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SIST-TS CEN/TS 843-6:2004

01-september-2004

Sodobna tehniļ na keramika ï Monolitska keramika - Mehanske lastnosti pri sobni temperaturi ï 6. del: Vodilo za fraktografsko raziskavo

Advanced technical ceramics - Monolithic ceramics. Mechanical properties at room temperature - Part 6: Guidance for fractographic investigation

Hochleistungskeramik - Monolithische Keramik - Mechanische Eigenschaften bei Raumtemperatur - Teil 6: Leitlinie für die fraktographische Untersuchung

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Céramiques techniques avancées - Céramiques monolithiques -Propriétés mécaniques a température ambiante - Partie 6: Guide pour l'analyse fractographique

SIST-TS CEN/TS 843-6:2004
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Ta slovenski standard je istoveten z: CEN/TS 843-6:2004

ICS:

81.060.30 Sodobna keramika Advanced ceramics

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SIST-TS CEN/TS 843-6:2004

TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

CEN/TS 843-6

June 2004

ICS 81.060.30

English version

Advanced technical ceramics - Monolithic ceramics. Mechanical properties at room temperature - Part 6: Guidance for fractographic investigation

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This Technical Specification (CEN/TS) was approved by CEN on 17 November 2003 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

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Ref. No. CEN/TS 843-6:2004: E

SIST-TS CEN/TS 843-6:2004

Contents

Page

iTeh STANDARD PREVIEW
(standards.iteh.ai)
SIST-TS CEN/TS 843-6:2004
https://standards.iteh.ai/catalog/standards/sist/16ef12ec-f40b-47aa-9014-90c622da8efa/sist-ts-cen-ts-843-6-2004

SIST-TS CEN/TS 843-6:2004

Foreword

This document CEN/TS 843-6:2004 has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

Annexes A to E are informative.

This document includes a Bibliography.

EN 843 *Advanced technical ceramics – Monolithic ceramics – Mechanical properties at room temperature* consists of six parts:

Part 1: *Determination of flexural strength*

Part 2: *Determination of elastic moduli*

Part 3: *Determination of subcritical crack growth parameters from constant stressing rate flexural strength tests*

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Part 4: *Vickers, Knoop and Rockwell superficial hardness tests*

Part 5: *Statistical analysis*

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Part 6: *Guidance for fractographic investigation*

At the time of publication of this Technical Specification, Part 1 is a European Standard, while Parts 2 to 5 are European Prestandards.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom

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1 Scope

This Technical Specification contains guidelines to be adopted when evaluating the appearance of the fracture surface of an advanced technical ceramic. The purpose in undertaking this procedure can be various, for example, for material development or quality assessment, to identify normal or abnormal causes of failure, or as a design aid.

NOTE Not all advanced technical ceramics are amenable to fractography. In particular, coarse-grained ceramics can show such rough surfaces that identifying the fracture origin may be impossible. Similarly, porous materials, especially those of a granular nature, tend not to fracture in a continuous manner, making analysis difficult.

2 Normative references

This Technical Specification incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this Technical Specification only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN ISO/IEC 17025 *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:1999).*

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3 Terms and definitions

For the purposes of this Technical Specification, the following terms and definitions apply.

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3.1 General terms

3.1.1
crack
distinct microstructural discontinuity arising during or after manufacture caused by the action of thermal and/or mechanical stress and leading to the generation of new surfaces which do not completely separate

3.1.2
flaw
inhomogeneity which, through stress concentration, can act as a strength defining feature

NOTE The term flaw used in this sense does not imply that the component is defective.

3.1.3
fracture
process of propagation of a crack through a test-piece or component

3.1.4
fracture origin
source from which failure commences

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3.2 Terms classifying inherently volume-distributed fracture origins

3.2.1
agglomerate
unintentional microstructural inhomogeneity usually of altered density, for example a cluster of grains of abnormal size, particles, platelets or whiskers, resulting from non-uniformity in processing

3.2.2
compositional inhomogeneity
local variations in chemical composition, usually manifest as agglomerates (3.2.1), or as areas denuded of or enriched in dispersed phases, or as changes in grain size

3.2.3
delamination
generally planar crack within a material arising from the method of manufacture

3.2.4
inclusion
discrete inhomogeneity, usually as a result of inorganic contamination by a foreign body not removed during firing

3.2.5
large grain
grain which is of abnormally large size as a result of poor particle size control or accelerated grain growth, and which can act as a flaw (3.1.2)

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3.2.6
pore
cavity or void within a material, which may be isolated or continuously interconnected with others

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3.2.7
porous region
zone of enhanced porosity, usually three-dimensional in nature and resulting from inhomogeneity or organic contamination in processing

3.2.8
porous seam
zone of enhanced porosity, usually linear or planar in nature and resulting from inhomogeneity or organic contamination in processing

3.3 Terms classifying inherently surface-distributed fracture origins

3.3.1
chip
small flake of material removed from a surface or an edge of an item or its fracture surface

3.3.2
handling damage
scratches, chips or other damage resulting from contact between items, test-pieces or fracture surfaces, not present normally

3.3.3
machining damage
result of removal of small chips (see 3.3.1) or the formation of scratches at, or cracks near, the surface resulting from abrasive removal of material

SIST-TS CEN/TS 843-6:2004

3.3.4
open pore
void connected to the external surface, usually by virtue of machining

3.3.5
pit
surface depression or surface connected shallow pore, usually resulting from manufacturing conditions or interaction with the external environment

3.4 Terms classifying features on fracture surfaces

3.4.1
fracture lines
ridges or troughs running approximately parallel to the direction of propagation of a crack front, usually in the hackle (3.4.2) region

NOTE In some cases, particularly with materials with low fracture toughness, additional lines can be found on fracture surfaces resulting from interactions of the crack with free surfaces or other features, including so-called Wallner lines, arrest lines, wake hackle, etc. Definitions of such terms can be found in ASTM C1256 (see reference [1] in the Bibliography).

3.4.2
hackle
region of rough fracture outside the mirror (3.4.3) and mist (3.4.4) regions, often with ridges or troughs emanating radially from the fracture origin (3.1.4)

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3.4.3
mirror
area of a fracture surface, usually approximately circular (or semicircular for near-edge fracture origins) and immediately surrounding a fracture origin (3.1.4), which is relatively flat and featureless compared with regions further removed from the fracture origin

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NOTE Not all materials or fractures show obvious fracture mirrors. They tend to be visible most clearly in high-stress, accelerating fractures from small flaws.

3.4.4
mist
halo around the outer region of the mirror (3.4.3) where the roughness is enhanced with a texture elongated in the direction of fracture

NOTE The mist region is most clearly seen in glasses, glass-ceramics or ceramics with very fine grain sizes which produce smooth surfaces on fracture.

4 Significance and use

Fractography is recommended as a routine diagnostic aid to the interpretation of fracture tests on test-pieces or of failures in components. Observation of the macroscopic features of fragments, such as cracks and their relative disposition, chips and scratches, provides information about the likely directions of stressing. Observation of intermediate scale features on the fracture surface, such as the shape of hackle (3.4.2) and fracture lines (3.4.1) give indications of the approximate position of the fracture origin (3.1.4). Microscopic observations give information on the nature of the fracture origin, and thus may provide evidence of the reasons for fracture.

The accumulation of additional information about the conditions of fracture (stresses, forces, temperature, time under stress, likelihood of impact, etc.) is highly desirable for achieving justifiable conclusions.

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5 Apparatus

5.1 Preparation and cleaning facilities

5.1.1 ***Cutting wheel,*** for large specimens. A diamond-bladed saw.

NOTE This is needed to cut small samples for microscope observation, particularly in the scanning electron microscope

5.1.2 ***Ultrasonic bath,*** for cleaning the fracture surface.

5.1.3 ***Compressed air supply,*** for drying specimens after cleaning and for removal of dust or lint. The supply should be dry and oil-free.

5.2 Observational facilities

5.2.1 ***Small hand lens,*** with a magnification in the range 3 to 8 times.

5.2.2 ***Optical microscope,*** preferably with photomicrographic facilities, and with variable magnification in the range 5 to 50 times.

NOTE As an alternative to photomicrographic facilities, a camera with appropriate lenses and a macrophotography stand.

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5.2.3 ***Illumination system,*** a light source that can be positioned to the side of the test-piece to provide contrast on the fracture surface.

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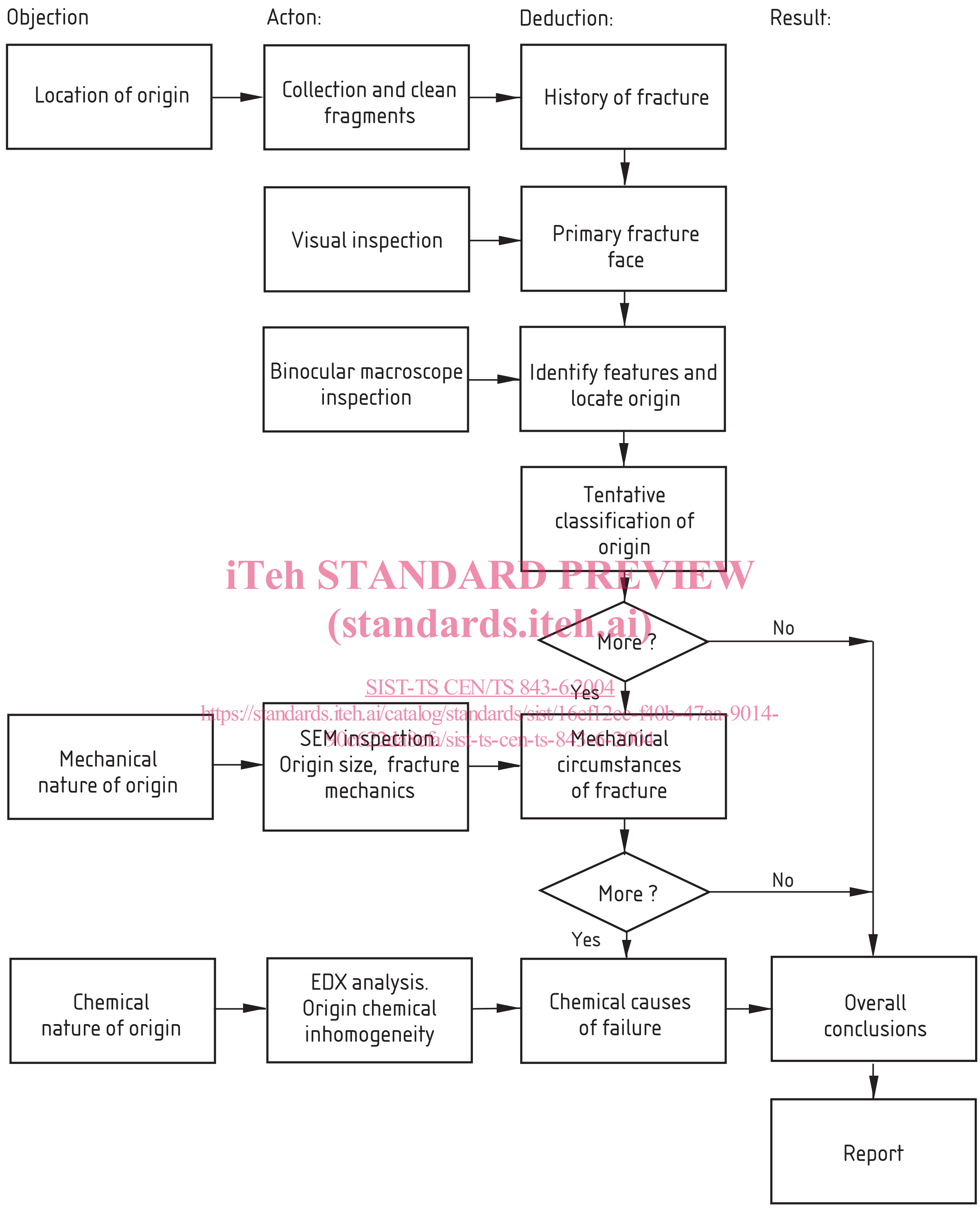
5.2.4 ***Scanning electron microscope (SEM),*** preferably with energy-dispersive X-ray (EDX) analysis equipment fitted.

SIST-TS CEN/TS 843-6:2004



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SIST-TS CEN/TS 843-6:2004
https://standards.iteh.ai/catalog/standards/sist/16ef12ee-f40b-47aa-9014-90...a/sist-ts-cen-ts-843-6-2004

Figure 1 - Flow chart for general fractographic procedure

SIST-TS CEN/TS 843-6:2004

6 Recommended procedure

6.1 Outline

The sequence of steps in undertaking fractography on a specimen is outlined in Figure 1. It should be noted that not all the steps will be necessary on every occasion; for example, if only a check on approximate position of failure is needed, SEM examination is not generally necessary. Thus, the following series of paragraphs should be used as appropriate to the task, defined by the type of investigation needed.

6.2 Specimen storage and cleaning of fracture surfaces

Fracture surfaces are rough and are prone to contamination in handling and storage. Contamination can lead to misinterpretation of observed features, especially in the SEM. Where possible, store fractured fragments separately in clean, dry, conditions in which the fracture surfaces cannot contact foreign bodies.

NOTE Storage in paper or plastic containers can lead to pick-up of contamination. Glass vials minimise risks, but can damage surfaces if the specimen is loose in the vial. It is recommended to avoid the use of tape or mouldable compounds as the adhesive is difficult to remove once contaminating the fracture surface.

Avoid handling with naked hands; use tweezers or surgical gloves to avoid contamination from body oils.

Cleaning facilities are required to allow removal of such contamination without damaging further the fracture surface. It is recommended that solvents such as acetone or ethyl alcohol are used in conjunction with a laboratory ultrasonic bath to remove soluble or loose contamination.

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Dry the specimens using compressed air.

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6.3 Visual inspection

6.3.1 Examine visually all the available fragments using a good light source and a hand lens as appropriate.

6.3.2 Label all fragments with an indelible marker at positions that are remote from the surfaces of interest. Make a sketch of the labelled fragments for future reference.

6.3.3 Where there are several fragments, use the pattern of cracks to identify the originating fracture surface (the primary fracture):

NOTE 1 Annex A contains some examples of crack patterns in test-pieces and components.

NOTE 2 It is recommended not to attempt to fit the fracture pieces tightly together since this may induce further damage on the fracture surfaces which will impede subsequent investigations.

6.3.4 Examine the primary fracture surface for evidence of an origin of the fracture. This may be identified by tracing back any radiating ridges or grooves.

NOTE 1 Annex B shows some examples of fracture surface patterns which may aid this step. However, it should be noted that:

1) Not all ceramic materials show clear fracture markings. High strength fine-grained or amorphous materials show fracture features the best. In contrast, the roughness of the fracture surface in coarse-grained or weaker materials may be too great, and obscures the fracture markings.

2) Features such as mist or hackle can be absent as a consequence of the size of the test-piece or the level of fracture stress. These features only develop if the crack reaches a sufficient velocity within the test-piece cross-section. An example is the case of subcritical crack growth, or in the fracture of small test-bars.

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NOTE 2 It can be useful to hold the fracture surface at grazing incidence to a light source and observe any changes in apparent roughness, using a hand lens if necessary. The region surrounding the fracture origin can be smoother than the remainder of the surface.

Note any evidence from the fragments.

6.4 Optical microscope examination

6.4.1 Using oblique illumination to highlight the roughness of the fracture surface, and hence the fracture markings, examine the fragments under an optical microscope at low magnification (x3 to x10) to confirm the visual findings concerning the approximate origin. Table 1 advises on the visibility of origins using optical microscopy.

NOTE 1 Many ceramics are translucent, and the scattering or oblique illumination in the surface layer can obscure fracture markings. It is recommended:

1) to place a height-adjustable light barrier parallel to the fracture surface to shield the side of the specimen;

2) if appropriate, to rotate the specimen so that a clear impression is obtained of the fracture markings under illumination from all directions;

3) if appropriate, to coat the fracture surface with a thin layer of an opaque substance, such as a metal, e.g. gold. However, coating should be used with discretion if subsequent SEM/EDX analysis is to be performed.

NOTE 2 It can be helpful to the identification of the fracture origin if the two mating halves of the fracture surface are placed side by side with the respective halves of the fracture origin adjacent. It is sometimes easier to see the radial pattern of marks in this way.

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6.4.2 If appropriate, sketch or record the images photographically.

SIST-TS CEN/TS 843-6:2004
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6.4.3 Increase the magnification in stages and examine the suspected origin. If possible, identify any feature at the origin, including the detailed pattern of local marks, or any marks or damage on the external surface which may have caused the failure. Take photomicrographs if appropriate.

NOTE 1 At magnifications above about x200 fracture surfaces are generally too non-planar for effective optical microscope examination, and are difficult to illuminate adequately from the side. In some cases, mixed normal and oblique lighting can reveal important features.

NOTE 2 The radiating pattern of fracture marks can often be traced back to the origin, but only if these are clearly identifiable.

6.5 Identification of major fracture surface features

Identify the major features of the fracture surface in terms of fracture lines (3.4.1) emanating from a focal point in an equivalent manner on the two fracture surfaces. Identify strongly hackled regions, and any mirror and mist regions. Identify the position and tentative nature of the fracture origin in relation to the component or test-piece geometry and likely stressing. Correlate these observations with any ancillary observations of the surface condition.

NOTE 1 The interpretation of the visual observations may not necessarily be straightforward, and optical microscopy may not have adequate resolution or clarity of image to allow positive identification of the cause of failure. If higher magnification is required, or confirmation of the chemical nature of the origin, SEM/EDX examination should be employed (6.6, 6.8). However, a number of possible types of feature can be identified (not all in every case), which will provide evidence for the report.

NOTE 2 The radius of the mirror, if present, is linked to the fracture stress at the point of failure through an empirical fracture mechanics relationship. If the fracture stress and the mirror constant are known (see annex D), the mirror size can be calculated, which is a guide to interpretation of a fracture origin. Alternatively, if the mirror radius and mirror constant are known, the fracture stress can be estimated.

NOTE 3 Particularly with regard to optical observations, it is important to describe the origin in terms of its physical form, and not how it appears under particular observational conditions.

SIST-TS CEN/TS 843-6:2004

Table 1 — Visibility of fracture origins

Origin name	Comment	Identifiable by optical microscopy or SEM	Examples in annex C or D
Pore (3.2.6)	Large single pores are often irregular in shape, and can act as fracture origins, especially when close to or connected to the surface, e.g. when exposed by virtue of machining.	Optical, although SEM better for translucent materials	C1.1, C1.2
Porous region (3.2.7)	A zone of closely spaced pores distributed in three dimensions can be difficult to identify positively except at high magnification.	SEM unless large	
Porous seam (3.2.8)	A zone of closely spaced pores distributed in a planar or near planar arrangement may result from incomplete compaction, or inadvertent organic matter, or a closed delamination.	SEM unless large	
Delamination (3.2.3) or green-body crack (3.1.1),	A planar or near planar open cavity resulting from fracture during pressing of the green shape, or during ejection from a die cavity, which does not heal completely in firing. Usually identifiable as being at an angle to the general plane of fracture, and as having a different internal surface topography from a fractured region.	Optical or SEM	C5.2
Inclusion (3.2.4)	An inhomogeneity of different chemical composition from that of the ceramic material which is often linked with a pore or locally modified grain size, but which may become obvious only with backscattered electron SEM or energy dispersive X-ray imaging.	SEM for chemical information, optical only if large and discoloured	C4.2
Large grain(s) (3.2.5)	A single or a group of abnormally large grains is usually caused by a compositional inhomogeneity, excessive firing temperature, or occasionally from poor milling of powders.	SEM or optical if large	C3.1
Agglomerate (3.2.1)	A dense cluster of grains distinguishable from the rest of the microstructure, but often surrounded by a porous seam created by differential shrinkage on sintering.	SEM	C2.1, C2.2
Compositional inhomogeneity (3.2.2)	A region where there is a local change in composition modifying the microstructure or creating a void.	SEM for chemical information	C4.1
Surface chip (3.3.1)	Damage at the external surface, often along an edge, can initiate cracking, and is usually identified by additional local damage. Fracture may initially be out of plane of final fracture.	Optical or SEM	D2
Surface crack (3.1.1)	A pre-existing crack which can result from mechanical or thermal damage or during handling in production can be hard to identify, but is usually out of the plane of final fracture.	Optical or SEM	C9
Surface pit (3.3.5)	A cavity at the surface resulting from external influences, e.g. oxidation, requires examination of the relationship between the fracture origin and the external surface.	Optical or SEM	C8.1, C8.2
Open pore (3.3.4)	A cavity at the surface which results from the processing method used to prepare the component or test-piece can typically be distinguished from a pit by its depth or by surface morphology similar to normal surface.	Optical or SEM	D1
Machining damage (3.3.3)	Surface or sub-surface shallow damage such as chips or cracks can be produced by machining, leading to apparently extended fracture origins, often of semi-elliptical shape.	SEM	C7.1, C7.2
Handling damage (3.3.2)	Scratches or other abnormal damage resulting from abnormal handling during processing.	Optical or SEM	C6

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90a622da8efa/sist-ts-cen-ts-843-6-2004