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Standard Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics[1]

This standard is issued under the fixed designation C1322; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 The objective of this practice is to provide an efficient and consistent methodology to locate and characterize fracture origins in advanced ceramics. It is applicable to advanced ceramics that are brittle; that is, fracture that takes place with little or no preceding plastic deformation. 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 word should not be construed to mean that the material has been prepared improperly or is somehow faulty.

1.2 Although this practice is primarily intended for laboratory test piece analysis, the general concepts and procedures may be applied to component fracture analyses as well. In many cases, component fracture analysis may be aided by cutting laboratory test pieces out of the component. Information gleaned from testing the laboratory pieces (for example, flaw types, general fracture features, fracture mirror constants) may then aid interpretation of component fractures. For more information on component fracture analysis, see Refs (**1, 2**).[2]

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

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

C162 Terminology of Glass and Glass Products
C242 Terminology of Ceramic Whitewares and Related Products
C1036 Specification for Flat Glass
C1145 Terminology of Advanced Ceramics
C1161 Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature
C1211 Test Method for Flexural Strength of Advanced Ceramics at Elevated Temperatures
C1239 Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics
C1499 Test Method for Monotonic Equibiaxial Flexural Strength of Advanced Ceramics at Ambient Temperature
C1678 Practice for Fractographic Analysis of Fracture Mirror Sizes in Ceramics and Glasses
F109 Terminology Relating to Surface Imperfections on Ceramics

2.2 *NIST Standard:*[4]

NIST Special Publication SP 960-16 Guide to Practice for Fractography of Ceramics and Glasses (**2**)

2.3 *CEN Standard:*[5]

EN 843-6 Advanced Technical Ceramics—Mechanical Properties of Monolithic Ceramics at Room Temperature—Part 6: Guidance for Fractographic Investigation

3. Terminology

3.1 *Definitions:*

3.1.1 *General*—The following terms are given as a basis for identifying fracture origins in advanced ceramics. It should be recognized that origins can manifest themselves differently in

[1] This practice is under the jurisdiction of ASTM Committee C28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.01 on Mechanical Properties and Performance.

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[2] The boldface numbers in parentheses refer to the list of references at the end of this standard.

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

[4] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

[5] Available from European Committee for Standardization (CEN), Avenue Marnix 17, B-1000, Brussels, Belgium, http://www.cen.eu.

various materials. The photographs in Appendix X1 show examples of the origins defined in 3.2.1 and 3.2.12. Terms that are contained in other ASTM standards are noted at the end of the each definition. The specific origin types listed in 3.2.1 – 3.2.12 are the most common types in advanced ceramics, but by no means cover all possibilities. NIST Special Publication SP 960-16 (**2**) includes many more origin types. Subsection 3.3 provides guidance on how to characterize or define other origin types. Some common origin types are identified in 3.2.1 – 3.2.12. These origin flaws are distributed throughout the bulk (inherently volume distributed) or are distributed on an exterior surface (inherently surface distributed). The distinction is very important for Weibull statistical analysis and size scaling of strength as discussed in Practice C1239. Subsection 7.2 provides guidance on interpretation

3.1.2 *advanced ceramic, n*—a highly engineered, high-performance, predominately nonmetallic, inorganic, ceramic material having specific functional attributes. **C1145**

3.1.3 *brittle fracture, n*—fracture that takes place with little or no preceding plastic deformation.

3.1.4 *flaw, n*—structural discontinuity in an advanced ceramic body that acts as a highly localized stress raiser.

3.1.4.1 *Discussion*—The presence of such discontinuities does not necessarily imply that the ceramic has been prepared improperly or is faulty.

3.1.5 *fractography, n*—means and methods for characterizing a fractured specimen or component. **C1145**

3.1.6 *fracture mirror, n*—as used in fractography of brittle materials, a relatively smooth region in the immediate vicinity of and surrounding the fracture origin.

3.1.7 *fracture origin, n*—the source from which brittle fracture commences. **C1145**

3.1.8 *grain boundary (GB), n*—as used in fractography, a volume-distributed flaw that is a boundary facet between two or more grains.

3.1.8.1 *Discussion*—This flaw is most apt to be strength limiting in coarse-grained ceramics.

3.1.9 *hackle, n*—as used in fractography, a line or lines on the crack surface running in the local direction of cracking, separating parallel but non-coplanar portions of the crack surface.

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

3.2 *Common Origins:*

3.2.1 *agglomerate (A), n*—as used in fractography, a volume-distributed flaw that is a cluster of grains, particles, platelets, or whiskers, or a combination thereof, present in a larger solid mass. **C1145**

3.2.2 *compositional inhomogeneity (CI), n*—as used in fractography, a volume-distributed flaw that is a microstructural irregularity related to the nonuniform distribution of the primary constituents or an additive or second phase. **C1145**

3.2.3 *crack (CK), n*—as used in fractography, a volume- or surface-distributed flaw that is a surface of fracture without complete separation. **C1145**

3.2.4 *handling damage (HD), n*—as used in fractography, surface-distributed flaws that include scratches, chips, cracks, etc., due to the handling of the specimen/component. **C1145**

3.2.5 *inclusion (I), n*—as used in fractography, a volume-distributed flaw that is a foreign body that has a composition different from the nominal composition of the bulk advanced ceramic. **C1145**

3.2.6 *large grain(s) (LG), n*—as used in fractography, a volume- or surface-distributed flaw that is a single (or cluster of) grain(s) having a size significantly greater than that encompassed by the normal grain size distribution. **C1145**

3.2.7 *machining damage (MD), n*—as used in fractography, a surface-distributed flaw that is a microcrack(s), chip(s), striation(s), or scratch(es), or a combination of these, created during the machining process.

3.2.7.1 *Discussion*—Machining may result in the formation of surface or subsurface damage, or both. **C1145**

3.2.8 *pit (PT), n*—as used in fractography, a surface-distributed flaw that is a cavity created on the specimen/component surface during the reaction/interaction between the material and the environment, for example, corrosion or oxidation. **C1145**

3.2.9 *pore (P(V)), n*—as used in fractography, a volume-distributed flaw that is a discrete cavity or void in a solid material. **C1145**

3.2.10 *porous region (PR), n*—as used in fractography, a volume-distributed flaw that is a three-dimensional zone of porosity or microporosity. **C1145**

3.2.11 *porous seam (PS), n*—as used in fractography, a volume-distributed flaw that is a two-dimensional area of porosity or microporosity. **C1145**

3.2.12 *surface void (SV), n*—as used in fractography, a surface-distributed flaw that is a cavity created at the surface/exterior as a consequence of the reaction/interaction between the material and the processing environment, for example, surface reaction layer or bubble that is trapped during processing. **C1145**

3.3 *Miscellaneous Origins:*

3.3.1 *unidentified origin (?), n*—as used in this practice, an uncertain or undetermined fracture origin.

3.4 Other terms or fracture origin types may be devised by the user if those listed in 3.2.1 – 3.2.12 are inadequate. In such instances, the user shall explicitly define the nature of the fracture origin (flaw) and whether it is inherently volume or surface distributed. Additional terms for surface imperfections can be found in Terminology F109 and supplementary fracture origin types for ceramics and glasses may be found in Terminologies C162 and C242 and in Specification C1036. Examples of additional terms are hard agglomerate, collapsed agglomerate, hard agglomerate (CEN 843-6) poorly bonded region, glassy inclusion, chip, closed chip, chip (CEN 843-6), delamination (CEN 843-6), grain boundary cracks, chatter cracks, sharp impact cracks, blunt impact cracks, C-cracks (ball bearings), baseline microstructural flaws (BMF), or mainstream microstructural flaws (MMF). See the "Guide to Practice for Fractography of Ceramics and Glasses" (**2**) for discussion and examples.

3.5 The word "surface" may have multiple meanings. It may refer to the intrinsic spatial distribution of flaws. The word "surface" also may refer to the exterior of a test specimen cut from a bulk ceramic or component, or alternatively, the original surface of the component in the as-fired state. It is recommended that the terms original-surface or as-processed surface be used if appropriate.

4. Summary of Practice

4.1 Prior to testing, mark the specimen or component orientation and location to aid in reconstruction of the specimen/component fragments. Marker lines made with a pencil or felt-tip marker may suffice. See Fig. 1.

4.2 Whenever possible, test the specimen(s)/component(s) to fracture in a fashion that preserves the primary fracture surface(s) and all associated fragments for further fractographic analysis.

4.3 Carefully handle and store the specimen(s)/component(s) to minimize additional damage or contamination of the fracture surface(s), or both.

4.4 Visually inspect the fractured specimen(s)/component(s) (1 to 10×) in order to determine crack branching patterns, any evidence of abnormal fracture patterns (indicative of testing misalignments), the primary fracture surfaces, the location of the mirror and, if possible, the fracture origin. Specimen/component reconstruction may be helpful in this step. Label the pieces with a letter or numerical code and photograph the assembly if appropriate.

4.5 Use an optical microscope (10 to 200×) to examine both mating halves of the primary fracture surface in order to locate and, if possible, characterize the origin. Repeat the examination of pieces as required. If the fracture origin cannot be characterized, then conduct the optical examination with the purpose of expediting subsequent examination with the scanning electron microscope (SEM).

4.6 Inspect the external surfaces of the specimen(s)/component(s) near the origin for evidence of handling or machining damage or any interactions that may have occurred between these surfaces and the environment.

4.7 Clean and prepare the specimen(s)/component(s) for SEM examination, if necessary.

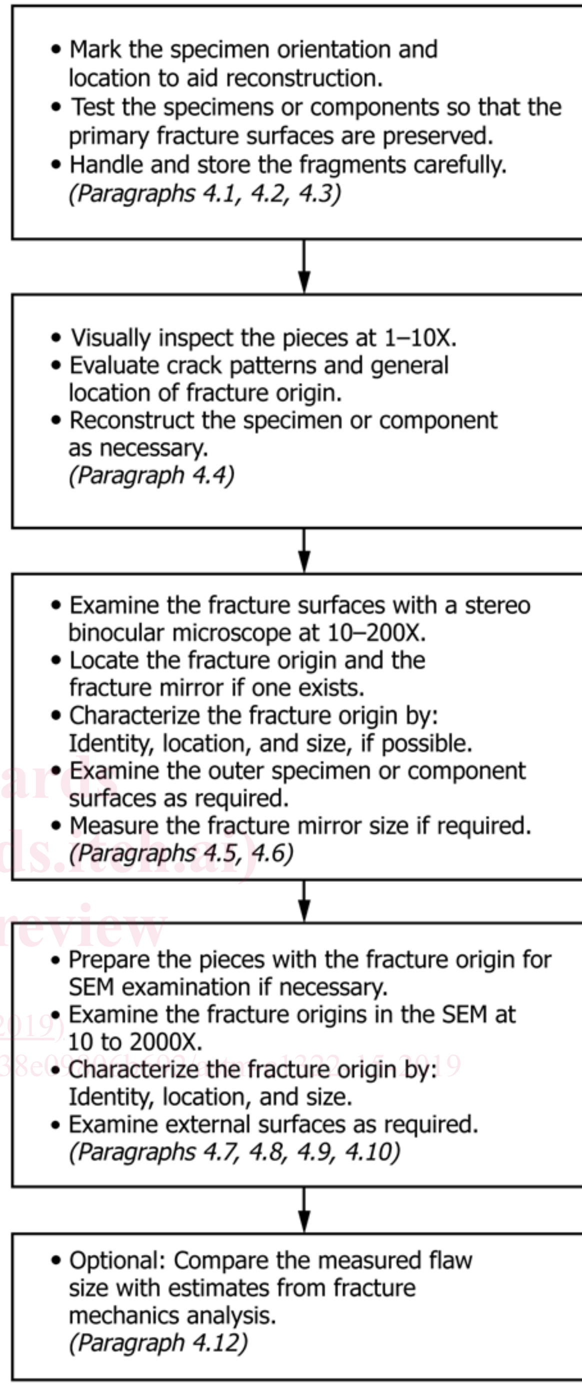
4.8 Carry out SEM examination (10 to 2000×) of both mating halves of the primary fracture surface.

4.9 Characterize the strength-limiting origin by its identity, location, and size. When appropriate, use the chemical analysis capability of the SEM to help characterize the origin.

4.10 If necessary, repeat 4.6 using the SEM.

4.11 Keep appropriate records, digital images, and photographs at each step in order to characterize the origin, show its location and the general features of the fractured specimen/component, as well as for future reference.

4.12 Compare the measured origin size to that estimated by fracture mechanics. If these sizes are not in general agreement, then an explanation shall be given to account for the discrepancy.



Keep appropriate records, digital images, and photographs at each step to assist in the origin characterization and for future reference.

FIG. 1 Simplified Schematic Diagram of the Fractographic Analysis Procedure

4.13 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, porosity, and phase distribution.

5. Significance and Use

5.1 This practice is suitable for monolithic and some composite ceramics, for example, 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.

5.2 This practice 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.

5.3 The procedures described within are primarily applicable to mechanical test specimens, although the same procedures may be relevant to component fracture analyses as well. It is customary practice to test a number of specimens (constituting a sample) 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 fracture analyses. Component fracture analysis is sometimes aided by cutting test pieces from the component and fracturing the test pieces. Fracture markings and fracture origins from the latter may aid component interpretation.

5.4 Optimum fractographic analysis requires examination of as many similar specimens or components as possible. This will enhance the chances of successful interpretations. Examination of only one or a few specimens can be misleading. Of course, in some instances the fractographer may have access to only one or a few fractured specimens or components.

5.5 Successful and complete fractography also requires careful consideration of all ancillary information that may be available, such as microstructural characteristics, material fabrication, properties and service histories, component or specimen machining, or preparation techniques.

5.6 Fractographic inspection and analysis can be a time-consuming process. Experience will, in general, enhance the chances of correct interpretation and characterization, but will not obviate the need for time and patience. Repeat examinations are often fruitful. For example, a particular origin type or key feature may be overlooked in the first few test pieces of a sample set. As the fractographer gains experience by looking at multiple examples, he or she may begin to appreciate some key feature that was initially overlooked.

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

5.8 The irregularities which act as fracture origins in advanced ceramics can develop during or after fabrication of the material. Large irregularities (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 manufacturing process. Other origins can be introduced after processing as a result of machining, handling, impact, wear, oxidation, and corrosion. These can be considered extrinsic origins. However, machining damage may be considered intrinsic to the manufacturing procedure to the extent that machining is a normal step of producing a finished specimen or component.

5.9 Regardless of how origins develop, they are either inherently volume distributed throughout the bulk (for example, agglomerates, large grains, or pores) or inherently surface distributed (for example, handling damage, pits from oxidation, or corrosion). The distinction is a consequence of how the specimen or component is prepared. For example, inclusions may be scattered throughout the bulk ceramic material (inherently volume distributed), but when a particular specimen is cut from the bulk ceramic material, the strength-limiting inclusion could be located at the specimen surface. This may frequently occur if the specimen is very thin. Thus, in a particular specimen, a volume-distributed origin can be volume located, surface located, near-surface located, or edge located. The distinction is important for Weibull analysis and strength scaling with size as discussed in Practice C1239.

5.10 As fabricators improve materials by careful process control, thus eliminating undesirable microstructural features, advanced ceramics will become strength-limited by origins that come from the large-sized end of the distribution of the normal microstructural features. Such origins can be considered mainstream microstructural features. In other instances, regions of slightly different microstructure (locally higher microporosity) or microcracks between grains (possibly introduced by thermoelastic strains) may act as fracture origins. 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. This practice can still be used to analyze such fracture origins, but specific origin definitions may need to be devised.

5.11 This practice is compatible with CEN Standard EN 843 Part 6.

6. Apparatus

6.1 *General*—Examples of the equipment described in 6.2 – 6.6 are illustrated in Appendix X4 and also the NIST Special Publication SP 960-16 (**2**).

6.2 *Binocular Stereomicroscope,* with adjustable magnification between 10 to 200× and directional light source (see Fig. X4.1). A camera or video monitor system used with this microscope is a useful option (see 6.6 and Fig. X4.2). Basic binocular stereomicroscopes have magnification ranges to the eyes of about 8 to 32× or 10 to 40×, but these limit one's view of a small fracture origin flaw and greater magnifications are needed. Stereomicroscopes with upper magnifications of 100× or as high as 300× (available with many stereomicroscopes) are