SLOVENSKI STANDARD
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Sodobna tehnična keramika – Preskusne metode za keramične prevleke – 11. del: Določanje notranjih napetosti po formuli Stoney

Advanced technical ceramics - Methods of test for ceramic coatings - Part 11: Determination of internal stress by the Stoney formula

Hochleistungskeramik - Verfahren zur Prüfung keramischer Schichten - Teil 11: Bestimmung der inneren Spannung nach der Stoney-Gleichung

Céramiques techniques avancées - Méthodes d'essais pour revetements céramiques - Partie 11 :Détermination de la contrainte interne par la formule de Stoney

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Advanced technical ceramics - Methods of test for ceramic coatings - Part 11: Determination of internal stress by the Stoney formula

Céramiques techniques avancées - Méthodes d'essais pour revêtements céramiques - Partie 11 :Détermination de la contrainte interne par la formule de Stoney

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Foreword

This CEN Technical Specification (CEN/TS 1071-11:2005) 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 filometer*

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*

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*

Part 9: *Determination of fracture strain*

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

Part 11: *Measurement of internal stress by the Stoney formula*

Parts 5 to 6 are European prestandards.

Parts 7 to 11 are Technical Specifications.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this CEN 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.

1 Scope

This Technical Specification specifies a method for the determination of the internal stress in thin ceramic coatings by application of the Stoney formula to the results obtained from measurement of the radius of curvature of coated strips or discs.

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

3 Principle

Coating stress often plays a major role in the performance of coated tools and machine parts. Different techniques have been developed for the determination of coating stress. The technique considered in this document calculates the stress from measurement of the bowing of thin discs or strips of well characterised materials of known thickness that have been coated on one side only. It is assumed that the deformation is elastic, i.e. if the coating were to be removed, the substrate would return to its initial shape.

Provided that the coating is thin compared to the thickness of the substrate (coating thickness < 2 % of substrate thickness); that the curvature has a spherical form; and that the substrate was initially flat or of known curvature, then the stress in the coating can be calculated using the Stoney formula (see 6.6) without the need to know the elastic properties of the coating material.

The technique requires an accurate knowledge of the thickness of the coating, the thickness of the substrate, and the Young's modulus and Poisson's ratio of the substrate material.

NOTE 1 Coating thickness can be determined by techniques such as step height measurement (see EN 1071-1 [1]), crater grinding (see EN 1071-2 [2]) and cross sectioning (see CEN/TS 1071-10 [3]).

As ceramic coatings are normally deposited at elevated temperatures, the stress determined at any other temperature will be a combination of the intrinsic growth stress and stress introduced by virtue of the difference in thermal expansion between the coating and the substrate.

The internal stress σ_o in the coating is deduced from the measured radius of curvature R_{exp}, through the application of the Stoney formula [4]:

$$\sigma_o = -\frac{1}{6}\frac{E_s}{1-\nu_s}\frac{h_s^2}{h_f}\frac{1}{R_{exp}}$$

where h_f and h_s denote the thickness of the coating and substrate respectively, and where E_s and ν_s denote Young's modulus and Poisson's ratio of the substrate respectively.

NOTE 2 σ_o is the mean value of the local stress through the thickness of the coating ($h_f << h_s$):

$$\sigma_o = \frac{1}{h_f}\int_0^{h_f} \sigma_f(z)dz$$

where $\sigma_f(z)$ is the film stress as a function of position perpendicular to the plane of the substrate.

The radius of curvature $R_{\exp}$ is obtained from the profile of the sample.

4 Apparatus

The profile can be measured by means of an optical profilometer, a high magnification optical microscope (resolution in the order of 1 µm), equipped with an accurate (better than 5 µm resolution) position sensor along the focusing direction and a micrometer equipped translation stage, or other suitable technique. For a disc shaped sample with a polished surface, e.g. a circle cut from a polished silicon wafer, the radius of curvature can be obtained by treating it as a convex or concave mirror and measured using an optical bench or other suitable technique, e.g. by the use of Newton's rings. However, in all cases it is essential to ensure that the measurement technique used does not alter the profile of the sample.

Where a microscope with a translation stage is used for the measurement, care shall be taken to ensure that the stage is level with respect to the perpendicular to the optical axis. The simplest way to check this is to ensure that the surface of the translation stage remains in focus over a distance equivalent to the overall length of the sample, with the microscope at its highest magnification. For all measurement methods, care shall be taken to ensure that they are calibrated and traceable to national standards.

If a contact probe profilometer is to be used, care shall be taken to use the lowest load possible, commensurate with obtaining an accurate result, in order to avoid the contact force changing the profile of the sample.

EXAMPLE The deflection of a beam, supported at its extremities, by the application of a load in the central zone is:

$$\delta = \frac{FL^3}{48EI}$$

where L is the length of the beam, F the applied load, E is Young's modulus and $I = \frac{bt^3}{12}$ (b = width and t = thickness).

Thus, for an Al substrate for which E = 70 GPa, L = 100 mm, b = 10 mm and t = 0,5 mm, replacing these values in the formulae gives:

$$\delta = 4{,}8 \times 10^{-3} F \text{ (m)}$$

For a 0,75 mN force (see EN ISO 3274 [5]), the deflection will be 3,6 microns, i.e. an error of ~ 0,5 % for a total deflection of 1 mm. It should be noted that, with this beam geometry, a total deflection of 1 mm corresponds to a curvature radius of 1,7 m and for this substrate thickness such a deflection can be reached by a 1 micron film with a 2,45 GPa residual stress.

NOTE If measurements are to be made during the deposition process or in other cases where the sample is not accessible, e.g. whilst it is held in a furnace in order to investigate thermal stress relief, it is possible to use a strip sample that is clamped at one end. The change in bowing can then be determined by treating the sample as an optical lever and measuring the deflection of a known point by use of a laser and suitable scale. However, it should be appreciated that the use of a sample that is free to bend during the coating deposition will result in the calculated stress being different from that determined using a fully clamped sample as the deposition conditions, particularly temperature, will be different in the two cases. In addition, as the sample begins to bend it may be possible for some coating to be deposited on the back surface thus reducing the curvature that would otherwise be measured.

5 Preparation of test specimens

As the test method depends upon the determination of the curvature introduced into a substrate by the intrinsic stresses in a coating deposited thereon, the use of a test specimen manufactured from a well-characterised material is a prerequisite for the method.

Test specimens with a strip-shaped geometry are to be preferred, but specimens in the form of a disc can be used. The test specimen shall be manufactured from a material of known mechanical properties that will not be affected by any elevated temperature experienced during the coating process. It shall have a uniform thickness and shall be in a stress free state prior to the deposition of the coating.

NOTE 1 If necessary, test specimens should be annealed at a temperature above the coating temperature prior to coating deposition in order to remove stresses induced by the manufacturing process, e.g. from rolling, grinding or polishing.

Test specimens shall have a surface finish on the side to be coated that is commensurate with accurate measurement of the radius of curvature produced by the coating. Where the value of internal stress obtained in the test will be used for modelling with real components, care shall be taken to ensure that the surface texture of the test specimen is close to that of these real components. For all other test specimens the surface finish produced by careful grinding on 1 200 grit emery paper is a minimum requirement.

The dimensions of the sample shall be chosen such that the radius of curvature after coating, R_{exp}, is as low as possible to improve the accuracy of the measurement. However, care should be taken in order not to have plastic deformation of the substrate. This may require that initial testing be done to obtain an approximate value for the stress in order that the test specimen dimensions can be selected more accurately.

NOTE 2 The elastic/plastic characteristics of the substrate materials depend on the temperature. Thus, to avoid plastic deformation, if depositions are performed with substrate heating and/or coated samples are submitted to annealing at high temperatures, the estimations for the admissible radius values should be done with the σ_y/E (σ_y = yield stress, E = Young's modulus) ratio of the substrate material determined at those temperatures (see Annex A).

Where measurement of the curvature is made at a temperature different from that at which the deposition is made, the measured stress will be a combination of intrinsic growth stresses and those resulting from differential thermal expansion between substrate and coating. In such cases computation of the coating intrinsic stress requires knowledge of the values for the coefficient of thermal expansion of both substrate and coating.

NOTE 3 Where coating materials with anisotropic properties, e.g. those with hexagonal closed packed crystallography, are the subject of the test, it is necessary to determine any crystallographic preferred orientation in the coating resulting from the deposition process.

6 Procedure

6.1 Measuring range and initial profile

Before depositing the coating, it is necessary to determine the initial profile of the test specimen as it cannot, necessarily, be considered to be perfectly flat. This is most conveniently carried out using a suitable microscope (see Clause 4), as it is difficult to accurately measure large (> 20 m) radii of curvature using optical techniques.

Before measuring the initial profile, define a measuring range (length Δ), along the length a of the substrate (along a diameter for the disc).

NOTE 1 In order to avoid any edge effects, the measuring range should stop at a distance from the ends equal to at least 20 times the sample thickness.

Make some reference marks, e.g. Vickers indentations, on the substrate, so that the final profile can be measured at the same location and in the same direction as the initial one. This procedure is essential when the initial profile is somewhat different from a perfect circle.

NOTE 2 It is recommended that, where hardness indents are used as reference marks, indentations should be produced using a micro-hardness tester at a load of no greater than 1 kg.

For measuring the profile, the sample shall lie freely, without any external stress. Place the strip on two supports, so that it overhangs each support by a quarter of its length; proceed similarly for a disc.

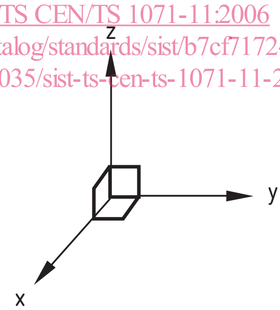
NOTE 3 In many cases supporting the sample on its ends will have negligible effect upon the curvature. However, the use of supports will avoid problems with stability of the sample if one or both ends of the sample are not perpendicular to the sides.

NOTE 4 Especially for thin samples, ensure that the weight of the sample does not change its shape: turning the strip (or disc) over on the supports should leave the profile unchanged, except for the sign.

In the case of optical microscopy, measure accurately for each profile the coordinates (x_i, z_i) of about ten points distributed uniformly along the predefined measuring range. The ordinate z_i corresponds to the vertical position of the microscope objective for which the point x_i of the surface of the sample is in the image plane. The focusing axis of the microscope shall be perpendicular to the surface to be coated, and especially to its longitudinal axis – see Figure 1.

The initial curvature can be disregarded in so far as it is very low (< 5 %) when compared with the final curvature.

After the initial profile is measured, the sample shall be handled in such a way as to avoid changes in internal stress or in irreversible deformation.



Key

Plane x – y: plane of lateral movement of the microscope stage and plane of test specimen surface to be coated

Direction x: longitudinal axis of the test specimen

Direction z: focusing axis of the microscope

Figure 1 — Relationship between plane of lateral movement of focusing stage, focusing axis and longitudinal axis of test specimen to be coated

6.2 Deposition of the coating

The test method relies upon the coating being deposited on one side only of the test specimen. The simplest way to achieve this is to clamp the test specimen at its ends and to use sacrificial pieces of the same material