BRITISH STANDARD

BS EN 1071-9:2009

Advanced technical ceramics — Methods of test for ceramic coatings

Part 9: Determination of fracture strain

ICS 81.060.30

British Standards

National foreword

This British Standard is the UK implementation of EN 1071-9:2009.

The UK participation in its preparation was entrusted to Technical Committee RPI/13, Advanced technical ceramics.

A list of organizations represented on this committee can be obtained on request to its secretary.

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 August 2009

ISBN 978 0 580 63848 0

Amendments/corrigenda issued since publication

Date	Comments

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 1071-9

July 2009

ICS 81.060.30

Supersedes CEN/TS 1071-9:2004

English Version

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 déformation à la rupture

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

This European Standard was approved by CEN on 19 June 2009.

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Ref. No. EN 1071-9:2009: E

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Foreword

This document (EN 1071-9:2009) has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by January 2010, and conflicting national standards shall be withdrawn at the latest by January 2010.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes CEN/TS 1071-9:2004.

EN 1071 *Advanced technical ceramics – Methods of test for ceramic coatings* consists of the following 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 by electron probe microanalysis (EPMA)*

— *Part 5: Determination of porosity* [withdrawn]

— *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* [withdrawn]

— *Part 8: Rockwell indentation test for evaluation of adhesion*

— *Part 9: Determination of fracture strain*

— *Part 10: Determination of coating thickness by cross sectioning*

— *Part 11: Determination of internal stress by the Stoney formula*

— *Part 12: Reciprocating wear test* [1)]

— *Part 13: Determination of wear rate by the pin-on-disk method* [1)]

Parts 7, 8 and 11 are Technical Specifications. Part 7 was withdrawn shortly after publication of EN ISO 14577-4:2007.

[1)] In preparation at the time of publication of this European Standard.

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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 lose 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 it is necessary to know the failure stress. For calculation of the stress both the fracture strain and Young's modulus of the coating should be known. EN ISO 14577-4 [1], which replaced Technical Specification 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 [2], [3].

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

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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*

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:2005)*

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

3 Terms and definitions

For the purposes of this document, 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 that 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

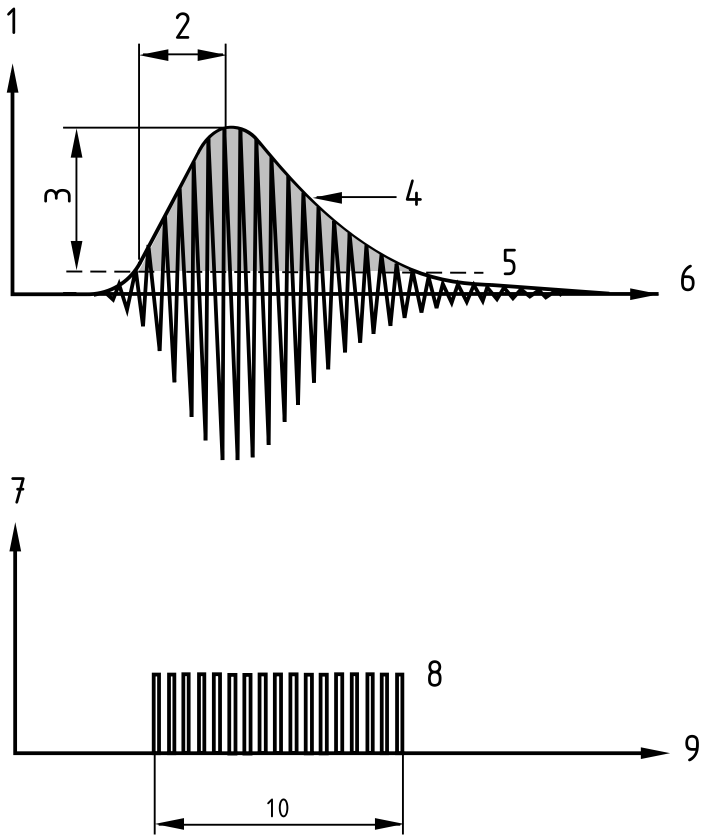
3.4
AE energy
area of the waveform of an AE hit

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



Key

1 Volts
2 Rise time
3 Amplitude
4 Energy
5 Threshold
6 Time
7 Threshold crossing
8 Counts
9 Time
10 Duration

Figure 1 - Schematic representation of AE signals

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.

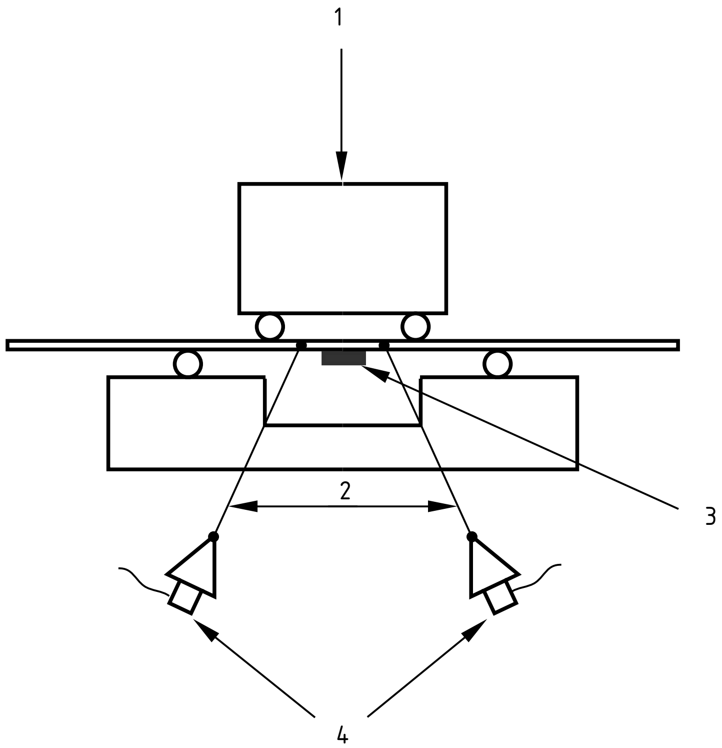
6.1.2 For flexural testing a suitable test jig is required – four-point bending is recommended as this applies more uniform bending moment over the gauge length. A suitable jig is shown in Figure 2.

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.



Key

1 Load

2 Waveguides

3 Coating

4 AE detectors

Figure 2 - Schematic diagram of a flexural jig with acoustic emission sensors

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.

6.2.7 Replicate tests shall be carried out, and at least five are required.

NOTE Coating fracture is well described by fracture mechanics in which defect size and distribution control behaviour. Thus to obtain a statistically valid result replicate testing is essential. It is recommended that ten samples should be tested, but it is recognised that these many samples may not always be available.

6.2.8 The coating/substrate specific parameters include:

a) coating thickness;

b) residual stress.

These parameters shall be kept constant if a direct comparison is made between two or more test pieces unless corrections are applied to normalise the data.

7 Test procedure

7.1 Calibration

The instrument shall be calibrated according to the procedures set out in the appropriate standard. Ensure that all systems are operating correctly, and introduce the test piece after appropriate preparation as detailed in 6.2.

7.2 Sample loading

The application of load may be performed under load, displacement or strain control. The rate of loading is not critical but should conform to the rates specified for tensile testing of metals at ambient (see EN 10002-1) or elevated temperatures (see EN 10002-5). Load shall be applied smoothly in order to avoid impact loading of the coating that may cause premature cracking. The loading mechanism shall also ensure that bending stresses due, for example, to twisting of a flexural beam or by misalignment of uniaxial specimens, are not introduced.

7.3 Strain determination

In four-point flexure, strain, ε, is calculated from the dimensions of the test jig and the relative displacement of the upper and lower rollers according to the following expression:

$$\varepsilon = \frac{3\,\delta\,d}{u(3s-4u)}$$

where

δ is the relative displacement between the upper and lower rollers in millimetres;

d is the test piece thickness in millimetres;

u is distance between outer and inner rollers in millimetres;

s is the spacing of the outer rollers in millimetres.

NOTE 1 This expression is valid for strains of < 0,3 % [4]. The spacing of the rollers is fixed and therefore does not vary from test to test; the thickness of the specimen is measured by micrometer to an accuracy of 0,01 mm; the relative displacement of the upper and lower rollers is measured by a transducer and an accuracy of 0,01 mm can readily be achieved. This leads to a repeatability for the measurement of ~ 1,5% of the value and a reproducibility of 8% if the difference in roller spacing between test jigs is 0,5 mm.

NOTE 2 To achieve the same repeatability in direct loading, extensometry should be used that will provide an uncertainty of < 1,5 % of the measured strain at the strain level of coating fracture. This can be achieved by extensometry of grade 0,2 as described in EN ISO 9513 [5] when the gauge length of the specimen exceeds 2,7 mm. A grade 0,5 extensometer requires a minimum gauge length of 6,8 mm.

7.4 Crack detection

7.4.1 Continuous monitoring of the AE from the specimen under test may provide enough information to determine the onset of coating cracking in many cases, but care shall always be taken to ensure that the signals detected originate from cracking events and not from other sources. The use of a two-sensor system with position location software is strongly recommended to identify the AE signals coming from within the gauge length of the specimen. A sample of the uncoated substrate material shall be tested in order to determine if any AE originates within the bulk material at strains similar to the fracture strain of the coating. If this proves to be the case then AE cannot be used as a reliable detection method for coating cracking. This test shall be used to set the AE threshold parameters to be used during the test with the coated sample.

NOTE 1 AE signals usually show the behaviour illustrated in Figures 3 and 4 and require careful interpretation to derive the failure strain of the coating. This failure strain can be defined by the back extrapolation of the region where the number of events is rising rapidly with strain. The tangent to the point of inflection on the cumulative AE plot is often used (Figure 4).

NOTE 2 Events occurring before this region can be attributed to cracking events that do not propagate through the entire thickness of the coating or to general 'noise' in the system. Events occurring in the upper plateau of the trace are attributed to repeated cracking of the coating leading to a decrease in crack spacing.

Other methods of determining the onset of coating fracture from the AE signals can be used. The first signal above the AE threshold level is also valid, and as can be seen from Figure 3 discrimination is good in this case. The method used should be optimised for the system under investigation. Also in certain cases particularly for very brittle coatings, the occurrence of a high AE energy can be a good indication of coating cracking.

7.4.2 Visual inspection of the specimens during the test is an alternative to AE. Video equipment with long focal length is available to enable direct observation during the test. Problems may be encountered during flexural loading but these can be overcome by the use of fibre optics and suitable jig design.

7.4.3 An alternative technique for room temperature testing is to periodically produce a plastic replica of the specimen surface for subsequent examination using scanning electron microscopy [6]. This method only gives intermittent strain monitoring.

7.5 Test parameters

The specific test parameters include:

a) loading rate;

b) applied load;

c) displacement;

d) time;

e) strain at onset of fracture;

f) AE threshold, if used;

g) strain at onset of AE, if used;

h) sample dimensions;

i) four-point bending jig inner and outer roller spacing, if used.

All the above parameters shall be kept constant if a direct comparison is made between two or more test pieces unless corrections are applied to normalise the data.

8 Report

The report shall be in accordance with the reporting provisions of EN ISO/IEC 17025 and shall contain at least the following information:

a) name and address of testing establishment;

b) date of test;

c) on each page, a unique report identification and page number;

d) customer name and address;

e) reference to this standard, i.e. 'determined in accordance with EN 1071-9';

f) an authorising signature;

g) any deviation from the method described, with appropriate validation, i.e. demonstrated to be acceptable to the parties involved;

h) a description of the test machine employed;

i) method of crack detection, and, if AE is used, the criterion for onset of cracking;

j) details of the checks carried out on the test machine including calibration;

k) details of the test material type, manufacturing code, batch number, etc.;

l) coating parameters for the test specimens - if known;

m) coating thickness;

n) details of the preparation of the surface and sample geometry;

o) strain rate;

p) individual test results, the arithmetic mean results and the standard deviation;

q) comments on the test results and methodology particularly noting departures from this method.

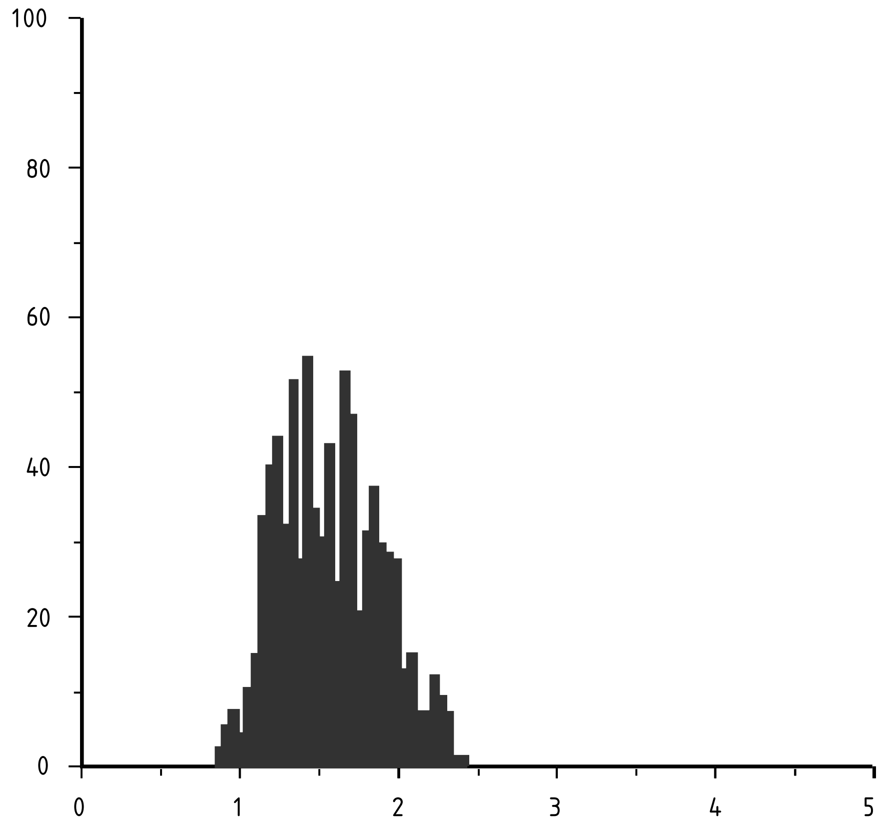
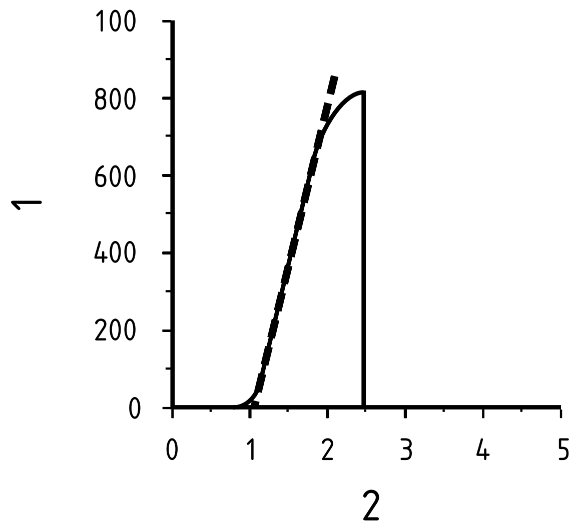


Figure 3 - AE hits versus strain during tensile tests of a coating showing onset of coating fracture at about 0,9% strain



Key

1 AE hits

2 Strain, %

Figure 4 - Analysis procedure for acoustic emission signals

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