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Sodobna tehnična keramika – Metode za preskušanje keramičnih prevlek – 9. del: Ugotavljanje pokanja zaradi obremenitve

Advanced technical ceramics - Methods of test for ceramic coatings - Part 9: Determination of fracture strain

Hochleistungskeramik - Verfahren zur Prüfung keramischer Schichten - Teil 9: Bestimmung der Bruchdehnung

Céramiques techniques avancées - Méthodes d'essai pour revetements céramiques - Partie 9: Détermination de la déformation a la rupture

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Advanced technical ceramics – Methods of test for ceramic coatings – Part 9: Determination of fracture strain

Céramiques techniques avancées – Méthodes d'essai pour revêtements céramiques – Partie 9: Détermination de la contrainte à la rupture

Hochleistungskeramik – Verfahren zur Prüfung keramischer Schichten – Teil 9: Bestimmung der Bruchdehnung

This Technical Specification (CEN/TS) was approved by CEN on 28 December 2003 for provisional application.

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Foreword

This Technical Specification (CEN/TS 1071-9:2004) has been prepared by Technical Committee CEN/TC 184 "Advanced technical ceramics", the secretariat of which is held by BSI.

EN 1071 *Advanced technical ceramics — Methods of test for ceramic coatings* consists of 11 parts:

Part 1: *Determination of coating thickness by contact probe profilometer*

Part 2: *Determination of coating thickness by the crater grinding method*

Part 3: *Determination of adhesion and other mechanical failure modes by a scratch test*

Part 4: *Determination of chemical composition*

Part 5: *Determination of porosity*

Part 6: *Determination of the abrasion resistance of coatings by a micro-abrasion wear test*

Part 7: *Determination of hardness and Young's modulus by instrumented indentation testing*

Part 8: *Rockwell indentation test for evaluation of adhesion*[1)]

Part 9: *Determination of fracture strain*

Part 10: *Determination of coating thickness by cross sectioning*[1)]

Part 11: *Measurement of internal stress with the Stoney formula*[1)]

Parts 7 to 11 are Technical Specifications.

This Technical Specification includes a bibliography.

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 the United Kingdom.

1) In preparation at the time of publication of this Technical Specification.

Introduction

The fracture strain of a coating is a critical factor often determining the performance of a coated product. Clearly if stressed either directly or due to thermal effects (thermal expansion coefficient mismatch between the coating and substrate) coating cracking can occur if the critical fracture stress/strain is exceeded, and in many cases the effectiveness of the coating will be reduced. For example, corrosion resistant coatings loose their protective character if cracking occurs, and optical coatings become ineffective when cracked. In many cases cracking is the first stage of a much more serious form of failure in which large areas of the coating can spall.

The extent to which coated components can withstand external applied loads is an important property in the application of any coated system, and usually the failure stress is required. For calculation of the stress both the fracture strain and Young's modulus of the coating should be known. CEN/TS 1071-7 can be used to measure the Young's modulus by depth sensing indentation, but there are other methods involving flexure and impact excitation that may also be applied [1], [2]

1 Scope

This part of EN 1071 describes a method of measuring the fracture strain of ceramic coatings by means of uniaxial tension or compression tests coupled with acoustic emission to monitor the onset of cracking of the coating. Tensile or compressive strains can also be applied by flexure using four-point bending. Measurements can be made in favourable cases at elevated temperatures as well as at room temperature.

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 10002-1, *Metallic materials - Tensile testing* – Part 1: *Method of test at ambient temperature*

EN 10002-5, *Metallic materials - Tensile testing* – Part 5: *Method of testing at elevated temperature*

ISO 12106, *Metallic materials - Fatigue testing - Axial-strain-controlled method*

3 Terms and definitions

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

3.1
fracture strain
strain required to create a detectable crack in the coating

NOTE The presence of the crack can be detected using optical or scanning electron microscopy, or indirectly using acoustic emission signals

3.2
acoustic emission (AE)
generation of acoustic signals; these signals are recorded as hits, counts, energy or amplitude

NOTE See Figure 1 for definition of AE signals.

3.3
AE hit
single acoustic event above a set threshold

3.4
AE energy
area of the waveform of an AE hit

3.5
AE amplitude
peak of the waveform of an AE hit

3.6
AE counts
number of times the AE waveform passes a set threshold within a single hit

3.7
AE threshold
arbitrary AE amplitude at which AE hits are deemed to be significant and above the AE signals generated by the test equipment

3.8
waveguide
metallic wire connecting (usually by spot welding) the sample to the AE transducer

4 Significance and use

This test procedure covers the measurement of fracture strain in tension or compression in coatings subject to mechanical stress at ambient or elevated temperature.

The method is applicable to cases where the substrate is sufficiently ductile such that fracture of the coating occurs before the substrate. In addition, if during plastic deformation of the substrate acoustic signals are generated, this may interfere with those caused by coating fracture. Where possible it is recommended that a test be carried out with the uncoated substrate to determine whether such extraneous AE signals occur.

5 Principle

Specimens of appropriate geometry are submitted to a mechanical stress; the subsequent strain is measured and the onset of coating failure is detected. The test draws upon the expertise of standard tensile and compressive tests but requires additional care due to the precision required of the measurements. The applied stress may be tensile or compressive and may be applied directly or in flexure. The test shall be carried out to satisfy the requirements of accepted standards for mechanical testing of materials under the selected method of loading.

NOTE 1 Detection of the fracture of coatings can be carried in a number of ways. The most convenient is to use acoustic emission (AE), which allows continuous monitoring of the specimen. Acoustic signals are produced when a crack forms. These signals are captured using suitable detectors and the signals generated are then analysed. In many cases a waveguide is used to carry the signal from the specimen to the detector; this waveguide is normally a metallic material. Use of two AE detectors can help to eliminate extraneous signals coming from the loading mechanism. Commercially available AE systems can be used for this work.

NOTE 2 Where AE cannot be used, crack detection is possible by high resolution video systems, which may allow continuous monitoring. Alternatively, optical or scanning electron microscopy can be used to examine the samples. Normally this is done post-test, but *in situ* examination is also possible.

6 Apparatus and materials

6.1 Instrumentation

6.1.1 In simplest terms the equipment required is a mechanism to apply load to the specimen; extensometry to measure the strain; and apparatus to detect/monitor fracture of the surface layer. Load is normally applied continuously through servo-electric testing machines; the load capacity of the frame should be sufficient to allow straining of the specimen to beyond the yield point of the substrate material. Continuation of the test to complete separation of the specimen is not normally required.

6.1.2 For flexural testing a suitable test jig is required – four-point bending is recommended as this applies more uniform bending moment over the gauge length. A suitable jig is shown in Figure 2.

6.1.3 Extensometry should be sufficiently precise to measure strain at a resolution of 0,01%.

6.1.4 For tests at high temperatures using the uniaxial test configuration a furnace is required which allows access for attachment of load frame, extensometry, thermocouples and waveguides to transmit the AE signals to the AE detector(s). For the four-point bend configuration, an oxidation resistant jig shall be used.

NOTE Deformation of oxide layers formed on a metallic jig will probably contribute to AE signals during the test.

6.1.5 Crack detection in the coating may be performed visually or by monitoring AE. Visual inspection requires suitable long focal length video facilities with a field of view containing the gauge length. At high temperatures the availability of a cool path to the video camera is also required to avoid shimmer of the image.

6.2 Specimen preparation

6.2.1 Standard specimens shall be used as appropriate for the uniaxial or flexure test configurations; for uniaxial tensile tests the specimen shapes are defined in EN 10002-1, for compression tests in ISO 12106 and for flexure simple bar shaped samples of appropriate thickness can be used. The coating may be deposited on to the sample after machining to the required shape, or in the case of flat specimens the test piece can be machined from the coated material. In the latter case, care shall be taken to avoid damage to the test region that may cause premature fracture. Generally the surface of the coating should not be ground or polished except where there is a requirement so to do.

6.2.2 The strain that is measured using this technique represents a summation of the inherent fracture strain of the surface layer and the residual strain present at the test temperature. For a sample with a residual compressive strain, the measured tensile strain is the sum of the coating fracture strain and the residual compressive strain, and vice versa for a sample with residual tensile strain. For most purposes it is the inherent fracture strain that is required, therefore it is recommended that the residual strain in the coating is measured at the test temperature by an appropriate technique e.g. X-ray diffraction for crystalline materials or the Stoney bend test for amorphous materials. This may be carried out on each test specimen but it is normally sufficient to measure only one specimen under each coating condition.

6.2.3 Specimens for testing under flexural loading with AE detection require that the coating is removed from one face in order to avoid AE detection of failure events from both tension and compression. In addition, it is also recommended that coating is removed from the region where contact is made in the test jig. This precaution reduces the amount of extraneous signal arising from local fracture of coating under high point loading. For testing at elevated temperatures it is also recommended that the specimen be coated with a corrosion-resistant coating that is acoustically quiet (suitable proprietary coatings are readily available). Where the material does not have sufficient oxidation resistance, consideration shall be given to carrying out the tests in an inert environment, since the oxide layers that would form could also crack and hence contribute to the AE signals. Care shall be taken to ensure that the sample has attained the test temperature before commencing the test.

6.2.4 Specimens for tests under flexural loading shall be simple beams with dimensions suitable for the test jig. Typical specimens are 50 x 5 x 2 mm, but the dimensions are dependent upon the strength of the material at the test temperature.

NOTE Care should be taken to ensure that the specimens have sufficient thickness that uniform straining is achieved - the onset of localised deformation around the rollers is material, thickness and temperature dependent; the specimen design for each material of interest should therefore be reviewed prior to starting the measurement programme.

6.2.5 Specimens for testing under direct tensile loading may be planar or circular cross-section. The choice is governed mainly by the form of material available, as both specimen types have advantages and disadvantages, with neither geometry showing sufficient superiority over the other to present a definitive case for its use. In both cases care shall be taken to minimise the introduction of stress concentrations that would induce early failure of the coating.

6.2.6 For testing under direct compression, specimens with circular gauge cross-section shall be used according to ISO 12106. Care shall be taken in the choice of gauge length and cross-section to avoid buckling and bulging of the specimen during compressive loading.