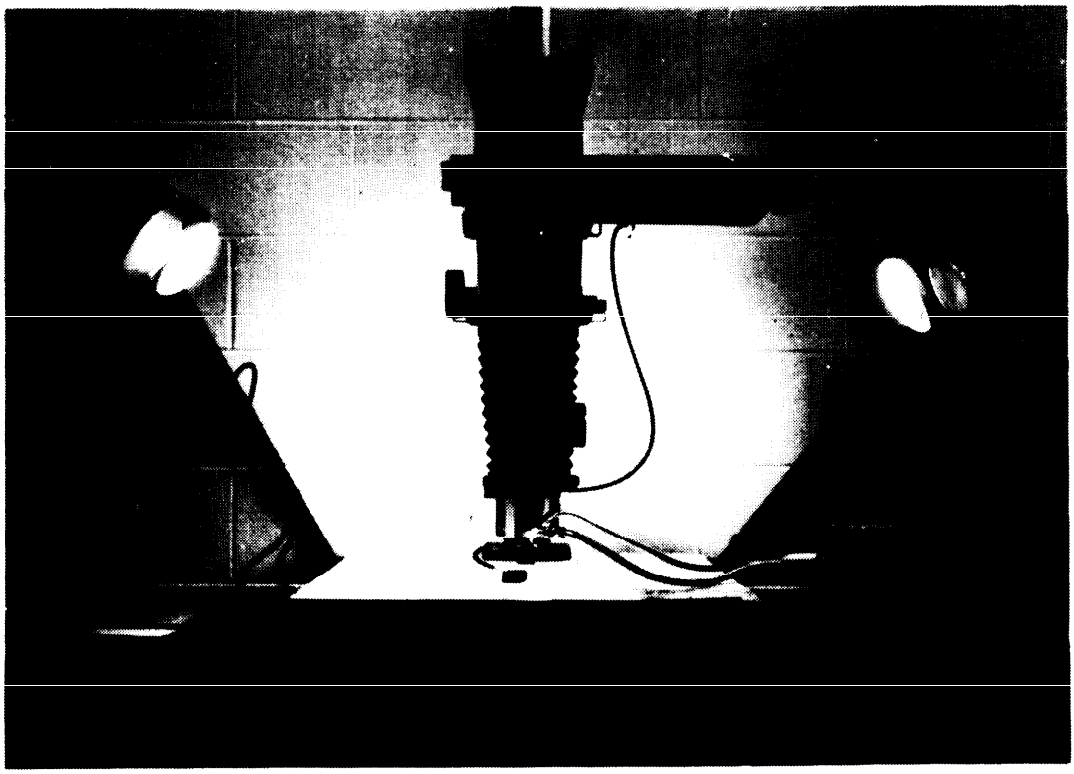

FIGURE 5. Macrophotographic camera stand for instant photographs.

Standards, Volume 10.4.

(b) ASTM Standard C-1209, "Standard Definition of Terms Relating to Surface Imperfections on Ceramics", Annual Book of Standards, Vol. 10.04.

(c) 1984 Ceramic Glossary, The American Ceramic Society.

(d) MTL TR 90-57, "A Proposed Standard Practice for Fractographic Analysis of Monolithic Advanced Ceramics," November 1990.

(e) ASTM Standard C-1211, "Standard Test Method for Flexural Strength of Advanced Ceramics at Elevated Temperatures", Annual Book of Standards, Vol. 15.01.

2. DETAILED PROCEDURES AND CHARACTERIZATION

2.1 Procedure.

2.1.1. General. Location, identification, and characterization of fracture origins in advanced structural ceramics can sometimes be accomplished using simple optical techniques though it more often requires scanning electron microscopy (SEM). It may not be feasible, practical, or even necessary to examine all fracture surfaces with the SEM. The extent of fractographic analysis required will depend upon the following factors:

(a) The conduciveness of the material to fractographic analysis. Some coarse-grained or porous materials will leave no markings that permit failure-origin identification. In other instances (especially in very strong ceramics), the fracture origin is very small and will be difficult to differentiate from the normal

microstructure. Alternatively, a low-to-medium strength material may have flaws easily characterized by optical means thus eliminating the need for SEM analysis.

(b) The purpose of the fractographic analysis: quality control, materials research and development, or design. Table I gives suggested sampling guidelines for medium-to-high strength advanced ceramics.

TABLE I. Suggested Sampling Guidelines.

	1-10X Visual	10-100X Optical	10-2000X SEM
Level 1 -Quality control	Specimens which fail to meet minimum strength requirements.	Specimens which fail to meet minimum strength requirements.	Optional
Level 2 -Quality Control -Materials Development	All specimens	All specimens	Representative specimen -2 of each flaw type -the 5 lowest strength specimens -at least 2 optically unidentifiable flaws
Level 3 -Materials Development -Design	All specimens	All specimens	All specimens, or as many specimens as necessary such that combined optical and SEM characterize 90% (100% for design) of all identifiable origins

2.1.2 To maximize the amount of information obtained from a fractographic exercise, care must be taken in all steps starting with the initial testing of the specimen or component. Note-taking and record-keeping during every step of the procedure will greatly assist the analyst in understanding the flaw populations of a material, comparing the populations between materials, and reviewing the data at some later date.

2.1.3 Specimens that fail during machining, handling, or without measurement of a failure stress, should be examined to determine the fracture origins when feasible. The fact that these types of fracture occurred should be noted and reported.

2.1.4 Mechanical Testing. A few simple precautions should be taken prior to breaking the sample. The test site should be kept clean to minimize pick-up of contaminants. Markings of some sort should be placed on the specimen to maintain a point of reference and to aid in the reconstruction of the specimen. The mark(s) should not damage the sample. A fine pencil line is often sufficient to mark the gauge length (maximum stress) or for a circular specimen an axial, zero-degree reference. Testing that allows the broken fragments of the specimen to hurtle about the test fixture must be avoided. Incidental impact damage to the fracture surfaces can destroy the origin, greatly change its appearance, or cause secondary fractures. A compliant material that covers the hard surfaces of the fixtures and/or prevents pieces from flying about is sufficient in minimizing this damage.

All fragments from the broken specimen should be retained for reconstruction, unless it can be positively established that some pieces are incidental or trivial.

2.1.5 Handling and Storage. Broken specimens should be handled and stored so as to minimize the possibility of damaging and/or contaminating the fracture surfaces. When possible, avoid handling the specimen, especially the fracture surface, with your hands. Body oils and skin fragments can easily change or obscure the character of the fracture surface. During reconstruction of the specimen, minimize rubbing the fragments together. This may abrade or chip the fracture surfaces, and change the surface character. Avoid picking or even touching the fracture surface with sharp instruments and pencils since they will contaminate the fracture surface. Pencil "lead" is a mixture of carbon and clay particles which can be difficult to remove and under the SEM looks convincing as failure origins (see Figure 6). The laboratory environment contains a myriad of materials such as clays, waxes, adhesives and resins that should be avoided wherever possible. Many of these materials, once they are affixed to the specimen, are very tenacious and often impossible to remove. Figures 7-10 show some other contaminants when viewed with an SEM. The specimen must be stored in a clean and orderly fashion as much time can be lost trying to sort out mixed-up specimens. Many container types are readily available for storage, Figure 4 and Table II.

TABLE II. List of some commonly used storage media for fractured specimens or components, see Figure 4.

STORAGE MEDIA	ADVANTAGE	DISADVANTAGE
Envelopes	Convenient for notes, minimal space required, inexpensive.	Lint contamination, specimen is free to move.
Glass Vials	Very clean, reusable	Hard surface could cause secondary fracture, specimen free to move, expensive.
Plastic Trays	Clean, inexpensive, saves space.	Plastic contamination, specimen free to move.
Tape	Inexpensive, mark primary fracture w/notes, maintain reconstructed specimen.	Adhesive contamination, limited shelf life.

2.1.6 Visual Inspection (1-10X). One of the principal goals of the visual inspection is to find the primary fracture surface, the general region of the strength-limiting flaw and, if possible, the fracture mirror. This inspection under ordinary light is integral to normal specimen reconstruction and often to optical microscopy (Section 2.1.7). Hand magnifiers can be helpful. Specimen reconstruction is valuable in observing the crack(s) and crack branching patterns which, in turn, helps determine the primary fracture surface. Special emphasis should be on determining whether the fracture pattern indicates misalignments or breakages at test grips (in tension), at stress concentrators (neck region in tension) or load application points (in flexure and disk tests). (For further information see "Ceramic Fracture Features, Observations, Mechanism and Uses," by Rice (1984) in the Bibliography.)

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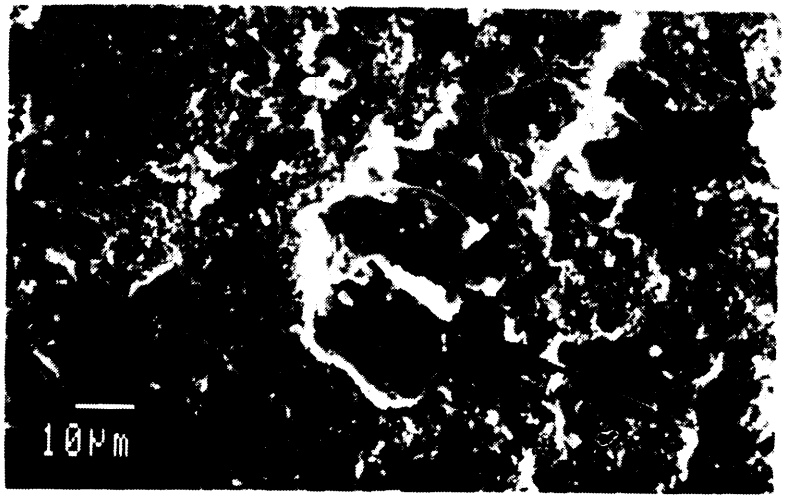


FIGURE 6. Contamination from particles of graphite from a common "leaded" pencil. These typically appear as globules, but since pencil graphite usually has a clay binder, it must be treated with caution.

FIGURE 7. Contamination from a smear of masking tape resin (white arrows) near a chamfer. Masking tape is sometimes used to hold pieces of a fractured specimen together, but should be avoided on the fracture and tensile surfaces. The smear blends into the fracture surface and is partially transparent to X-rays as shown. An energy dispersive analysis identified the smear as having potassium, chlorine and sulphur. Trichloroethylene is an effective solvent to remove the resin.

FIGURE 8. Contamination from particles of paper lint (black arrows) from a common manila specimen envelope. These are easy to blow off or eliminate by a sonic bath.

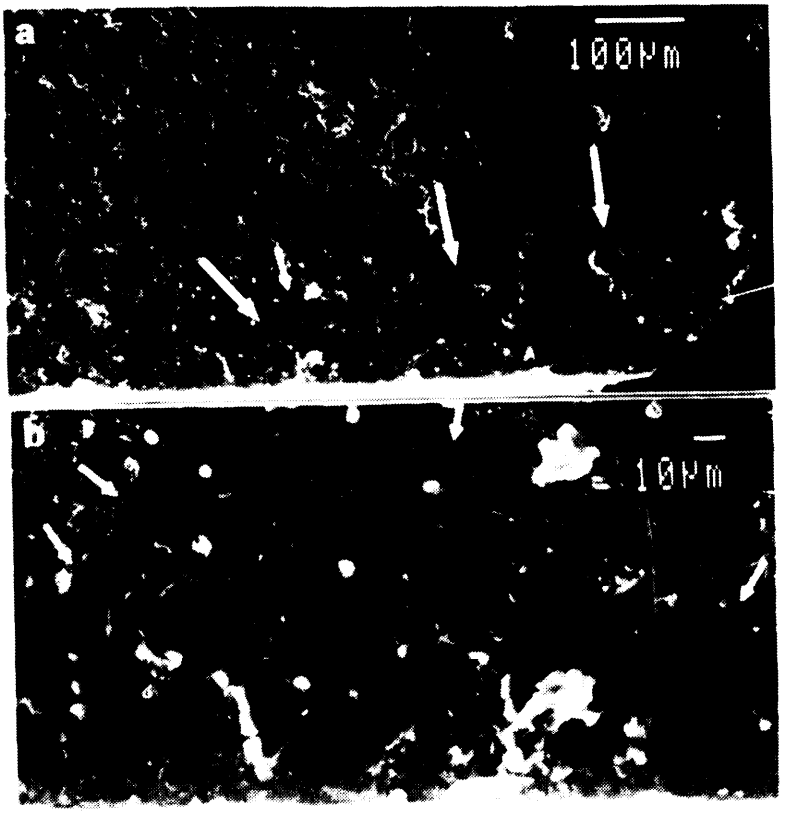


FIGURE 9. What might be the most pernicious contaminant in the fractographic laboratory: mounting clay. The white arrows in (a) show a region where clay was dabbed on with tweezers. The clay appears to be a genuine inclusion that blends directly into the underlying ceramic. It is extremely difficult to remove once it gets onto the specimen and it looks quite appropriate on the fracture surface. It should not be used. (b) is a close-up of the region of the small arrow from (a). An energy-dispersive analysis revealed Si, Al, and Ti. The Si is indistinguishable from the silicon nitride specimen.

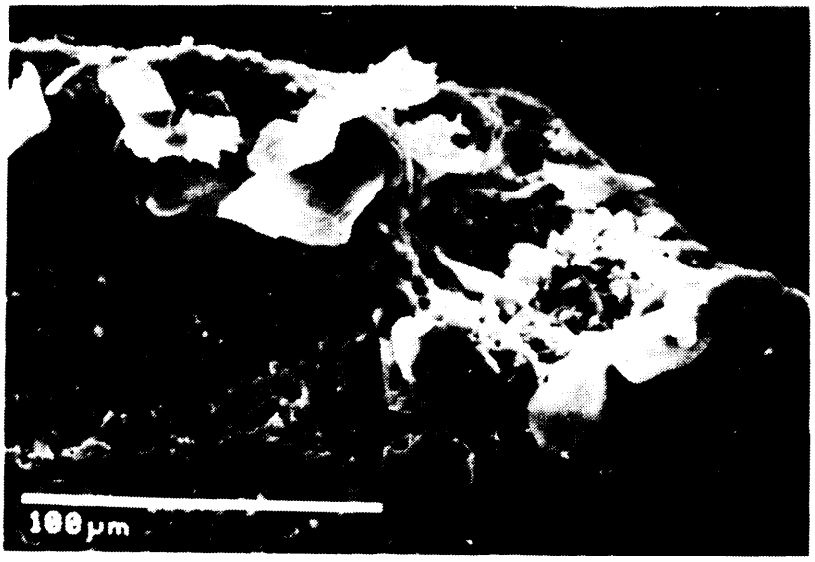


FIGURE 10. Contamination from human skin. (Courtesy of A. Pasto, GTE Laboratory, now with Oak Ridge National Laboratory).

(a) Multiple fractures (some with origins) are common to high strength ceramics that store large amounts of elastic energy during testing. Upon failure, this energy is released and reflects from free surfaces back through the body of the material causing additional failures. Many of the secondary fractures can be quickly eliminated. Misalignment or deviation from the assumed stress state can be discerned by primary fracture surfaces that are at an irregular angle (not 90°) to the anticipated principal stress. Branching angles can be helpful in detecting multiaxial stress states. Figures 11 and 12 show many potential fracture patterns in some common test specimens.

(b) Depending upon the test specimen or component geometry and the stress states in the body, the crack pattern can range from the very simple to quite complex. A hierarchy or sequence of crack propagation can assist in back-tracking to the fracture origin. Crack branching can be used to determine the direction of crack propagation, see Figure 13. A traveling macro-crack will typically branch into successively more cracks and will rarely rejoin another crack to form a single crack. A crack that intersects another crack at angles close to 90° will usually be a secondary crack and will not contain the fracture origin. For specimens that do not show macroscopic crack branching, incipient branching in the form of shallow cracks can often be found along the edge of the main crack on the exterior surface. As with the macroscopic cracks, the angle of these shallow cracks in relation to the main crack indicate the local direction of crack growth. Vicinal illumination and/or dye penetrants can be used to make these cracks more easily discernible.

(c) Dense, fine-grained or amorphous ceramics are usually very conducive to fractography and will leave distinct fracture markings (hackle and mirror) which will aid in locating the origin, see Figure 14. Hackle on the fracture surface refers to lines running in the direction of crack propagation, and separates parallel but non-coplanar portions of the crack surface. Coarse-sized hackle lines

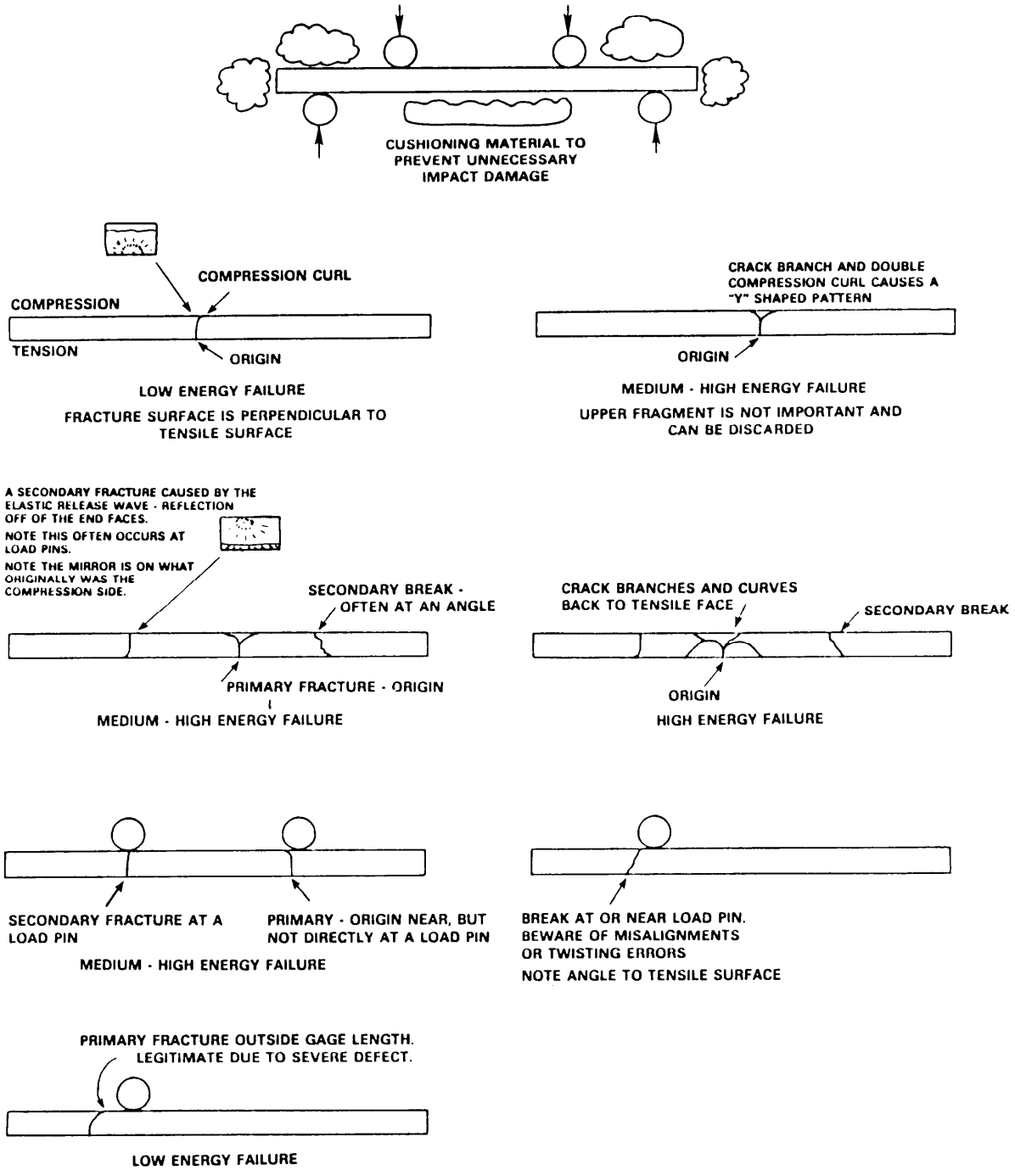


FIGURE 11. Typical fracture and crack patterns of flexure specimens.

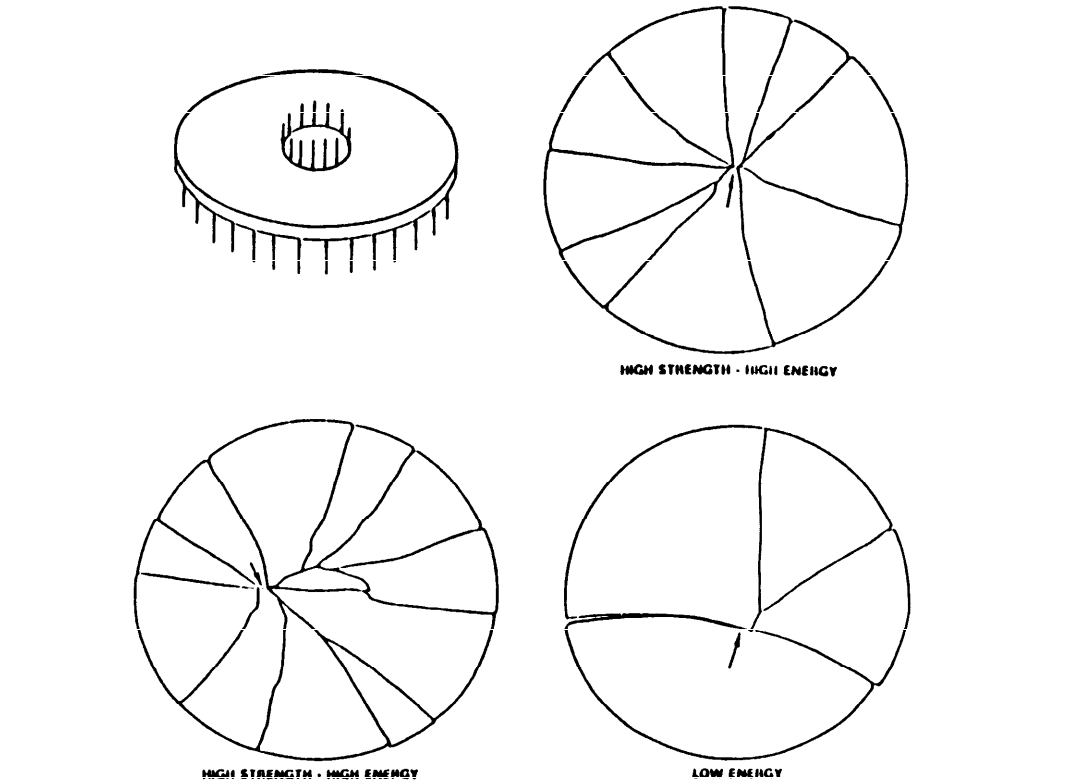


FIGURE 12. Typical fracture and crack patterns of: (a) biaxial flexure specimens and (b) diametrical compression specimens.

and ridges on the fracture surface are extremely helpful in locating the general vicinity of a fracture origin (Figure 14a). They will radiate from, and thus point the way back to the fracture origin. They are best highlighted by low incident angle lighting which will create useful shadows. Fracture mirrors are telltale features that are centered on strength-limiting flaws. If the specimen or component is highly stressed, and the material is fine-grained and dense, a distinct fracture mirror will form as shown in Figures 14b and 14c. On the other hand, lower-energy fractures and those in coarse-grained or porous ceramics will not leave a fracture mirror. The coarse hackle markings and Wallner lines or ridges can still be used to determine the vicinity of the fracture origin,especially with oblique lighting. (MTL TR 90-57 and several references in the Bibliography illustrate and discuss further means of locating the fracture origin.)

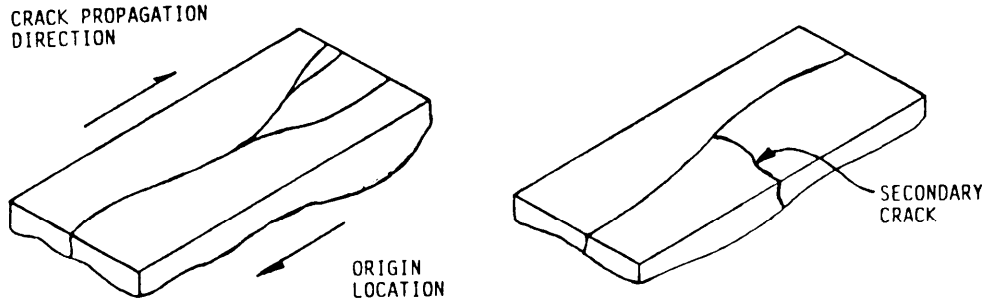


FIGURE 13. Schematic of typical fracture patterns showing crack branching.

2.1.7 Optical Microscopy (10-100X). All specimens should undergo optical examination after fracture. This is often performed in conjunction with the visual inspection. The purpose of the optical examination is to locate the fracture origin on the primary fracture surface (Table I, Levels 2-3, (see Section 2.1.6(b)) and attempt to characterize the flaw. If characterization is not possible during this step, the optical examination helps to minimize the time spent during the subsequent examination using the scanning electron microscope.

(a) If the visual inspection has not been performed to identify the primary fracture surface(s), follow the procedures outlined in sections 2.1.6 (a) and (b) using the optical microscope.

(b) A stereomicroscope is preferred for examining fracture surfaces due to its excellent depth of field. Viewing will be most effective in the 10-100X range since at higher magnifications the depth of field is reduced. A traversing stage coupled with crosshairs or a graduated reticule in the eyepiece are useful for measuring the size and/or area of the mirror and, if possible, the flaw. Illumination should be provided by a common microscope light source with adjustable intensity and angle of incidence to provide a means of variable lighting. These variations can highlight aspects of the fracture surface which may be hidden if one is restricted to a single view.

(c) The specimen should be mounted to view the fracture and external surfaces. A simple alligator clip attached to a stand and having a

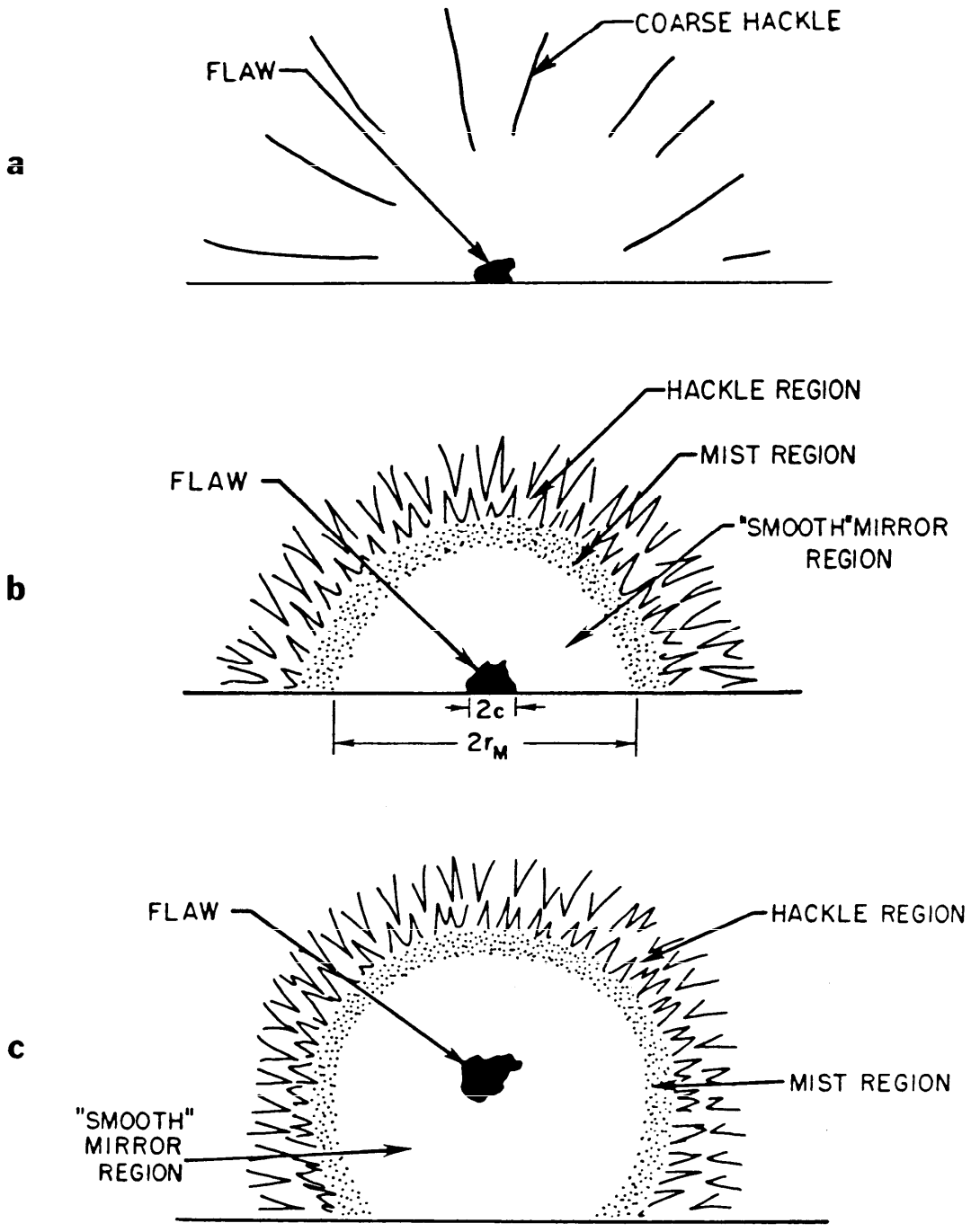


FIGURE 14. Schematic of fracture surfaces of advanced ceramics which failed in a brittle manner showing (a) the coarse hackle lines which emanate from the origin (b) a flaw located at the surface and (c) a flaw located in the volume. NOTE: the mirror can be centered around a portion of the flaw and not the entire flaw. In ceramic terminology "smooth" is a relative term.

compliant coating or sheath covering the teeth, provides a sturdy grip (Figure 4b) for examination. A specimen holder, Figure 4c, can be created to hold the entire specimen at a convienient working height to view the external surfaces. A flat or a vee groove in the holder will aid in the examination of the surfaces and edges for handling or machining damage. DO NOT use clays or waxes for mounting because these materials can contaminate the fracture surface and are very difficult to remove. Surface contaminants such as lint and dust, can easily be removed with canned or filtered compressed air. Viewing the matching primary fracture surfaces simultaneously can expedite and improve the quality of the analysis since what might appear to be a pore on one half may show an agglomerate on the other (i.e., flexure specimens should be mounted tensile surface-to-tensile surface). Care must be taken so that extraneous damage is not created.

(d) At the lowest magnification, locate the mirror(s) using the hackle on the fracture surface. In high-strength, fine-grained and dense ceramics this will be approximately centered in the fracture mirror as shown in Figures 14b and 14c. Hackle lines and ridges will be very helpful since they will radiate outward from the fracture origin and mirror. As discussed in Section 2.1.6, low-energy fractures or fractures in porous or coarse-grained ceramics may not lead to mirror formation, but the same principals of using the hackle lines apply. If the flaw is not evident on the fracture surface, the external surfaces should be viewed for handling or machining damage. Twist hackle lines are especially helpful and occur when a crack encounters a principal stress field that is not perpendicular to the original plane of fracture. Twist hackle commences as finely spaced parallel lines which usually merge in the direction of crack propagation, giving rise to the well known "river pattern" as shown in Figure 15.

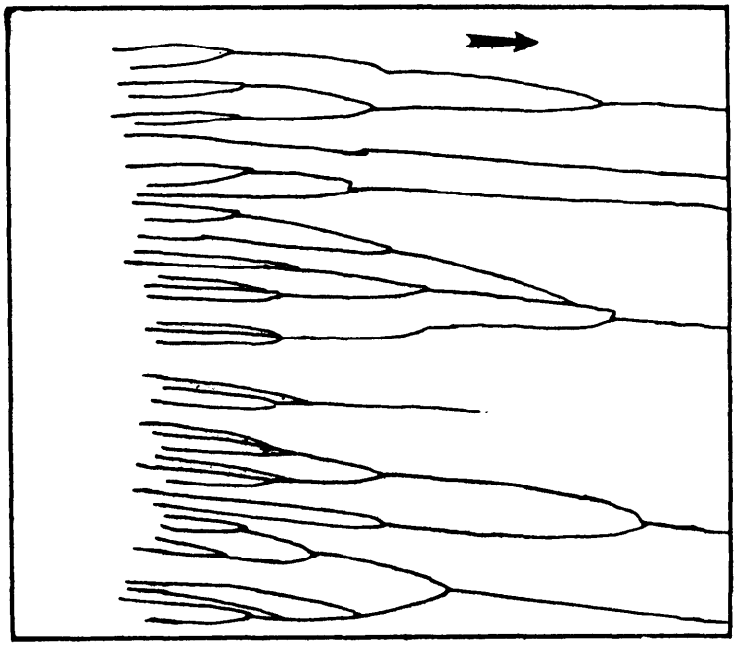


FIGURE 15. Schematic of a river pattern with an arrow showing the direction of crack propagation.

NOTE: The merger of twist hackle in the direction of propagation is usually opposite to the tendency of macrocracks to diverge as discussed in Section 2.1.6 (a). These features are usually well defined in glasses and very fine-grained, fully dense polycrystalline ceramics. Such twist hackle often occurs on individual grains in coarse-grained polycrystalline ceramics. (See Failure Analyses of Brittle Materials, by Frechette (1990) in the Bibliography for a discussion and illustration of these features.)

(e) Characterize the strength-limiting flaw as outlined in Section 2.2. Record observations pertaining to features specific to the lighting, such as color and reflectivity. These records should include, but not be limited to, notes, sketches and photographs. Although this extra step may seem time consuming, it often leads to greater efficiency in the long run. These records are extremely useful for publication and minimizing the search time with the SEM. The latter point can not be underestimated. Novices often lose much time searching for the origin or examining the wrong area. SEM images are quite different from optical images and a reorientation time is sometimes necessary.

NOTE: It is sometimes necessary, especially with a new, unfamiliar material, to reexamine all of the specimens after the initial optical examination, since a particular flaw is sometimes overlooked or misidentified during the initial viewing.

(f) Photomacrography (Figure 5) is flexible in that control of overall resolution and depth of field is possible and the system is not expensive. On the other hand, the convenience of having a camera mounted directly to the binocular microscope, photomicrography (Figure 2), is a great time saver. With built-in zoom ranges of 5 to 1 and beam splitters, it is possible to frame, focus and shoot quickly and efficiently. Modern built-in video cameras with monitors can be coupled to color printers which give photograph-size hard copies in less than one minute and without the need to deal with film and negatives. These video images, with appropriate software, can also be stored in a digital format (i.e. floppy or laser disk). Such images can then be retrieved and displayed on a video monitor or on the SEM monitor. This is a very efficient means of coupling the two methods, and enhanced productivity will result. (The Metals Handbook, Vol. 12, on Fractography has some helpful tips on lighting techniques for photomacrography.)

(g) For some translucent ceramics it is useful to coat the fracture surface with evaporated carbon or sputtered gold-platinum prior to optical examination. It will often improve the visibility of some crack propagation patterns, eliminate subsurface reflections and improve the quality of the photos taken of the fracture surface.

NOTE: Be careful! In very high-strength advanced ceramics thick coatings can cover or obscure submicron pores and subtle features. In these instances it is suggested that the SEM examination be carried out on uncoated specimens at a low voltage prior to this coating.

(h) In some applications, replicas of a fracture surface can be used advantageously. Although extra preparation steps are involved, cellulose acetate, polyvinyl chloride (PVC), or silicon elastomer replicas can record important features, both for optical and SEM examination. Advantages include: eliminating obscuring subsurface features in the optical microscopy of transparent or translucent ceramics; provision of an easily stored record of the fracture

surface of a critical specimen; greater accessibility of curved surfaces to high-magnification optical study; or study of unique specimen geometries. A disadvantage is the risk of altering the fracture origin (e.g. pull-out of an agglomerate). (See Failure Analysis of Brittle Materials, by Frechette (1990) in the Bibliography.)

2.1.8 SEM Examination. Optical microscopy is not always adequate to characterize flaws. This is especially true for strong materials which have very small mirror regions and flaws. Nevertheless, it is an essential adjunct to SEM, since certain features such as a telltale color or reflectively are completely lost in electron-microscopic viewing. Once optical fractography is complete and the flaws are characterized as well as possible, a subset of specimens should be prepared for analysis by scanning electron microscopy. Determination of the number of specimens which will comprise the subset will depend on the intent of the analysis, see Table I.

2.1.8.1 Preparation.

(a) Specimens should be cut to a consistent height that allows for ease of installation and movement in the SEM. The cutting should be done wet so as to flush specimen and cutting wheel debris away. They should be cut as flat as possible to eliminate problems due to excessive tilt, although a slight tilt backwards can be beneficial on flexure bars so that not only the fracture surface but the tensile surface can be viewed. During the cutting process, every possible measure should be taken to prevent damage to the fracture and external surfaces.

(b) Cut specimens should be ultrasonically cleaned in water or an agent to remove any cutting solutions or other contaminants. Several cleaning agents are listed in Table III. Specimens should then be rinsed in a quickly evaporating solvent to remove any final residue. Solvents such as acetone or ethanol are recommended for this step. Once cleaned, each specimen should be properly labeled and placed in a separate glass or plastic container to prevent further contamination. All subsequent handling should only be done with tweezers or lint-free gloves and the specimens should not be brought into contact with tapes, clays, waxes or fibrous materials.

(c) Coating of a ceramic is widely used to reduce "charging" of the surface. New SEM equipment is capable of operating at low accelerating voltages which minimizes charging. If such equipment is available, and time permits, it is suggested that the fracture surfaces first be viewed without a coating. The use of low accelerating voltages can provide a better view of the surface topography. However, coatings can enhance resolution and contrast. If a coating is needed it should be carefully applied. Coatings which are too thick or multiple coatings may obscure features and lead to misinterpretation of the flaws.

(d) A thin coating, typically 5 nm, of carbon or gold-palladium should be applied onto the specimens using a vacuum evaporator or sputter coater. The gold-palladium coating is recommended for imaging purposes since it provides better conductivity. Carbon coatings prepared by evaporation are preferred for x-ray emission analysis because the carbon is nearly transparent to x-rays. A thermal evaporation method for metal coatings can be used with a tilted specimen relative to the metal source, creating an oblique deposition. This can be used to create shadowing to highlight very

machining damage. Twist hackle flaws are especially helpful and

fine markings on the specimen. (See "Fractography" in Metals Handbook (1987) in Bibliography.)

TABLE III. Cleaning Agents.

Agent	Advantages	Disadvantages
Trichloroethylene	-Removes oils, adhesives, grease -Fast cleaner	-Health hazard with excessive use -Produces chlorofluorocarbons -Skin & eye irritant
Xylene & Toluene	-Removes oils, adhesives, grease	-Severe eye irritant -Skin irritant -Flammable
Acetone & Ethanol	-Limited health hazard -Readily available -Inexpensive	-Does not remove all oils, adhesives, grease -Longer cleaning time required -Residual film from acetone
Cleaning Powder mixed w/distilled water & heated (e.g. Alconox)	-Inexpensive -Readily available	-Potential for soap residue to remain on the specimen -Difficulty in removing most oils, adhesives, grease -Longer cleaning time required

(e) Specimens can be mounted for examination either singly or multiply on stubs using conductive paints. Specimens should be mounted with the cut surface down and care should be taken to avoid getting conductive paint on the fracture surface or upper portion of the external surfaces. The specimens should be mounted in a systematic fashion to permit rapid orientation by the observer. For example, flexure bars should be aligned with their tensile surfaces the same way. If a pencil is used to mark the specimen orientation or the approximate location of the origin, exercise care that no traces of the pencil material get on or near the fracture surface. Once mounted, specimens should be sprayed with compressed air to remove any lint or lightly clinging debris.

2.1.8.2 Examination. The specimen should be oriented at the lowest possible magnification and the fracture mirror located. It is often useful to use an optical photograph as a guide when trying to locate the fracture mirror. Adjust the contrast and brightness to provide the maximum amount of information. The entire surface should be photographed at a low magnification to provide a frame of reference for later work. Conventional practice is to orient the specimen image in a consistent manner, i.e. place the tensile surface of all flexure specimen at the bottom of the photograph.

(a) The SEM can be used either in the secondary electron or backscattered electron modes. The former gives a fully illuminated image of the surface topography, with better spatial resolution. The backscatter mode provides greater height contrast due to its sensitivity to the detector orientation. Features not in direct line with the detector are darker or even in shadow. Backscattered electrons carry both topographic and compositional data since higher atomic weight elements will give a greater electron signal. This is valuable for detecting second phases and inclusions. The

topographic and compositional signals can be separated for further analytical flexibility (See Healey and Mecholsky, "Scanning Electron Microscopy Techniques and Their Application to Failure Analysis of Brittle Materials" (1984) in the Bibliography).

(b) Once the specimen is properly oriented, the flaw can be located by using the fracture mirror and hackle lines, if present, as references. If there is uncertainty in determining the strength-limiting flaw, an <u>estimate of the flaw size</u> can be made by noting that outer mirror (mist-hackle)-to-flaw radius ratios are typically about 13 to 1 and the inner mirror (mist) ratio is between 6 to 1 and 10 to 1. Alternatively, flaw size can be estimated if the fracture toughness of the material is known (at the size scale of the microstructure):

$$c = \left(\frac{K_{IC}}{\sigma Y} \right)^2 \qquad (1)$$

Where c is the flaw radius, K_{IC} is the fracture toughness, Y is the shape factor for the flaw and σ is the failure stress.

<u>NOTE</u>: With the exception of some types of machining damage, most ceramic flaws are too irregular to permit accurate shape factor determination. Also, at the scale of the microstructure, it may be unclear whether a single crystal or polycrystal fracture toughness should be used. (Flaw severity (Y) = 1.13 for a penny shaped flaw in the interior; Y = 1.4 for a semi-circular flaw at the surface; and Y = 2.0 for a long surface crack.)

Although in most laboratory test specimens the stress at failure is known, this may not be the case for components. In such cases, an estimate of failure stress can be obtained from the mirror size according to:

$$\sigma = A \frac{1}{\sqrt{r}} \qquad (2)$$

where r is the mirror size (radius) and A is the mirror constant (mirror-mist, or mist-hackle). (Further details and references with mirror constants for a range of ceramics are given in the Fracture Mirror section of the Bibliography.)

(c) After locating the flaw, characterize it according to section 2.2. It may be necessary to acquire an energy- or wavelength-dispersive x-ray analysis of both the flaw and the background to determine whether there are any chemical differences. Conventional energy-dispersive x-ray analyzers are used to obtain an x-ray spectrum for sodium (z=11) and higher atomic number elements. The spatial resolution is of the order of 1 μm with a penetration of 1-2 μm into the specimen. Wavelength dispersive x-ray analyzers are available which can detect elements down to boron (z=5). These are less commonly used since they require extremely flat and smooth surfaces and crystal spectrometers that are tuned to specific wavelengths (elements). Direct correlations between structure and composition can be made by directing x-ray returns onto the SEM monitor thereby creating an x-ray dot map of the elements present.

(d) In some cases, such as when handling or machining damage are suspected, it may be necessary to tilt the specimen slightly in order to view a portion of the external surfaces. Sometimes a 180°

rotation can help discern subsurface machining damage cracks. Once the flaw is located, a photograph should be taken at a magnification in which the flaw accounts for half of the frame. A photograph showing the fracture mirror and some hackle is also very helpful for later reassessment of a flaw. This will typically be in the 200-1000X range. In many cases, photographs at varying magnifications are necessary to yield all the required information regarding the failure of the specimen. It is recommended that a consistent set of magnifications and orientations be used to permit easy comparative assessments between specimens.

(e) It is often useful to maintain notes on the examination including: sketches of the fracture surface, notes on the flaw type and appearance, locations of photographs taken, magnification and reference numbers of photographs, whether or not x-ray spectra were acquired, and the location used to acquire the spectra. When maintaining notes of acquired x-ray spectra, always include the accelerating voltage, probe current, magnification, dead time, counts and scan time, working distance and whether the spectra was taken in scan or spot mode.

2.1.9 Recording Fractographic Observations. If feasible three photographs should be taken of each fracture surface (one set per pair of fracture halves is adequate). As seen in the schematic Figure 16 these should include, but not be limited to:

a. A photo (optical or SEM) of the entire fracture surface;
b. A photo of the entire fracture mirror and some surrounding detail;
c. A photo of the flaw.

It is highly recommended that a representative polished section be made and photographed to reveal the normal microstructure and allow an assessment of whether flaws are abnormal or normal microstructural features.

2.2 Flaw Characterization

2.2.1 General. The fracture origin shall be characterized for each specimen by the following three attributes: **IDENTITY, LOCATION,** and **SIZE** (optional) as summarized in Table IV.

TABLE IV. Flaw Characterization Scheme

IDENTITY	**LOCATION**	**SIZE (Optional)**
Nomenclature & spatial distribution; volume or surface	Volume (bulk), Surface, Near-Surface, Edge	Estimate of the major and minor axes

Origins are either inherently volume-distributed throughout the bulk of the material (e.g. agglomerates, large grains or pores) or inherently surface-distributed "on" the material (e.g. handling damage, pits from oxidation or corrosion). The volume-distributed origins in a ceramic material can, in any test specimen, be located in the bulk, at the surface or near-to-the-surface, or at an edge of a specimen as seen in Figure 17. The variety of locations for volume-distribued flaws is a consequence of the random sampling procedure incurred in preparing test specimens (e.g. machining). (The Bibliography contains several excellent sources concerning flaws in ceramics, their formation, and their characterization.)

the formulation for devil's food cake mix shall be as follows:

statistically differentiate flaw populations!

(b) In some instances, it is useful to specify further the flaw location if it is near the surface, but not in direct contact. This location category is be termed near surface (NS). This additional specification of location is important for fracture mechanics evaluation of flaws and service-performance issues but not to differentiate the inherent flaw population. For example, some near-surface flaws may be more susceptible to time-dependent crack growth than equivalent flaws in the bulk. Near surface flaws may also be likely to link up with surface machining and/or impact damage or to extend subcritically to the surface prior to catastrophic fracture. Due to the difficulty in defining "near surface" and because this location category may only be applicable to design (Table I, Level 3) it is suggested that the analyst consult the design engineer for a definition before continuing with SEM fractography. The criteria, with supporting reasoning, shall be included in the report section. The proximity to the surface shall be noted by estimating the perpendicular distance from the surface to the closest point of the flaw.

2.2.4 Flaw Characterization - SIZE (optional).

(a) Flaw size characterization is only required by this MIL HBK in a qualitative sense as necessary to identify the general nature of flaws (i.e., the 20 μm pore versus the 1 μm porosity). For equiaxed flaws the mean diameter shall be reported and for nonequiaxed flaws the major and minor axis shall be reported.

NOTE: Precise flaw measurements are usually not helpful since the flaws' true size may not be revealed on the fracture surface, and fracture mechanics analyses of most flaws are not possible due to their complex shape. (An important exception is machining damage wherein flaw size measurements may be very useful for estimates of fracture toughness).

2.3 Report.

2.3.1 General. A sample reporting format is shown in Figure 18. The report shall contain the following:

a. Fractographer's identity;
b. Equipment used;
c. Overall flaw types identified;
d. The Flaw Identity, Location, Size (optional) and the mode of viewing (optical vs SEM) for each specimen.
e. The inspection criteria (e.g., as per Table 1);
f. Supplemental observations such as transgranular or intergranular fracture (or the approximate ratio of each) are highly encouraged.

2.3.2 To the extent possible, couple the fractographic observations directly to process history and resultant microstructure. Representative micrographs of polished sections of the microstructure showing porosity and grain size distribution are highly recommended.

2.3.3 Couple the fractographic observations directly to the mechanical test results. Fractographic montages and labeled Weibull or other strength graphs (figures 19-21) are an exceptionally versatile means of accomplishing this. Montages present the fractographic results in a comprehensive manner.

Strength Information			Fractographic Information				
ID	Stress	Comments	Rank	IDENTITY	LOCATION	SIZE	VIEWING MODE

Date:_____ Fractographer:_____

Equipment Used:_____

Inspection Criterion:_____

Overall Flaws Types Identified: _____

Microstructure: Grain Size _____; Porosity _____

Other: _____

Comments:_____

_____

_____

FIGURE 18. A sample reporting format. This reporting format is consistent with that in ASTM Standard C1211 "Standard Test Method for Flexural Strength of Advanced Ceramics at Elevated Temperatures".

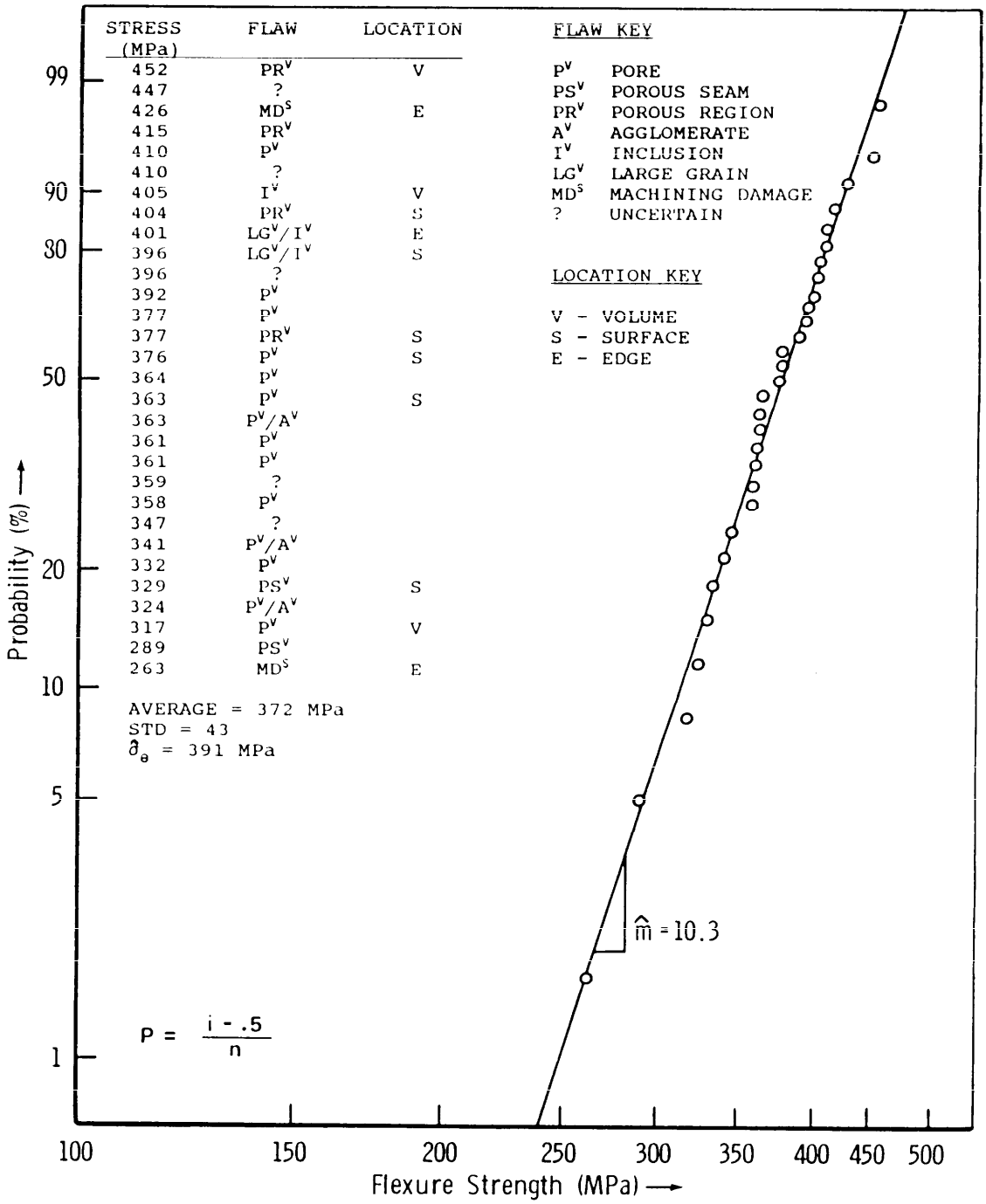


FIGURE 19. A labelled Weibull graph including a listing of strength values, identified flaw types and locations. Flaw and location keys are added for ease in interpretation of both statistical strength distributions (Normal & Weibull). The majority of the origins identified in this example are volume-distributed, although as the location column shows some of the individual flaws were located at the specimen surface. The superscript "V" stands for inherently volume-distributed flaws and a superscript "S" for inherently surface-distributed flaws.

ons (e.g., machining). (The Bibliography contains several) ...incurred in preparing test specime

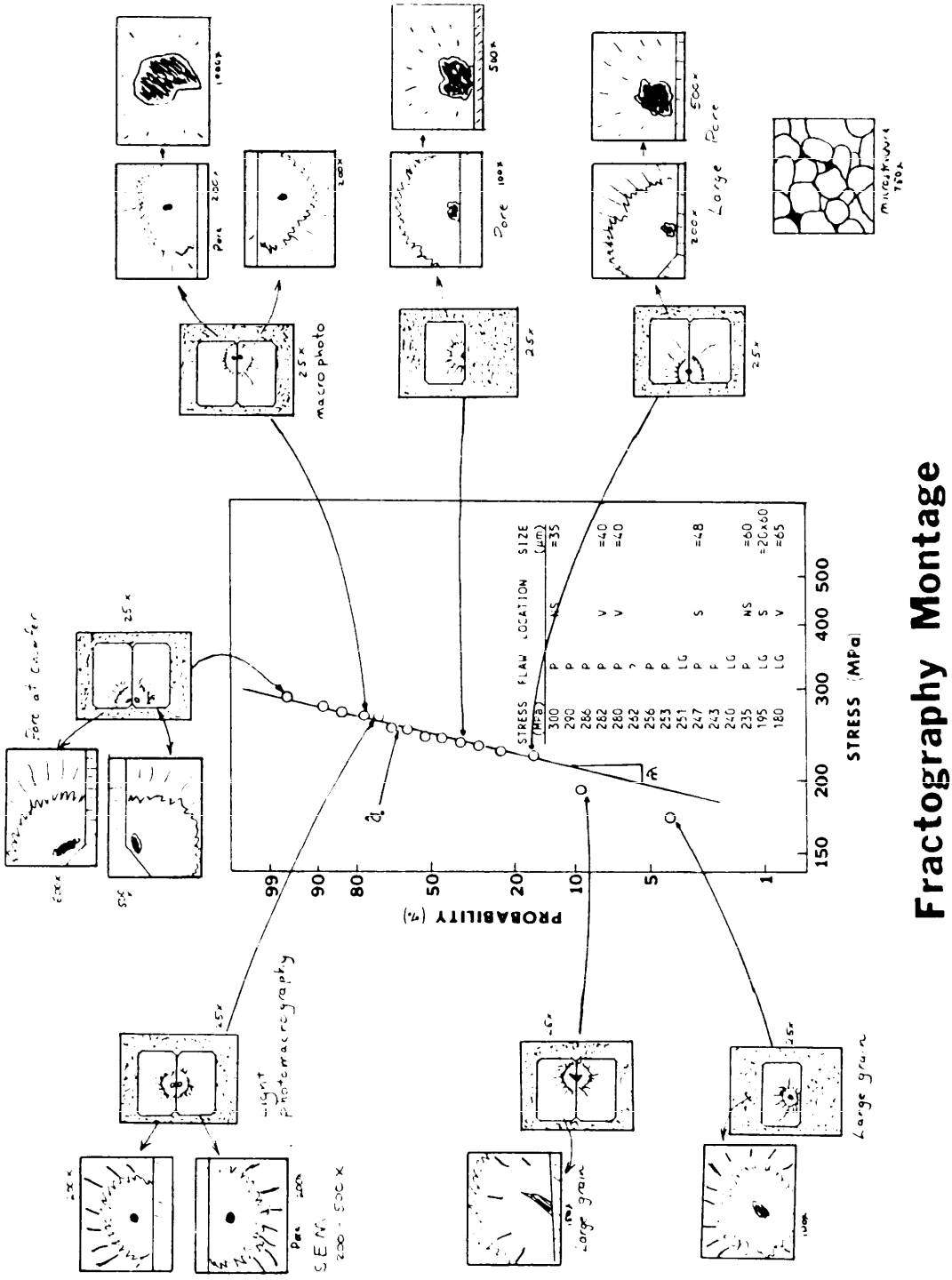


FIGURE 20. A schematic of a working fractographic montage linking fractographs and strength plot. This worksheet is beneficial for quick calculations of mirror-flaw size ratios and approximate fracture toughness calculations. Photos of microstructure including porosity and grain size should also be included on the montage.

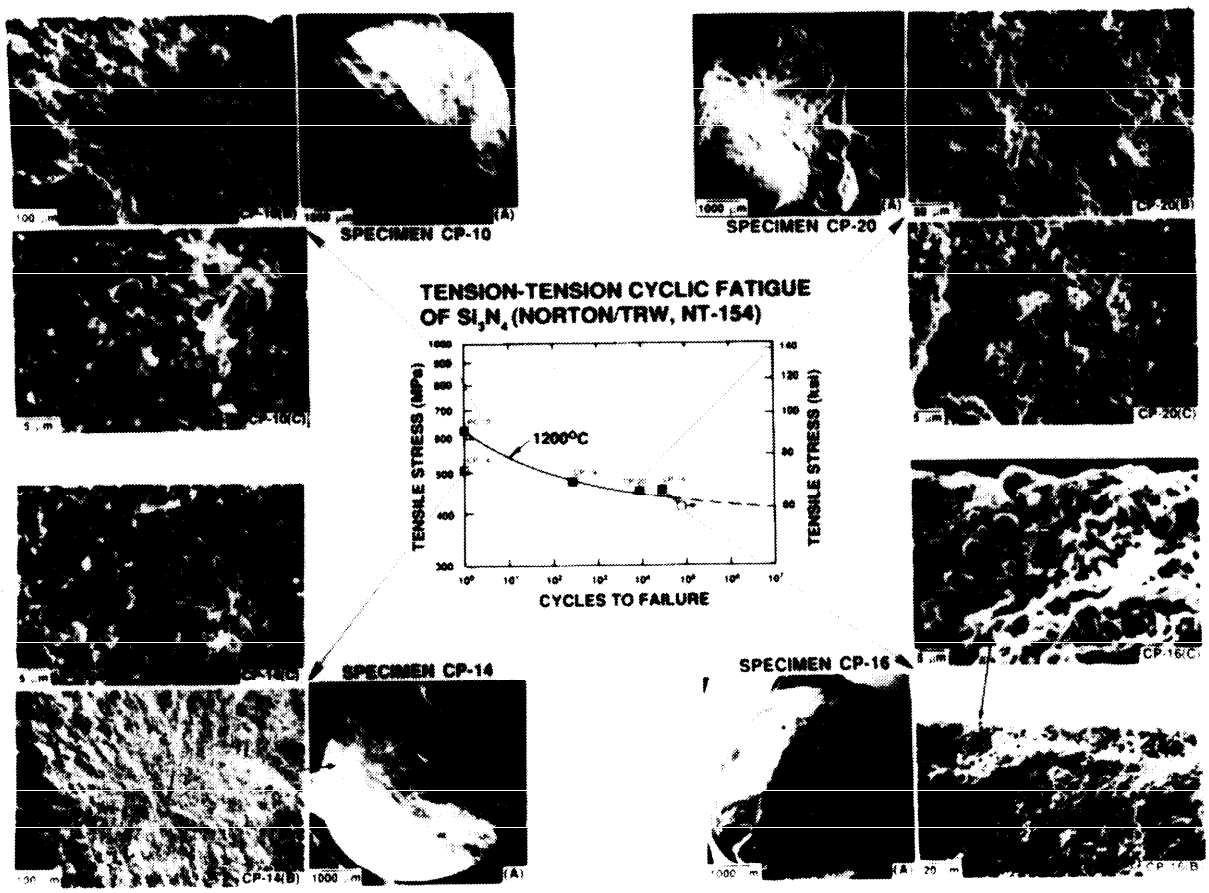


FIGURE 21. Example of a publication quality montage of tension fatigue data. Note that the specimens shown have 3 fractographs each: overall fracture surface, fracture mirror, and flaw close-up. (Courtesy of K. Liu, Oak Ridge National Laboratory).

3. DEFINITIONS

3.1 General. The following definitions are given as a basis for identifying the flaws which are common to advanced structural ceramics. However, it should be recognized that flaws can manifest themselves differently in various materials. The photographs shown in Figures 22-35 gives examples of flaws defined below.

Definitions noted with a * are from the 1984 Ceramic Glossary and those with # are from ASTM F109-73 (Reapproved 1991) "Standard Terminology Relating to Surface Imperfections on Ceramics".

NOTE: In the flaw codes a superscript "V" stands for inherently volume-distributed flaws and a superscript "S" for inherently surface-distributed flaws.

Flaw	Code
3.2 **INHERENTLY VOLUME-DISTRIBUTED FLAWS.**	
3.2.1 Pore. A discrete cavity or void in a solid material.	P^V
3.2.2 Porous seam. A 2-dimensional area of porosity or microporosity.	PS^V
3.2.3 Porous region. A 3-dimensional area of porosity or microporosity.	PR^V
3.2.4 Agglomerate. A cluster of particles, or whiskers or a combination thereof, into a larger solid mass.	A^V
3.2.5 Inclusion. A foreign body from other than the normal composition enclosed in the matrix.*,#	I^V
3.2.6 Second-phase inhomogeneity. A microstructural irregularity related to the non-uniform distribution of a second phase, e.g., an atypically large pocket of a second phase or a zone of composition having a crystalline phase structure different than the matrix material.	$2P^V$
3.2.7 Large grain(s). A single (or cluster of) unusually large grain(s).	LG^V
3.2.8 Crack. A line of fracture without complete separation*,#	CK^V

Flaw	Code
3.3 **INHERENTLY SURFACE-DISTRIBUTED FLAWS.**	
3.3.1 Machining damage. Surface microcracks or damage resulting from the machining process, i.e., striations, scratches, impact cracks. (NOTE: surface and subsurface damage are intrinsic to the machining damage).	MD^S
3.3.2 Handling damage. Scratches, chips, cracks, etc., due to the handling of the specimen.	HD^S
3.3.3 Pit. A flaw created by a reaction with the environment e.g., corrosion, thermal cycling, etc.	PT^S

3.3.4 Surface void. A void which is located at the surface and is a consequence of processing, i.e., surface reaction layer, as distinguished from a volume-distributed flaw. SV^S

Flaw	Code
3.4 MISCELLANEOUS.	
3.4.1 Other. A flaw specific to a material.	@
NOTE: the code used for Other types of flaws is up to the discretion of the user and should be carefully defined by the user.	
3.4.2 ?????. An uncertain or undetermined flaw.	?

4. CONCLUDING MATERIAL

4.1 Intended use. This handbook contains requirements for characterizing fracture origins in advanced structural ceramics. It is limited to monolithic ceramics and some composite ceramics.

4.2 Subject term (key-word) listing.

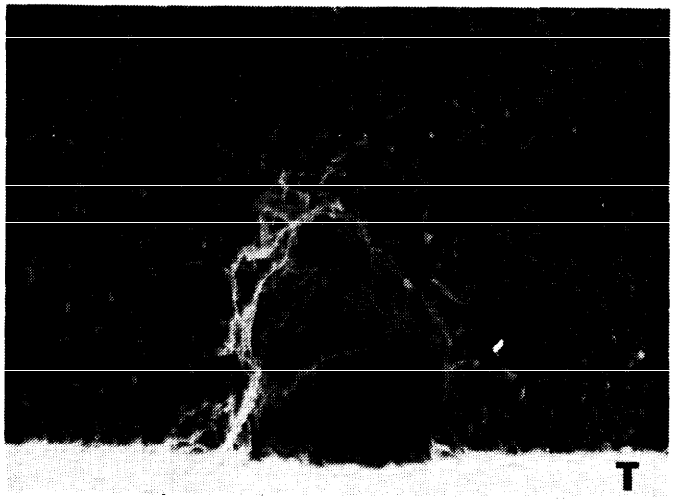
Flaw	Microscope
Pore	Crack
Agglomerate	Machining damage
Inclusion	Pit
Void	Fractography

Custodians:
Army - MR
Navy - AS
Air Force - 99

Preparing Activity:
Army-MR

Project 9350-1006

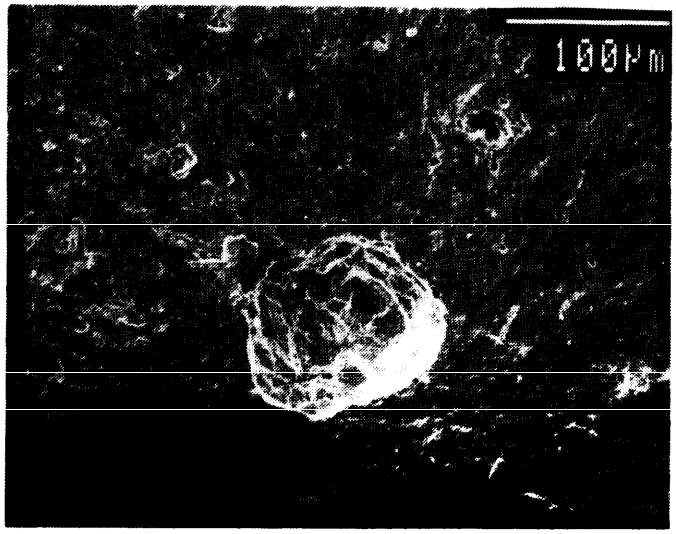
Review activities:
Army - AT, EA, ER, MI, ME
Navy - SH, OS, YD
Air Force - 11, 84
DLA - GS



MATERIAL: Sintered (99.9% pure) Alumina, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

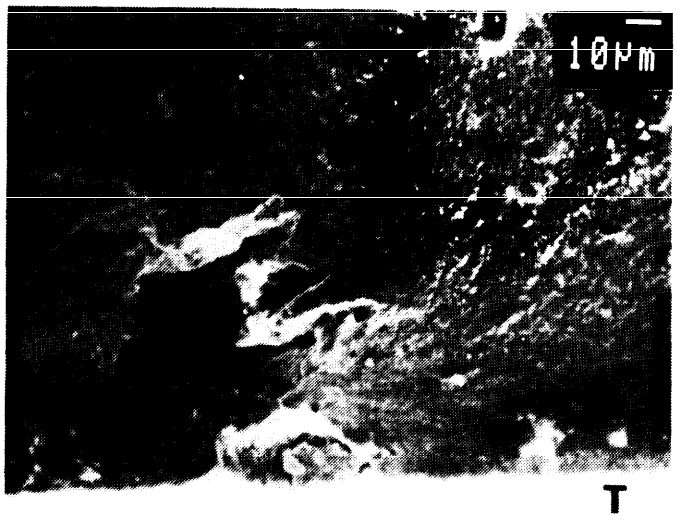
COMMENTS: σ = 337 MPa



MATERIAL: Sintered Yttria-tetragonal zirconia polycrystal, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature after being exposed to ≈ 800 Pa water vapor pressure at 200C for 50 hours

COMMENTS: σ = 544 MPa

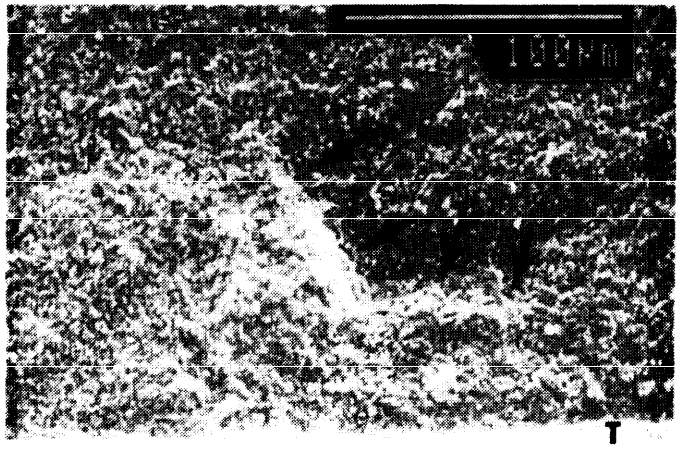


MATERIAL: Sintered Yttria-tetragonal zirconia polycrystal, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

COMMENTS: σ = 594 MPa

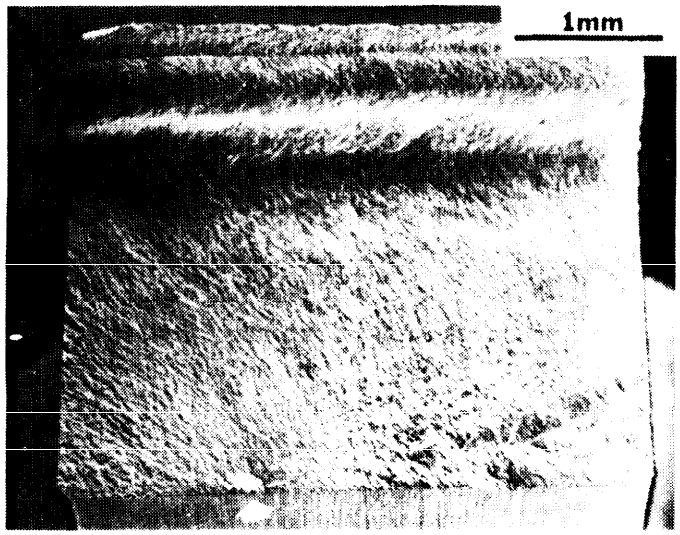
FIGURE 22. Examples of pores.



MATERIAL: Sintered (99.9% pure) Alumina, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

COMMENTS: σ = 300 MPa



MATERIAL: Sintered (99.9% pure) Alumina, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

COMMENTS: σ = 329 MPa;
-Photo A is room low magnification SEM analysis;
-Photo B is from high magnification SEM analysis

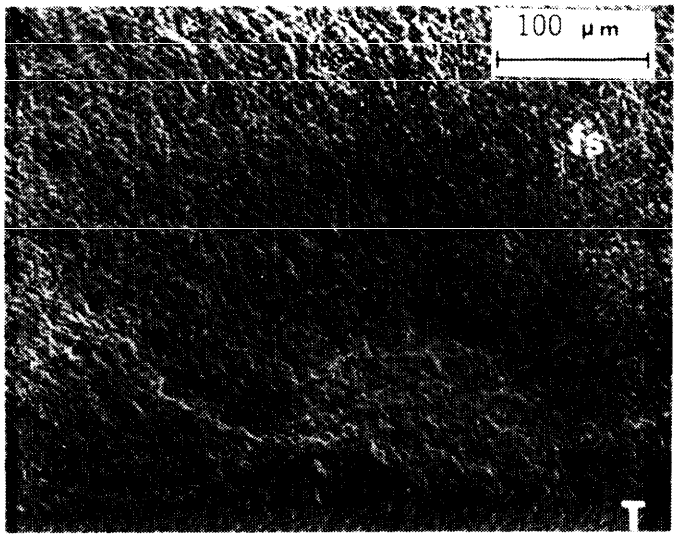
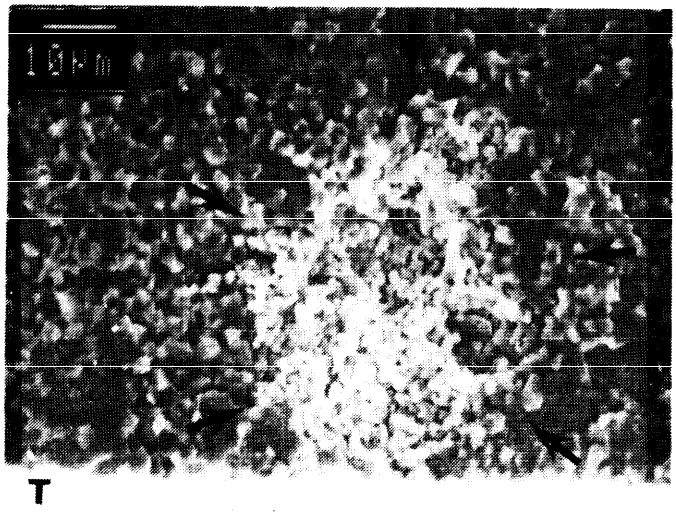


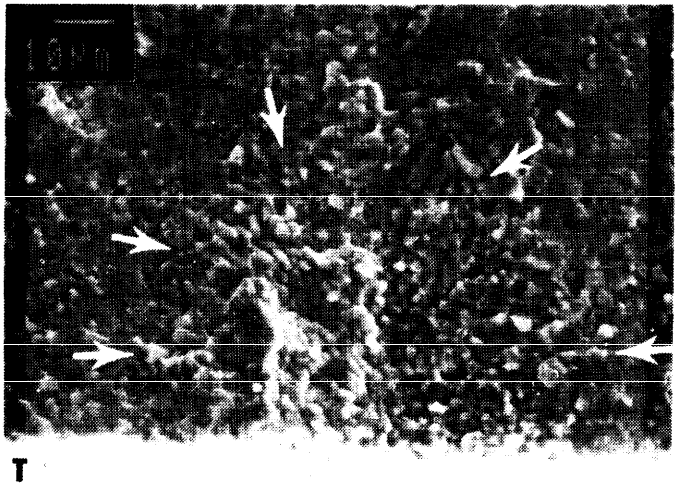
FIGURE 23. Examples of porous seams.



MATERIAL: Sintered (99.9% pure) Alumina, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

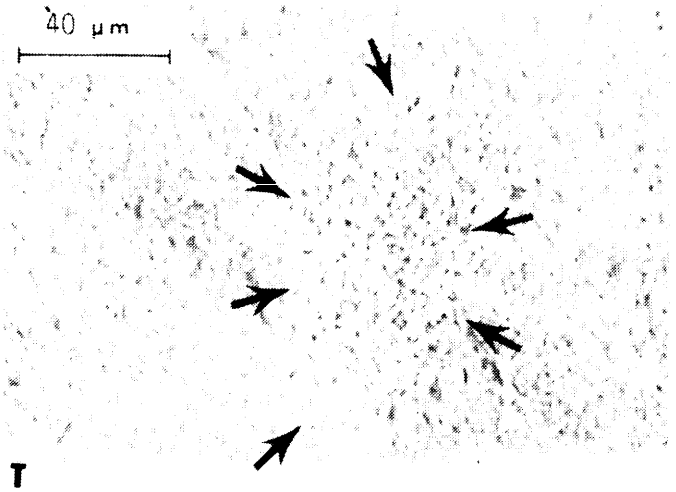
COMMENTS: σ = 430 MPa



MATERIAL: Sintered (99.9% pure) Alumina, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

COMMENTS: σ = 419 MPa; -Flaw is a zone of concentrated microporosity

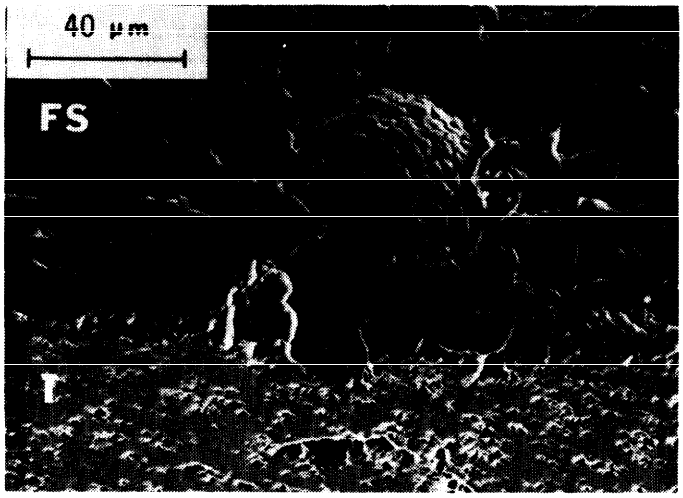


MATERIAL: Sintered Sialon with Yttria & Alumina additions, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

COMMENTS: σ = 450 MPa

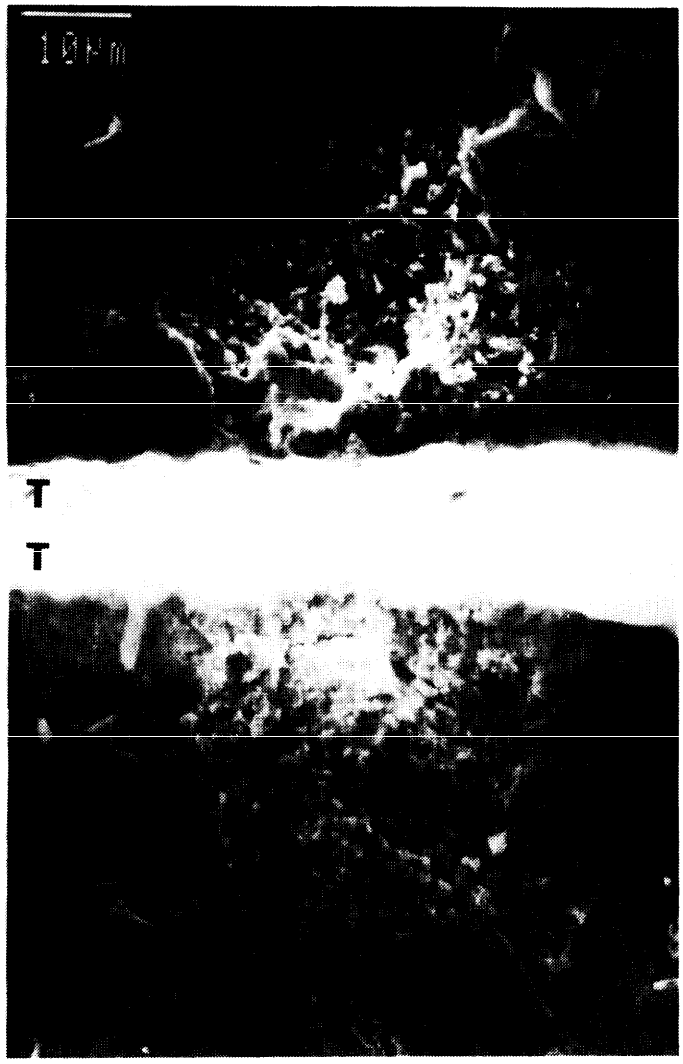
FIGURE 24. Examples of porous regions.



MATERIAL: Sintered α-Silicon Carbide, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

COMMENTS: $\sigma = \bar{362}$ MPa;
-The formation of the agglomerate can be traced to the spray drying process used to manufacture the starting powder

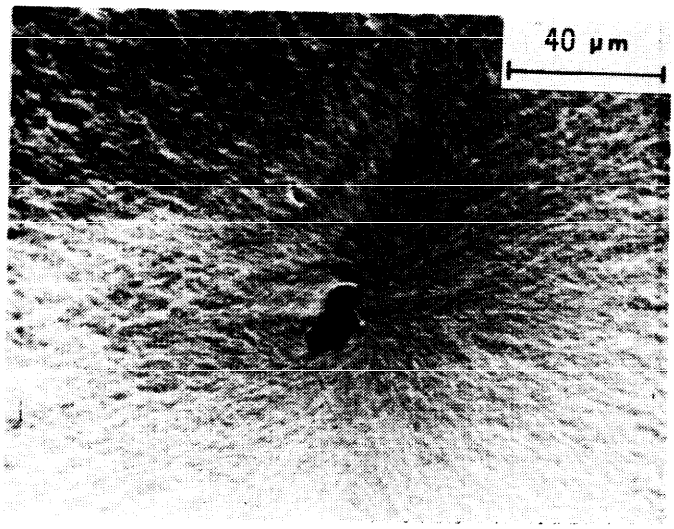


MATERIAL: Sintered Yttria-Tetragonal Zirconia Polycrystal, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

COMMENTS: σ = 765 MPa;
-Photos show the mating halves of the fracture surface

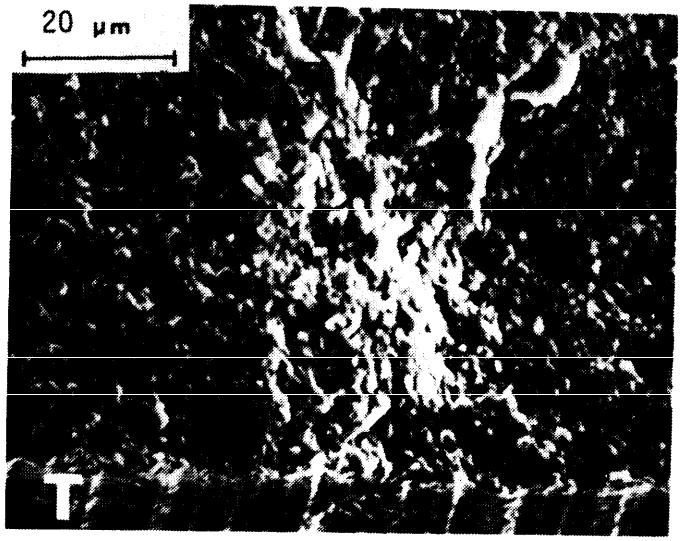
FIGURE 25. Examples of agglomerates.



MATERIAL: Hot pressed Yttria-Tetragonal Zirconia Polycrystal, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

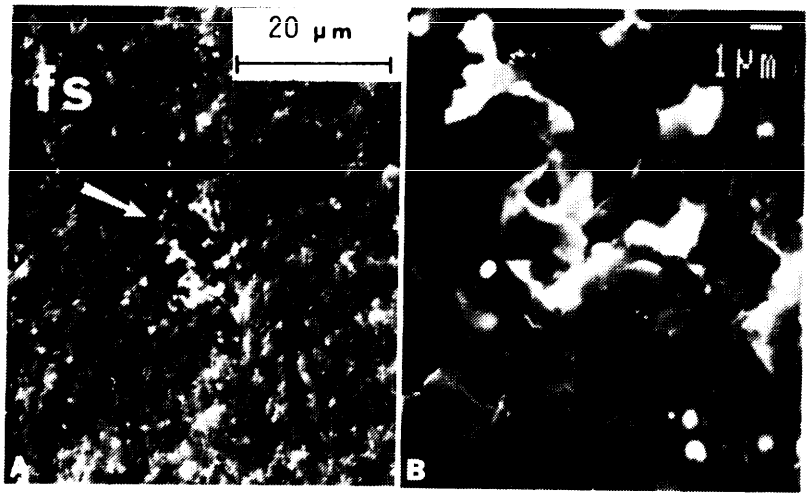
COMMENTS: σ = 1029 MPa;
-Inclusion is Silicon



MATERIAL: Sintered Sialon with Yttria & Alumina additions, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

COMMENTS: σ = 545 MPa;
-Inclusion contains Fe & Cr

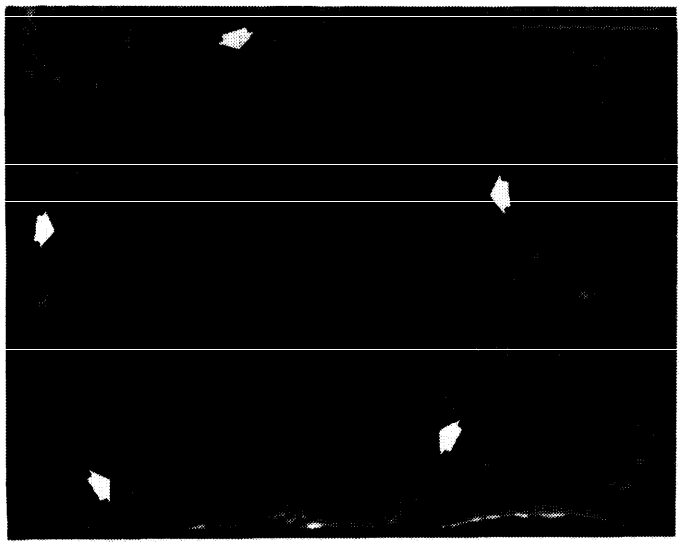


MATERIAL: Hot pressed Silicon Nitride with Magnesia additive, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

COMMENTS: σ = 831 MPa;
-Inclusion is a series of small tungsten particles
-Photo B is an enlargement of A

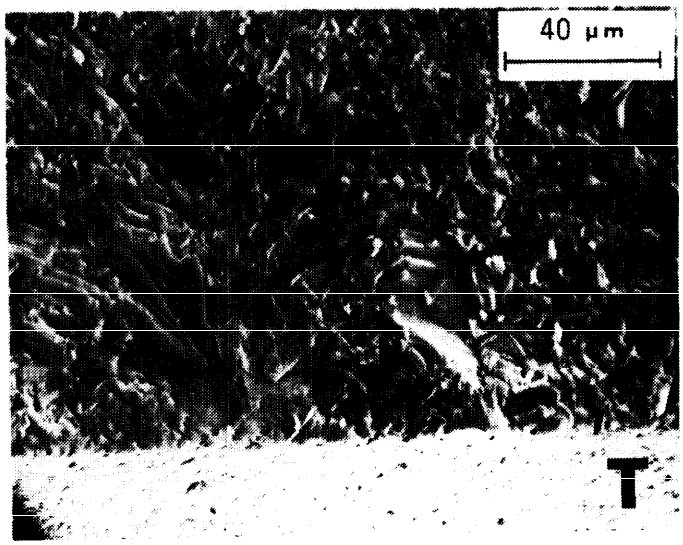
FIGURE 26. Examples of inclusions.



MATERIAL: Hot pressed Yttria-Tetragonal Zirconia Polycrystal, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature after being exposed to 1000C for 500 hours

COMMENTS: σ = 1033 MPa;
-EDAX analysis shows the second phase to contain elemental Al.
-Chemical analysis of the bulk material shows it contains between 0.1% to 0.7% Al

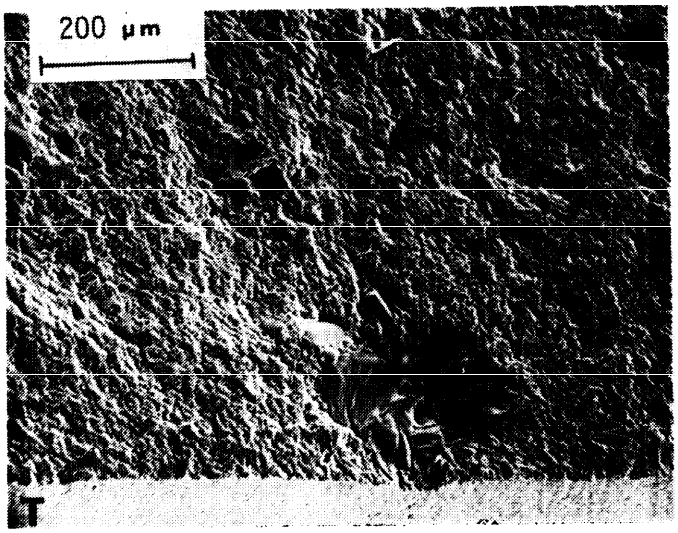


MATERIAL: Siliconized Silicon Carbide with Silicon Carbide whisker reinforcement, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

COMMENTS: σ = 455 MPa;
-Flaw is a "lake" of free Silicon

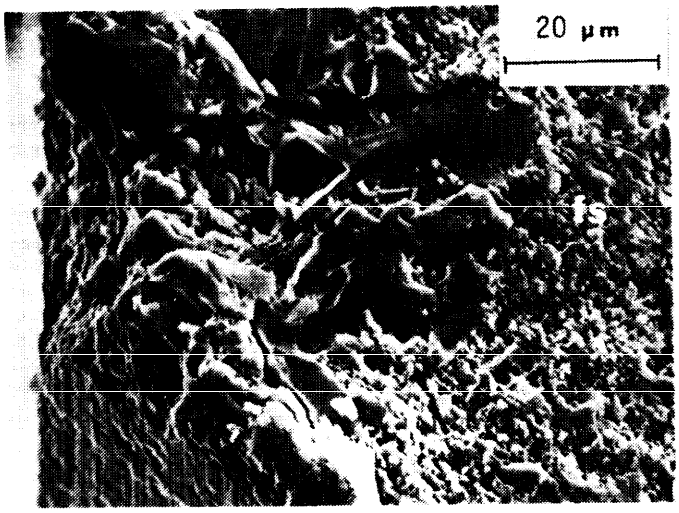
FIGURE 27. Examples of second phase inhomogeneities.



MATERIAL: Siliconized Silicon Carbide, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

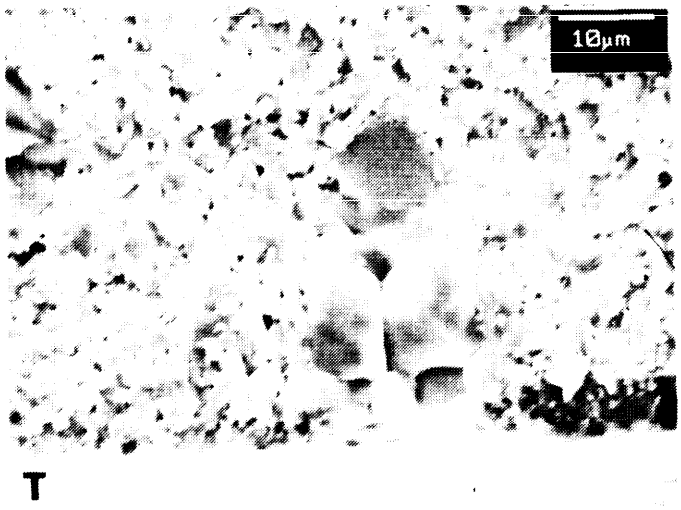
COMMENTS: σ = 169 MPa;
-Flaw is large grains of Silicon Carbide
-Interpretation was aided by photographs of a representative polished section



MATERIAL: Sintered (99.9% pure) Alumina, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

COMMENTS: σ = 404 MPa;
-Large grains in this particular specimen are located near the edge

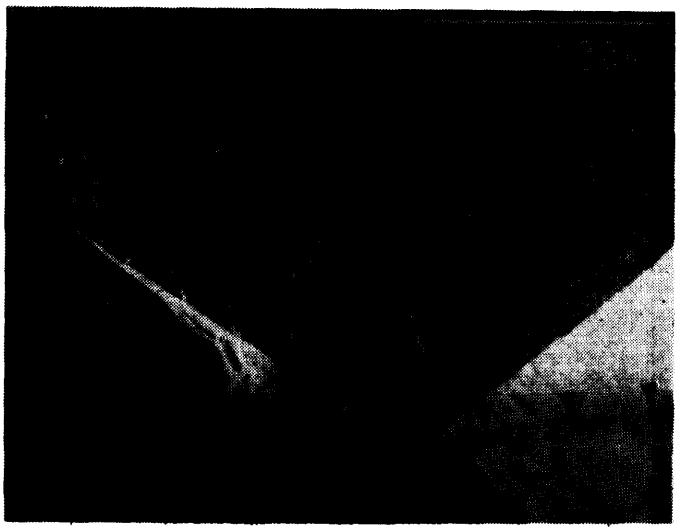


MATERIAL: Hot pressed Alumina reinforced with Silicon Carbide whiskers, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature after being exposed to 1000C for 500 hours

COMMENTS: σ = 220 MPa

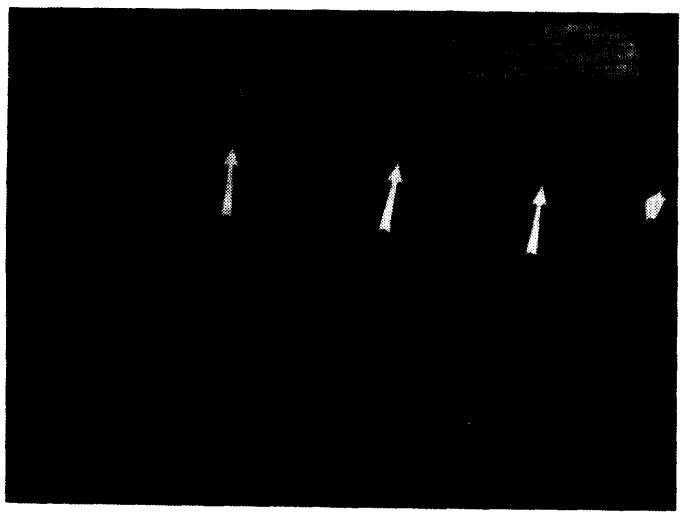
FIGURE 28. Examples of large grains.



MATERIAL: Hot pressed Yttria-Tetragonal Zirconia Polycrystal, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature after being exposed to ≈ 800 Pa water vapor pressure 200C for 50 hours

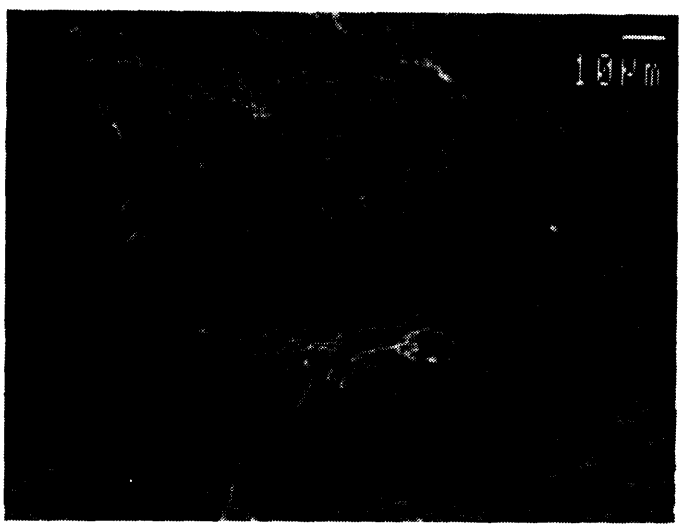
COMMENTS: σ = 514 MPa



MATERIAL: Sintered β-Silicon Carbide, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

COMMENTS: σ = 340 MPa;
-Optical photo of the tensile surface of a flexure bar showing a crack in the material (large white arrows)
-Failure originated on the tensile surface at the point marked by the small white arrow

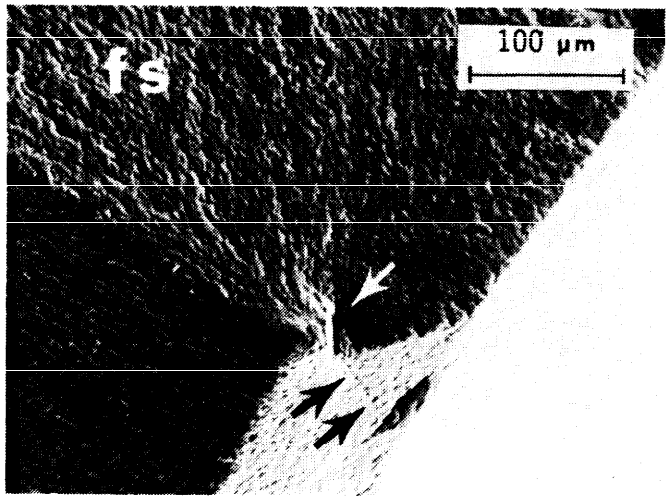


MATERIAL: Hot isostatically pressed Yttria-Tetragonal Zirconia Material, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature after being exposed to 1000C for 500 hours

COMMENTS: σ = 973 MPa;
-This could be a pore that collapsed during the HIPing process

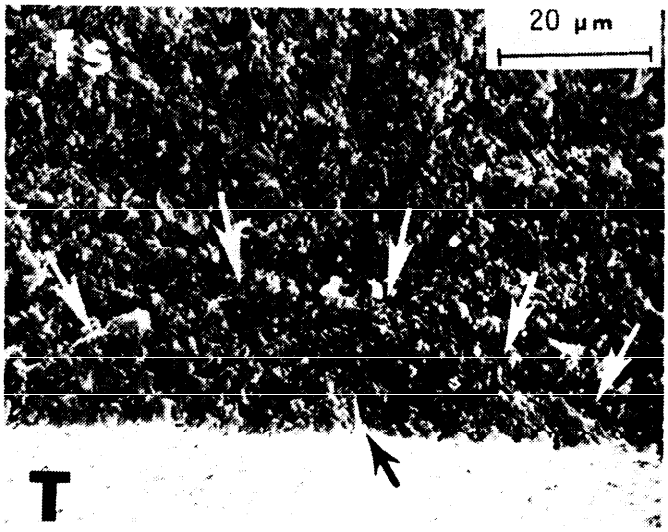
FIGURE 29. Examples of cracks.



MATERIAL: Reaction Bonded Silicon Nitride, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

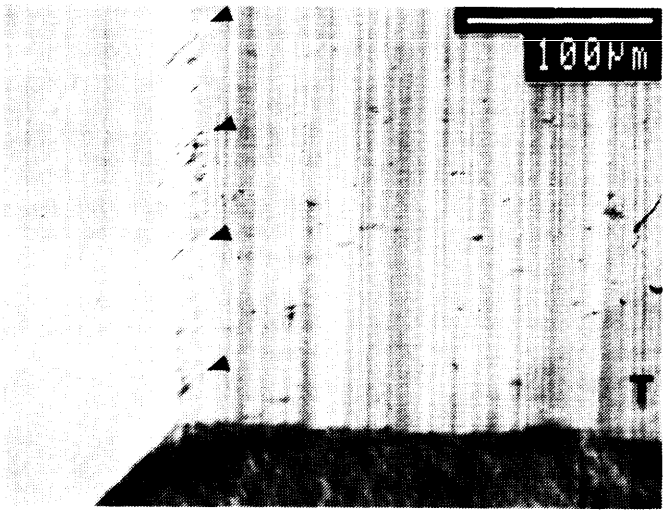
COMMENTS: σ = 264 MPa;
-White arrows mark the subsurface machining damage associated with the machining of the chamfer



MATERIAL: Hot pressed Silicon Nitride with Magnesia additions, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

COMMENTS: σ = 890 MPa;
-Subsurface semielliptical crack (white arrows) induced by the machining process
-A deep striation with a small crack is marked by the black arrow

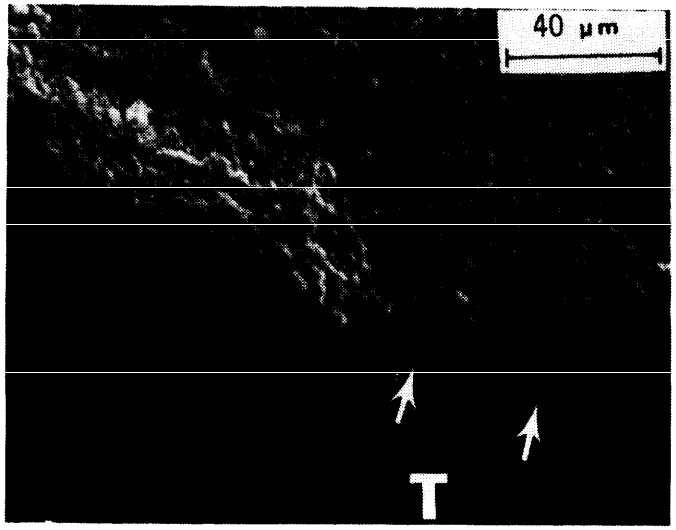


MATERIAL: Sintered Yttria-Tetragonal Zirconia Polycrystal, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature after being exposure to 1000C for 500 hours

COMMENTS: σ = 662 MPa

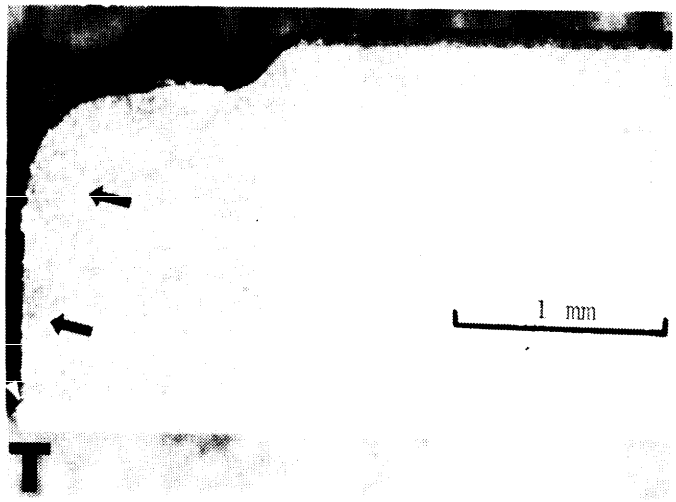
FIGURE 30. Examples of machining damage.



MATERIAL: Sintered Sialon with Yttria & Alumina additions, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

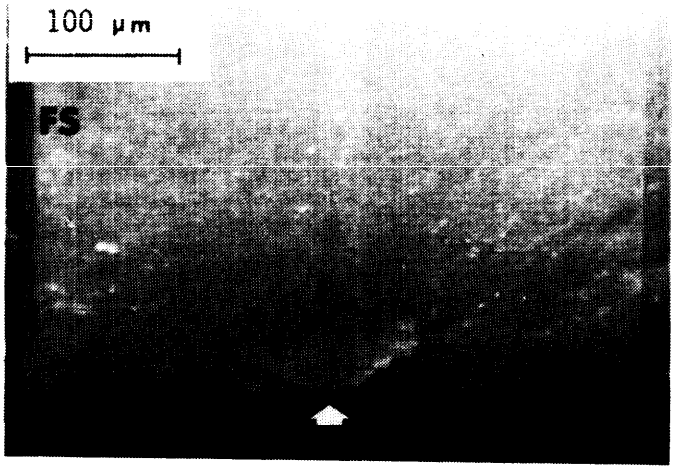
COMMENTS: σ = 595 MPa;
-Scratch on the tensile surface



MATERIAL: Reaction bonded Silicon Nitride, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

COMMENTS: σ = 257 MPa;
-Side view of a flexure bar showing a scratch (black arrows) which caused fracture at the chamfer

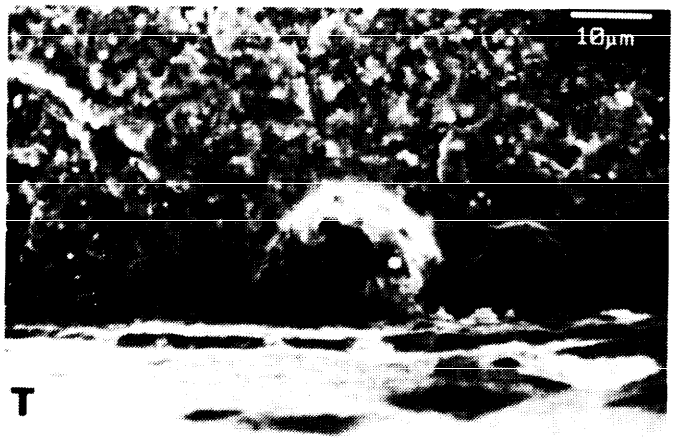


MATERIAL: Sintered (99.9% pure) Alumina, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

COMMENTS: σ = 387 MPa;
-Hertzian cone crack from impact damage

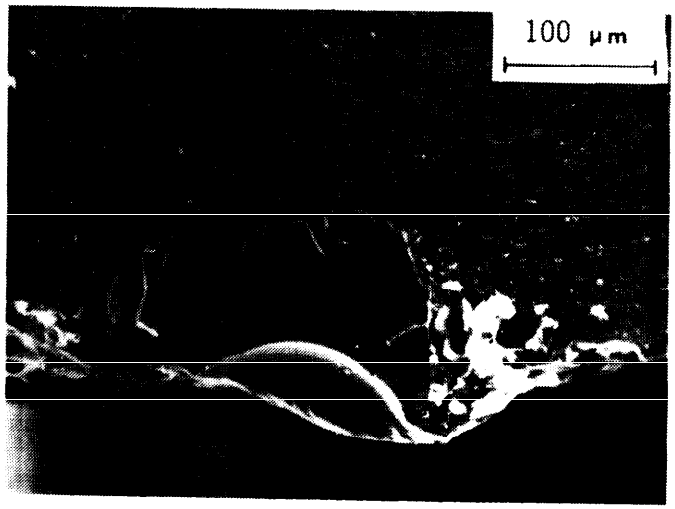
FIGURE 31. Examples of handling damage.



MATERIAL: Hot pressed Silicon Nitride with Magnesia additions, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature after being exposed to 1000C for 500 hours

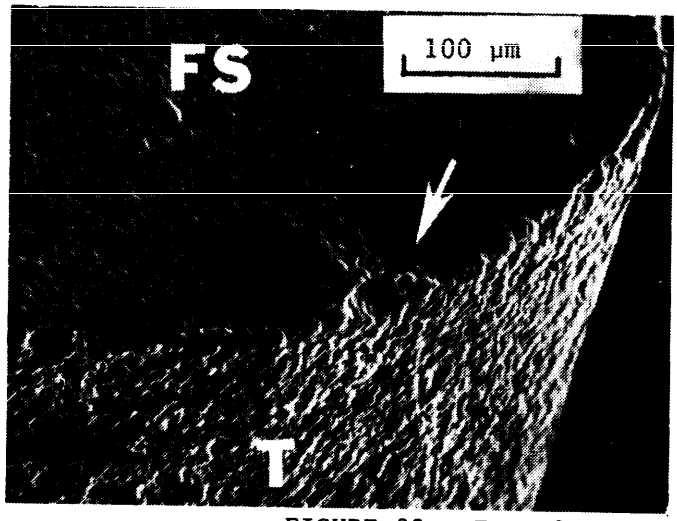
COMMENTS: σ = 598 MPa;
-Pits formed due to oxidation



MATERIAL: Hot pressed Silicon Nitride with Magnesia additions, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature after being exposed to 1000C for 500 hours in a corrosive environment

COMMENTS: σ = 292 MPa;
-Pit formed as a result of corrosion
-The pit contains Na from the sodium sulfate corrosion material

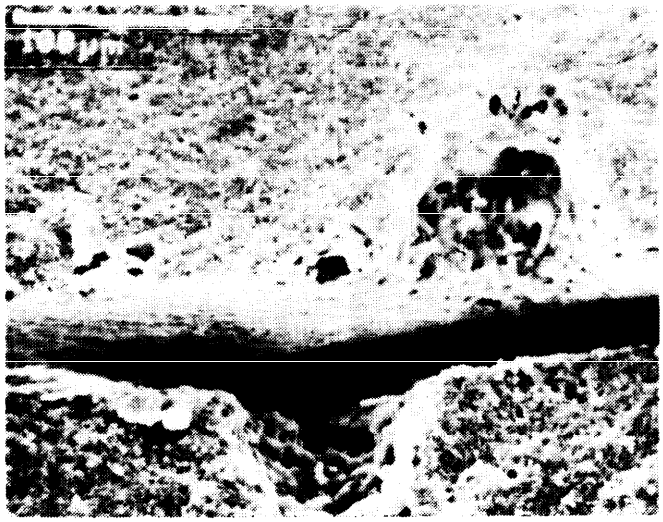


MATERIAL: Reaction bonded Silicon Nitride, as machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature after being exposure to a severe cycling sequence involving alternating oxidation heat treatments & thermal cycling in a gas torch

COMMENTS: σ = 223 MPa;
-The exposure has caused the chamfer region to round

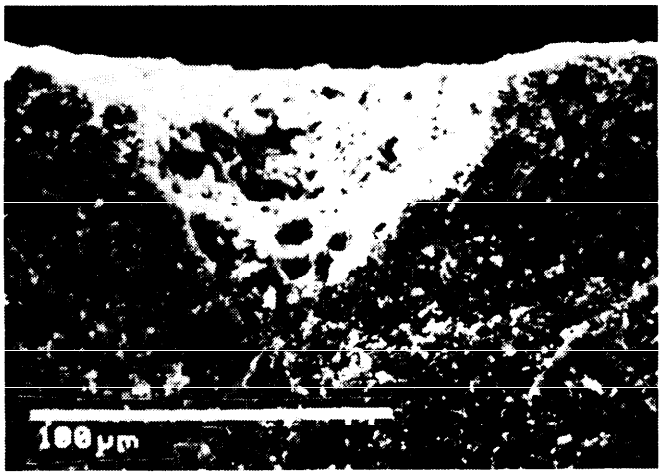
FIGURE 32. Examples of pits.



MATERIAL: Injection Molded & Sintered Silicon Nitride with Yttria & Alumina additions, as-fired

TEST CONDITION: Fast fracture in 4-point flexure at room temperature

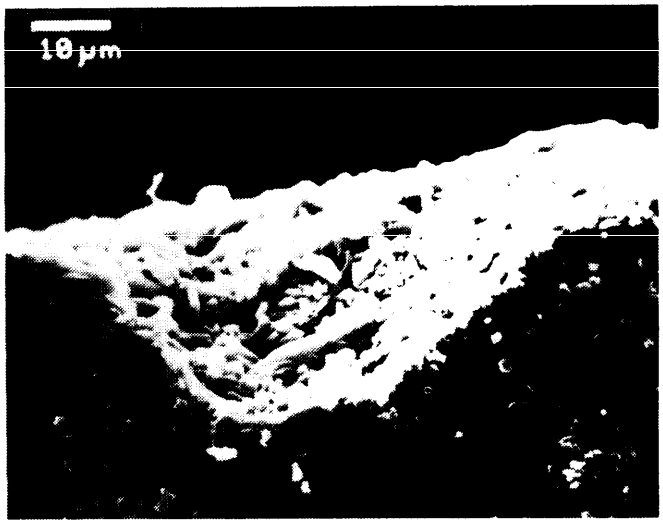
COMMENTS: σ = 472 MPa;
-Photo shows both halves of the fracture surface



MATERIAL: Injection Molded & Sintered Silicon Nitride with Yttria & Alumina additions, as-fired

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

COMMENTS: σ = 513 MPa



MATERIAL: Injection Molded & HIP'ed without a cladding, Silicon Nitride with Yttria & Alumina additions, as-fired

TEST CONDITION: Fast fracture in 4-point flexure at room temperature

COMMENTS: σ = 476 MPa;
-Surface pore is flattened at the edges since there was no cladding

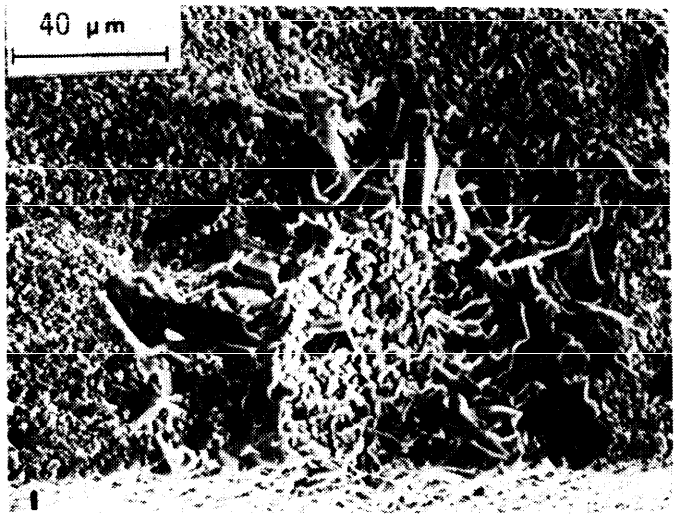
FIGURE 33. Examples of surface voids. (Courtesy of A. Pasto, GTE Laboratory, now with Oak Ridge National Laboratory).

MATERIAL: Siliconized Silicon Carbide, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

COMMENTS: σ = 282 MPa;
-Flaw is a vein of elemental Si

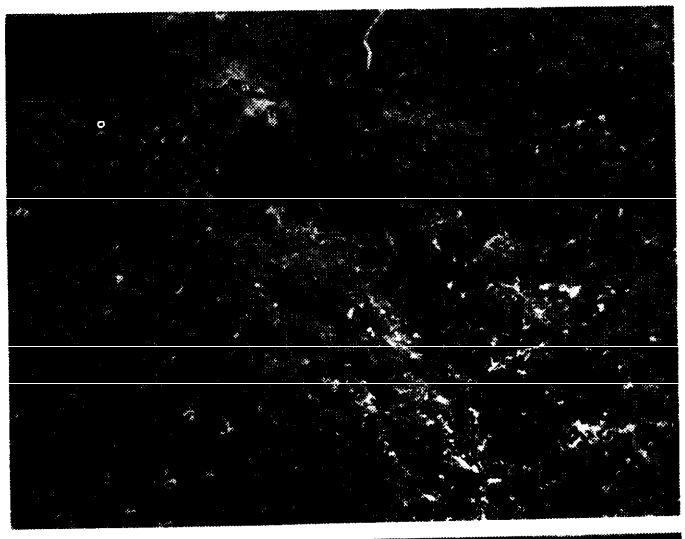
FIGURE 34. Examples of less common "other" flaws.



MATERIAL: Sintered (99.9% pure) Alumina, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

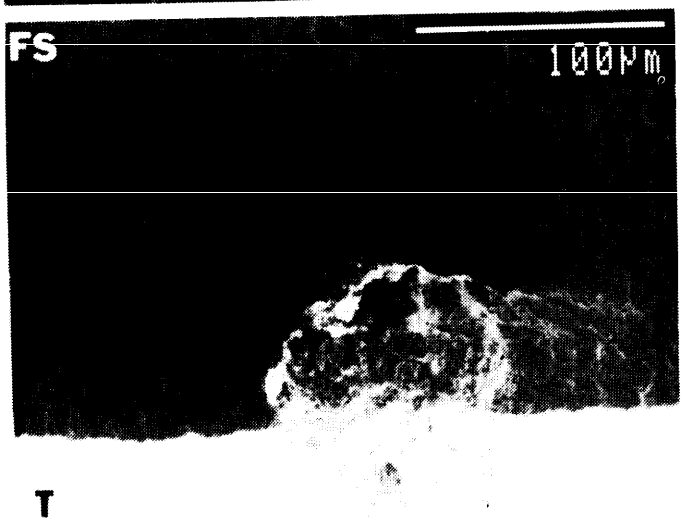
COMMENTS: σ = 396 MPa;
-Flaw could be classified either as a porous region or large grains (PR/LG)



MATERIAL: Sintered Yttria-Tetragonal Zirconia Polycrystal, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

COMMENTS: σ = 395 MPa;
-Flaw could be classified either as a large pore or a porous region (P/PR)



MATERIAL: Sintered Yttria-Tetragonal Zirconia Polycrystal, as-machined

TEST CONDITIONS: Fast fracture in 4-point flexure at room temperature

COMMENTS: σ = 583 MPa;
-Flaw could be classified either as a porous region or an agglomerate (PR/A)

FIGURE 35. Examples of coincidental flaws.

A SELECT BIBLIOGRAPHY ON FRACTOGRAPHY AND FLAWS IN CERAMICS

BOOKS ON ADVANCED CERAMICS FRACTOGRAPHY

1. Fractography of Glasses and Ceramics, Advances in Ceramics Vol. 22, eds., J. Varner, and V. Frechette, American Ceramic Society, Westerville, OH, 1988.

   * Eight papers on ceramics from a symposium in Philadelphia in April 1982. Includes the comprehensive review paper by Rice, and papers by Pantano and Kelso, and Healy and Mecholsky (cited below).

2. Fractography of Ceramic and Metal Failures, eds. J. Mecholsky, Jr., and S. Powell, Jr., ASTM STP 827, Philadelphia, PA, 1984.

   * Proceedings of a conference of the same name held at Alfred University in 1988. Sections on: fundamental phenomena, high temperature fracture, fractography and fracture mechanics, fractography in development and test, and field failures.

3. Concepts, Flaws and Fractography, Fracture Mechanics of Ceramics, Vol. 1., eds., R. Bradt, D. Hasselman, and F. Lange, Plenum Press, New York, 1974.

   * Proceedings of a conference at the Pennsylvania State University in 1973 with 23 papers on fracture mechanics applied to flaw detection and fractography in ceramics. The later volume of this series also have relevant fractography papers.

4. Failure Analysis of Brittle Materials, Advances in Ceramics, Vol. 28, V.D. Frechette, American Ceramic Society, Westerville, OH, 1990.

   * A must for the serious fractographer. This book covers all aspects of the fractography of glasses including fundamental markings on crack surfaces (Wallner lines, hackle, etc), crack forking, failure origins, estimates of stress at fracture and fractographic techniques. Superbly illustrated with a number of service failures and case histories presented.

MICROSCOPIC TECHNIQUES

1. C.G. Pantano and J.F. Kelso, "Chemical Analysis of Fracture Surfaces," in Fractography of Ceramic and Metal Failures, ASTM STP 827, 1984, pp 139-156.

   * The applicability of various instrumental techniques for chemical analysis of fracture surfaces is reviewed. The relative merits and spatial and depth resolutions of Auger microscopy and energy or wavelength dispersive electron microscopy are given.

2. J.T. Healy, and J.J. Mecholsky, Jr., "Scanning Electron Microscopy Techniques and Their Application to Failure Analysis of Brittle Materials," in Fractography of Ceramic and Metal Failures, ASTM STP 827, 1984, pp 157-181.

   * Discusses cleaning, coating and other procedures for SEM specimens. The merits and differential emphases of secondary and backscattered electron imaging are presented.

3. "Fractography", Metals Handbook, 9th Edition, Volume 12, ASM, Metals Park, OH, 1987.

* An excellent handbook on fractography of metals. Some generic sections including photographic, optical inspection and electron microscopy techniques are directly applicable to ceramic fractography. Light, secondary electron and backscattered electron photos of identical locations in metal specimens are compared. Caution: Some cleaning and preparation techniques such as surface coatings, replicating tapes, replicating tape stripping, and aggressive detergent cleaning which are prescribed for metals are not recommended for ceramic fracture surfaces.

4. V.D. Frechette, "Markings on Crack Surfaces of Brittle Materials: A Suggested Unified Nomenclature," in Fractography of Ceramic and Metal Failures, ASTM STP 827, pp 104-109.

* An attempt to develop a common nomenclature for fracture surface markings such as the various Wallner lines, scarps, hackle, etc.

FRACTOGRAPHY OF CERAMICS, OVERVIEW PAPERS

1. J.J. Mecholsky, Jr., and S.W. Freiman, "Determination of Fracture Mechanics Parameters Through Fractographic Analysis of Ceramics," in Fracture Mechanics of Ceramics Applied to Brittle Materials, Ed. S. Freiman, ASTM STP 678, 1979, pp 136-150.

* A short but useful overview of the utility of fractography as a quantitative tool to determine strength-limiting flaws, the stress at failure, and critical fracture toughness.

2. R.W. Rice, "Fractographic Identification of Strength Controlling Flaws and Microstructure," in Fracture Mechanics of Ceramics, Vol. 1, Eds. R.Bradt, D. Hasselman, and F. Lange, (Plenum, N.Y., 1974) pp 323-345.

* A short but valuable discussion of several key flaws (pores, pore groups and large grains) and their relationship to fracture energy. The fracture energy can either be a single-crystal or polycrystalline value depending upon the relative sizes of flaw and microstructure.

3. G.D. Quinn, J.J. Swab, and M.J. Slavin, "A Proposed Standard Practice for Fractographic Analysis of Monolithic Advanced Ceramics", MTL TR 90-57, November 1990, NTIS Access No. ADA-231989.

* The basis for this MIL HBK. Discusses essential background information and the rationale for consistency in characterization. From this information a standard nomenclature and flaw-characterization scheme are created. Also includes a detailed bibliography and examples of the various flaw types.

4. R.W. Rice, "Topography of Ceramics," in Surfaces and Interfaces of Glass and Ceramics, eds., V. Frechette, W. LaCourse, and V. Burdick, (Plenum, N.Y., 1974) pp 439-472.

* A very helpful introduction describes the role of unaided eye, hand lense, optical, scanning and transmission electron microscopy. Figure 1 shows optical and SEM photos of the same flaw. Fracture surface features such as trans- and intergranular fracture, crack microstructure interactions, crack branching, mirrors and single crystal fractography are discussed.

5. R.W. Rice, "Ceramic Fracture Features, Observations, Mechanism and Uses", in Fractography of Ceramic and Metal Failures, ASTM STP 827, 1984, pp 5-103.

* A lengthy review paper with a detailed technical discussion of fracture mirrors and related features (mist, hackle and branching) in glasses, polycrystals, and single crystals. The "bluntness" of flaws (round pores versus sharp machining cracks) will alter the mirror-to-defect radius ratio. A useful table of branch angle as a function of mode of loading (flexure, tensile, biaxial, thermal) for several materials is given.

6. D.W. Richerson, "Failure Analysis", in Modern Ceramic Engineering, Marcel Dekker, Inc., New York, pp 325-375 (1982).

* An outstanding presentation of both intrinsic and extrinsic flaws in silicon nitride and silicon carbide. Richly illustrated, this chapter carefully relates flaws to processing and service conditions.

7. "Failure Analysis", in Engineering Materials Handbook, Volume 4, Ceramics and Glasses, pp 629-673 (1991).

* Chapter 9 includes articles by J. Varner on Descriptive Fractography, Quantitative Analysis by T. Michalske, Optical Fiber Analysis by J. Mecholsky, and Glass Ceramic Failure Analysis by B. Adams, and S. DeMartino.

FLAWS IN ADVANCED CERAMICS

1. H. Kirchner, R. Gruver, and W. Sotter, "Characteristics of Flaws at Fracture Origins and Fracture Stress - Flaw Size Relations in Various Ceramics," Mat. Sci. and Eng., 22 (1976) pp 147-156.

* A concise but useful report on strength-limiting defects in alumina, silicon nitride and silicon carbide with a detailed tabulation of different flaw types. Emphasis is on porosity, large grains and machining flaws. An important observation (Fig. 1b) is that flaws in the center of fracture mirrors may intersect the fracture surface at an angle, and a true view of the flaw may not be seen.

2. H. Baumgartner, and D. Richerson, "Inclusion Effects on the Strength of Hot Pressed Si_3N_4," in Fracture Mechanics of Ceramics, Vol. 1, (1974) pp 367-86.

* Good characterization of machining damage and inclusions in silicon nitride. The inclusions are much smaller than expected (based on a penny shaped crack model) evidently the result of a locally degraded fracture toughness!

3. M.G. Gee, and R. Morrell, "Fracture Mechanics and Microstructures," in Fracture Mechanics of Ceramics, Vol. 8, eds. R. Bradt, A. Evans, D. Hasselman, and F. Lange, (Plenum, N.Y., 1986) pp 1-22.

* Principly a discussion of the application of fracture mechanics theories to strength. Microstructural influences will significantly complicate this and may limit utility to qualitative issues. The nature of strength-limiting flaws and their severity is discussed. In some instances, sharp cracks will not appear until stress is applied.

4. A.G. Evans, "Structural Reliability, A Processing Dependent Phenomenon," J. Am. Ceram. Soc., 65 #3 (1982) pp 127-39.

* Emphasis on the micromechanics of fracture with a good discussion of the effect of thermal and mechanical property mismatches between a flaw and the matrix. Only a few photos, but includes some excellent schematics. Includes a well known graph of stress versus flaw size for silicon nitride showing the relative severity of different flaws (WC, Fe, Si, C inclusions, porosity and machining damage).

5. R.W. Rice, "Failure Initiation in Ceramics: Challenges of NDE and Processing," in preparation. (see note below)

* A comprehensive, well illustrated review of failure initiating flaws. Nearly an encyclopedia of flaws. Flaws include: agglomerates, pores, large grains, inclusions, machining damage, handling damage, thermocouple beads, ball mills, dandruff, insects, feces, inadequate mixing of constituents, etc.
NOTE: A short summary has been published in "Ceramic Developments," eds. C. Sorrell and B. Ben-Nissan, Materials Science Forum, Vol. 34-36, Trans. Tech. Publ. Ltd. Switzerland, (1988) pp 1057-1064.

6. R.W. Rice, "Processing Induced Sources of Mechanical Failure in Ceramics," in Processing of Crystalline Ceramics, eds. H. Palmour, R. Davis, and T. Hare, (Plenum, N.Y., 1978) pp 303-319.

* A short, well illustrated review of flaws. A good starting point.

7. R.W. Rice, J.J. Mecholsky, Jr., and P.F. Becher, "The Effect of Grinding Direction on Flaw Character and Strength of Single Crystal and Polycrystalline Ceramics," J. Mat. Sci. 16 (1981) pp 853-862.

* Machining damage in a variety of ceramics is well illustrated by nine figures.

8. J.J. Mecholsky, Jr., S.W. Freiman, and R.W. Rice, "Effects of Grinding on Flaw Geometry and Fracture of Glass," J. Am. Ceram. Soc., 60 #3-4 (1977) pp 114-117.

* Two primary sets of flaws result from surface grinding. These are schematically shown and complemented by SEM photos and related to fracture mechanics parameters.

9. R.W. Rice, "Pores as Fracture Origins in Ceramics," J. Mat. Sci., 19 (1984) 895-914.

* A well illustrated examination of pores in glassy and polycrystalline materials. Pores tend to be "sharper" in the latter than in the former.

FRACTURE MIRRORS

1. J.J. Mecholsky, Jr., S.W. Freiman, and R.W. Rice, "Fracture Surface Analysis of Ceramics," J. Mat. Sci., 11 (1976) pp 1310-19.

* A detailed correlation of flaw size, fracture mirror sizes and characterization, and fracture mechanics parameters for single and polycrystalline ceramics. A table of mirror constants is given for a range of ceramics and it is demonstrated that the outer mirror (hackle) to flaw size ratio is about 13 to 1. The inner mirror (mist) ratio is 6-10 to 1.

2. J.J. Mecholsky, Jr., R.W. Rice, and S.W. Freiman, "Prediction of Fracture Energy and Flaw Sizes in Glasses from Measurements of Mirror Size," J. Am. Ceram. Soc., 57 #10 (1974) pp 440-443.

* Details of fracture mirror features are discussed and related to fracture mechanics parameters for glasses. A table of mirror constants is included. A now famous schematic rendition of a fracture mirror showing the flaw, mist and hackle is presented.

3. H.P. Kirchner, R.M. Gruver, and W.A. Sotter, "Fracture Stress - Mirror Size Relations for Polycrystalline Ceramics," Phil. Mag., [33] #5 1976 pp 775-780.

* Many mirror constants for a range of ceramics.

4. H.P. Kirchner, and J.C. Conway, Jr., "Fracture Mechanics of Crack Branching in Ceramics," in Fractography of Glass and Ceramics, Advances in Ceramics, Vol. 22, (American Ceramic Society, Westerville, Ohio, 1988) pp 187-213.

* An analysis which extends the analysis that the mirror features are controlled by stress intensity.

5. J.J. Mecholsky, Jr., and S.W. Freiman, "Determination of Fracture Mechanics Parameters Through Fractographic Analysis of Ceramics," in Fracture Mechanics Applied to Brittle Materials, ASTM STP 678, Ed S. Freiman, ASTM 1979, pp 136-150.

* A short discussion of fracture mirrors and mirror constants with a comparative table of mirror constants. Comments on useful techniques to measure mirror parameters.

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