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Fine ceramics (advanced ceramics, advanced technical ceramics) — Methods of test for ceramic coatings — Determination of internal stress in ceramic coatings by application of the Stoney formula

Céramiques fines (céramiques avancées, céramiques techniques avancées) — Méthodes d'essai des revêtements céramiques — Détermination de la contrainte interne des revêtements céramiques par application de la formule de Stoney

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Foreword

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This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Introduction

There is an increasing use of coatings to improve the functional performance of materials and components. This can be to protect against damage due to exposure to demanding environments including high stresses and aggressive chemical environments, but can also be to modify many other properties, e.g. thermal conductivity through thermal barrier coatings, friction through low friction coatings, such as diamond like carbon (DLC), and optical reflectivity through coatings with controlled optical properties.

Appropriate choice of coatings for particular applications depends on the mechanical and other functional requirements that arise. One factor that can be crucial in determining coating performance and lifetime is the residual stress that is generated by the deposition process and/or by thermal expansion mismatch between the coating and the substrate as the component is cooled from the processing temperature.

This document describes the application of a simple experimental technique using the Stoney formula to analyse the coating induced bending of coupons, of known mechanical properties, to determine the residual stress in the coating.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Methods of test for ceramic coatings — Determination of internal stress in ceramic coatings by application of the Stoney formula

1 Scope

This document 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

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 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-characterized 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 7.6) without the need to know the elastic properties of the coating material.

The technique does require accurate knowledge of the thickness of the coating, the thickness of the substrate, and 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 ISO 18452), crater grinding (see ISO 26423), and cross-sectioning (see EN 1071-10[4]).

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_0, in the coating is deduced from the measured radius of curvature, R_{exp}, through the application of the Stoney formula[6] as shown in Formula (1):

$$\sigma_0 = -\frac{1}{6}\frac{E_s}{1-\nu_s}\frac{h_s^2}{h_f}\frac{1}{R_{exp}} \tag{1}$$

where

h_f is the thickness of the coating;

h_s is the thickness of the substrate;

E_s is Young's modulus of the substrate;

ν_s is Poisson's ratio of the substrate.

NOTE 2 σ_0 is the mean value of the local stress through the thickness of the coating ($h_f << h_s$):

$$\sigma_0 = \frac{1}{h_f}\int_0^{h_f} \sigma_f(z)dz$$

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.

5 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 with a 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, care should be taken 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 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 should 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 is the applied load;

E is Young's modulus;

$$I = \frac{bt^3}{12}$$

where

b is the width;

t is the thickness.

Thus, for an Al substrate with E = 70 GPa, L = 100 mm, b = 10 mm, t = 0,5 mm, replacing these values in the formulae gives Formula (2):

$$\delta = 4{,}8 \times 10^{-3} F(\mathrm{m}) \quad (2)$$

For a 0,75 mN force (see ISO 3274), the deflection will be 3,6 µm, 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 µm 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. while 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, please note 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 can be possible for some coating to be deposited on the back surface, thus reducing the curvature that would otherwise be measured.

6 Preparation of test specimens

6.1 Material

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-characterized material is a prerequisite for the method.

6.2 Sample geometry

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.

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.

6.3 Sample surface finish

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 1200 grit emery paper is a minimum requirement.

6.4 Sample dimensions

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.

The elastic/plastic characteristics of the substrate material 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 $\frac{\sigma_y}{E}$ (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.

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

7 Procedure

7.1 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 6) 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).

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.

It is recommended that, where hardness indents are used as reference marks, indentations should be produced using a microhardness 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 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.

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, for each profile, measure the coordinates (x_i, z_i) of about 10 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.

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.

7.2 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 along the sides of the sample to ensure coating thickness uniformity across the sample. In order to ensure that the sample stays in contact with the support during temperature changes associated with the coating process, it is recommended that the sample support be manufactured from the same material as the sample. Alternatively, the sample can be clamped around its perimeter using a mask with a suitable cut-out. However, as the use of such a mask will leave uncoated regions along both sides, it will be necessary to make allowance for this in the final result, or the uncoated sides of the specimen can be removed to ensure that the measured curvature after coating is not influenced by the presence of uncoated regions, but care is needed to avoid introducing any stress during such operations.

Where the value of internal stress obtained from the test will be used for modelling with real components, care should be taken to ensure that the deposition process is as near as practical to that used with these real components. In particular, where a physical vapour deposition process is used, it is essential to adopt the same substrate orientation to the depositing flux, the same substrate bias and chamber pressure and, if possible, the same deposition rate. These precautions will help to ensure that the same growth morphology is obtained on the test specimen as on the real components. All relevant deposition parameters shall be monitored and recorded.

7.3 Coating thickness

In order to calculate the internal stress in the coating, it is necessary to know the coating thickness, h_f. Measure the coating thickness at a series of points on the test specimen, using an appropriate method. Where a destructive technique is used, e.g. crater grinding (see Reference [3]), this should be carried out after measuring the final profile.

7.4 Adjusting the sample geometry after deposition of the coating

Adjusting the sample geometry after deposition is considered undesirable and should be unnecessary provided the recommended procedures given in 7.2 are followed and that the length masked at each end by the clamps is small compared to the total length of the sample.

Any cutting of the specimen should be undertaken with the greatest of care in order to avoid introducing plastic deformation. Where necessary, cutting perpendicular to the coating plane should be realized carefully by slow cutting with a cooled diamond saw and with the specimen fully supported. Cutting parallel to the coating plane is not permitted.

7.5 Measuring the final profile

Measure the final profile of the specimen using an appropriate technique, such as that described in 7.1. Correct the measured value for any initial curvature of the test specimen.

NOTE 1 Where the initial and final profiles are determined using the method described in 7.1, this correction can be made by subtracting the initial values of z_i from the values obtained after coating, provided that both measurements are made at the same position on the sample.

Check that the profile $z_R(x)$ is close to a circle and determine the radius, R_{exp}, of the circle. The assumed conditions ($R >> \Delta$) usually allow the use of the simple method that consists of approximating the arc of the circle to a parabola, i.e. $z_R(x)=M_0+M_1x+M_2x^2$. The measured radius of curvature then is $R_{exp}=1/(2M_2)$.

NOTE 2 The profile is close to a circle if the radius of curvature is approximately constant along the profile. For verifying this, in the case where the profile is represented for instance by 10 points, i.e. (x_i, z_i; i = 1 to 10), compare the radii of curvature obtained on considering the points i = 1 to i = 5, then i = 3 to i = 8, and then i = 5 to i = 10. The various radii should be nearly equal (<5 % difference between largest and smallest).

NOTE 3 A difference of the curvatures measured along different directions in the plane of the coating (easy to measure in the case of a disc) is a sign of a strong geometrical nonlinearity.

7.6 Calculation of stress

7.6.1 $h_f / h_s < 0{,}02$

Calculate the coating stress, σ_0, using the simplified Stoney formula shown in Formula (3):

$$\sigma_0 = -\frac{1}{6}\frac{E_s}{1-\nu_s}\frac{h_s^2}{h_f}\frac{1}{R_{exp}} \tag{3}$$

As the presence of the coating will affect the value of E_s, determination of E_s should ideally be carried out on the coated substrate rather than being obtained from literature. Both E_s and E_f (see 7.6.2) can be measured using the technique described in DIN 50992-1[5] or in References [7] and [8].

7.6.2 $0{,}02 < h_f / h_s < 0{,}1$

Where the condition $h_f / h_s < 0{,}02$ is not satisfied, then provided $h_f / h_s < 0{,}1$, the following relation, shown in Formula (4), shall be used instead of the simple Stoney formula[9][10].

$$h_f\sigma_0 = -\frac{1}{6}\frac{E_s}{1-\nu_s}\frac{h_s^2}{R_{exp}}\left[1+\left(4\frac{E_f}{E_s}\frac{1-\nu_s}{1-\nu_f}-1\right)\frac{h_f}{h_s}\right] \tag{4}$$

where E_f and ν_f are Young's modulus and Poisson's ratio of the film, respectively.

NOTE The value of E_f and ν_f can be determined using surface acoustic waves[5] or the combined impact excitation and depth sensing indentation method described in References [7] and [8]. However, very accurate values for E_f and ν_f are not required, since the term between brackets is only corrective.

7.7 Number of repeat measurements

It is recommended that at least five separate measurements of R_{exp} be made along parallel lines spaced uniformly across the sample, for a rectangular sample, or on different diameters for a disc-shaped sample. If these different measurements result in different values of stress, this suggests coating non-uniformity across the specimen.

8 Limits to method

The method often requires specific specimens to be of specific size, thickness, substrate flatness, etc., for the curvature to be neither too low (far larger than the detection limit made possible by the experimental arrangement) nor too high (in order to avoid nonlinearity, especially of a geometrical nature, that results from high curvatures).

Only one side of the specimen should be coated.

NOTE If both sides are coated, the curvature is zero because of symmetry.

The Stoney formula implicitly results from certain simplifying assumptions, particularly in relation to geometry, that must be satisfied (Clause 5).

9 Test report

The test report shall include the following information:

a) the name and address of the testing establishment;

b) the date of the test;

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

d) the customer name and address;

e) a reference to this document, i.e. ISO 19674;

f) an authorizing signature;

g) any deviation from the method described, with appropriate validation demonstration, to be acceptable for the parties involved;

h) a description of the test material: type and material of specimen, type of coating, coating process and relevant deposition parameters;

i) the method of test specimen preparation and measurement, including any post coat sample modification;

j) the method of calculation of internal stress;

k) the internal stress values for at least five separate measurements of sample curvature;

l) the test results;

m) the comments about the test or the test results.

Annex A
(informative)

Determination of suitable dimensions for the test sample

Assuming that an order of magnitude value for the stress σ_0 is known, the thickness of the substrate, h_s, should be fixed so that the resulting radius of curvature, R_{exp}, can be measured under good conditions (usually around 1 m). In a beam of rectangular cross-section ($b \times t$; b is the width and t is the thickness of the beam) submitted to bending, the maximum absolute value of the deformation in the x direction, ε_{xx}, occurs in the surface of the beam and depends on the radius of curvature according to Formula (A.1):

$$\varepsilon_{xx} = \frac{t}{2R} \tag{A.1}$$

Thus, taking into account Hooke's law, $\varepsilon_{xx} = \frac{\sigma_y}{E}$ (σ_y = yield stress, E = Young's modulus), in order to avoid plastic deformation of the substrate, it is necessary, as shown in Formula (A.2), that:

$$\frac{\sigma_y}{E} > \frac{t}{2R} \tag{A.2}$$

EXAMPLE For a low strength steel substrate (σ_y = 220 MPa and E = 220 GPa) 1 mm thick, plastic deformation occurs for radii of curvature lower than 0,5 m.

NOTE In the case of coatings deposited with substrate heating and/or submitted to high temperature annealing, it is important to note that residual stresses exert their effect on a material with $\frac{\sigma_y}{E}$ ratios different from those determined at room temperature. The $\frac{\sigma_y}{E}$ ratio is not constant with temperature increase, with σ_y usually being observed to decrease more strongly than E with an increase in the testing temperature.