INTERNATIONAL STANDARD

ISO 19674

First edition
2017-09

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

Reference number
ISO 19674:2017(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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):

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

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.