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Thermal spraying — Determination of interfacial toughness of ceramic coatings by indentation

Projection thermique — Détermination de la ténacité interfaciale des revêtements céramiques par indentation

Reference number
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Foreword

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This document was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*.

Introduction

The interfacial toughness of thermal spray ceramic coatings is required for two reasons:

a) to understand quantitatively degradation of adhesion in service;

b) for life assessment of the coated components.

Adhesion strength of thermal spray coatings is usually measured in accordance with the tensile method specified by ISO 14916. However, there is a technical limitation in the application of ISO 14916. It requires preparation of a tensile adhesion test specimen using glue and, as such, coatings with higher adhesion strength than glue cannot be quantitatively evaluated. By virtue of its simplicity and practicality, an indentation method is a promising method to evaluate the adhesion of such coatings. In this method, a Vickers hardness tester is used. An interfacial toughness value is evaluated by measuring a length of the crack formed after pushing the indenter on the coating interface. The application of the method specified in this document can reduce uncertainty over the adhesion strength evaluation of coatings and makes it possible to evaluate the adhesion strength in a simpler way.

The results can be expressed either as a stress intensity factor, in $MN \cdot m^{-3/2}$, or as a fracture surface energy, in $J \cdot m^{-2}$.

"Interfacial toughness" can have several meanings.

a) Interfacial fracture toughness, K_{Ic}, in $MN \cdot m^{-3/2}$, is a material constant that shows how easily the coating may be peeled away from a substrate.

b) Energy release rate (or work done to fracture), G, is an alternative expression for interfacial fracture toughness, often obtained by converting K to G [i.e. $G = K^2(1 - \nu^2)/E$, where E is Young's modulus and ν is Poisson's ratio]. G has units of $J \cdot m^{-2}$.

For the purposes of developing the test method, the term indentation interfacial toughness, K_{IFC}, is defined separately from interfacial fracture toughness, K_{Ic}. The indentation interfacial toughness is a value obtained by using Vickers hardness tester, and is calculated from the total length of cracks induced along the interface by the indentation. Shorter crack lengths indicate that the coating has higher interfacial toughness or adhesion strength than other samples with longer cracks. There is no standard method to determine such interfacial toughness for thermal spray ceramic coatings, and different organizations or groups currently use their own evaluation procedures.

When measurements are carefully performed following the methods defined in this document, the evaluation of crack length, and thus interfacial toughness, will not require much effort and can describe the adhesion characteristics of the system (substrate chemistry, pre-preparation/roughness of substrate surface, coating chemistry and properties). This document recommends good practice to minimize uncertainty in the measurement procedures.

Thermal spraying — Determination of interfacial toughness of ceramic coatings by indentation

1 Scope

This document specifies a method for measuring the interfacial toughness of thermal spray ceramic coatings at room temperature based on an indentation method. The interfacial toughness is calculated from the mean length of cracks emanating from the corners of the impression induced by a Vickers hardness tester, and it is intended for use with ceramic coatings with a single layer or multilayers. The test procedures proposed in this document are intended for use in an ambient environment.

This document is recommended for thermal spray ceramic coatings such as thermal barrier coatings, wear resistant coatings and electrical insulating coatings.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

ISO 6507-1, *Metallic materials — Vickers hardness test — Part 1: Test method*

ISO 6507-2, *Metallic materials — Vickers hardness test — Part 2: Verification and calibration of testing machines*

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:

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

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

4 Symbols and units

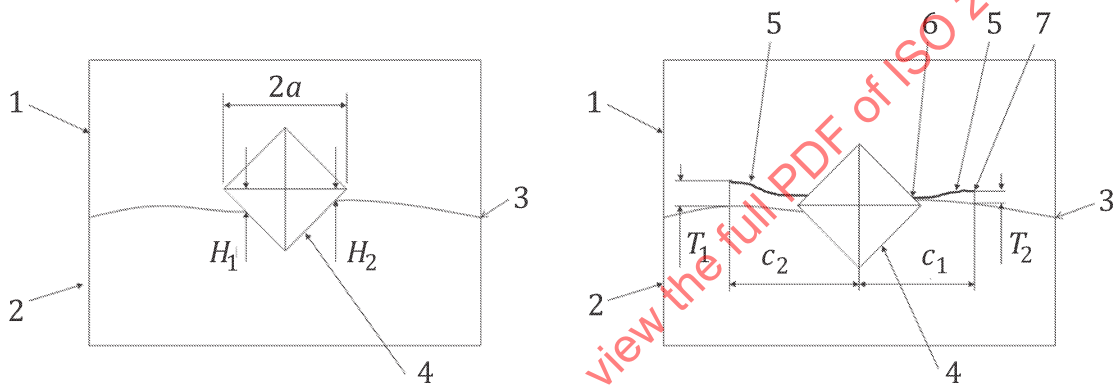
For the purposes of this document, the symbols and units given in Table 1 apply (see also Figures 1 and 2).

Table 1 — Symbols and designations

Symbol	Designation	Unit
a	diagonal half-length of impression parallel to interface	m
c	mean value of half crack length: $(c_1 + c_2)/2$	m
c_1, c_2	individual crack lengths at corner of impression (lengths from crack tip to centre of impression, parallel with the macroscopic interface)	m
E_1	Young's modulus of ceramic coating	$GN \cdot m^{-2}$
E_2	Young's modulus of substrate (or bond coat)	$GN \cdot m^{-2}$
F	test force (indentation load)	N
H	mean value of distance from centre of impression to the interface: $(H_1 + H_2)/2$	mm
H_1, H_2	individual distance from centre of impression to interface	mm

Table 1 *(continued)*

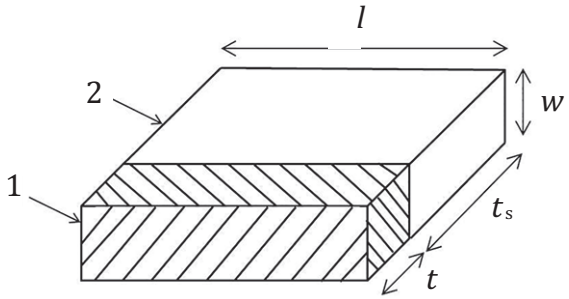
Symbol	Designation	Unit
K_{IFC}	indentation interfacial toughness	$MN \cdot m^{-3/2}$
l	sample length	mm
T	mean value of distance between crack tip and interface: $(T_1 + T_2)/2$	mm
T_1, T_2	individual distance between crack tip and interface	mm
t	ceramic coating thickness	mm
t_s	substrate (including bond coat) thickness	mm
w	sample width	mm
ε	bimaterial constant	-
η	constant value: 0,081	-
ν_1	Poisson's ratio of ceramic coating	
ν_2	Poisson's ratio of substrate (or bond coat)	-



Key

1 ceramic coating
2 substrate (or bond coat)
3 interface
4 impression
5 visible crack
6 crack root
7 crack tip

Figure 1 — Schematic diagram of indentation characteristics



Key

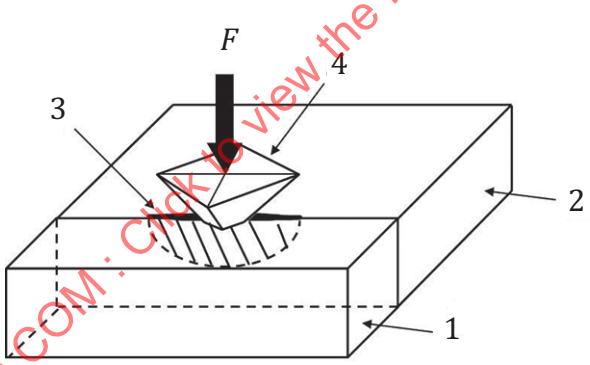
1 ceramic coating

2 substrate (with bond coat)

Figure 2 — Sample geometry

5 Principle

When a pyramidal Vickers diamond indenter is indented at the interface of the thermal spray coatings (see Figure 3), an interfacial crack is generated from a corner of the impression. The interfacial toughness is proportional to the indentation load divided by the square root of half the crack length.



Key

1 ceramic coating

2 substrate (with bond coat)

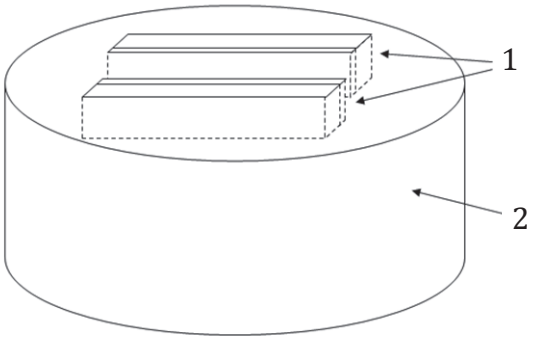
3 visible crack

4 Vickers diamond indenter

Figure 3 — Interfacial indentation test

6 Test pieces and sample preparation

The substrate surface should be flat. After thermal spraying, samples are cut from sprayed plate. The samples shall be embedded in resin (see Figure 4).



Key

1 coating samples

2 resin

Figure 4 — Test piece for indentation test

6.1 Thicknesses of coating and substrate

The substrate shall have sufficient thickness. It is recommended that the thickness of substrate (including bond coat), t_s, (see Figure 2) is at least 5 mm.

The minimum thickness of the ceramic coating, t, (see Figure 2) shall be at least 3 times the half-length of the impression, and a thickness of at least 5 times the half-length is recommended.

6.2 Sample size

The width of the cut samples, w, (see Figure 2) shall be sufficiently larger compared to the depth of the indentation. It is recommended that the width of the cut sample is at least 5 mm.

The length of the cut samples, l, (see Figure 2) shall be of a sufficient length to enable the indenter to be applied at adequate intervals. It is recommended that the length of the cut samples, l, is larger than 20 mm.

NOTE It has also been suggested[5] that the sample width, w, be at least larger than 10 times the crack length. A narrow sample might not give representative results because the stress state will be dependent on the amount of material supporting the indentation and its associated cracks.

6.3 Sample cutting

An appropriate cutting method and condition shall be selected in order to minimize cutting damage to the sample. Wheel cutting or electrospark discharge machines are convenient.

6.4 Mounting

The samples shall be embedded in resin with no gap between the resin and the samples.

6.5 Surface polishing

The test surface shall be ground back so that it removes any damaged layer induced by cutting and other specimen preparations. The surface shall then be polished to a mirror surface.

It is recommended that the surface is ground back by at least 0,25 mm depth.

7 Apparatus

7.1 Indentation testing machines, capable of applying predetermined indentation forces within the required scope.

It shall fulfil the requirements of ISO 6507-2.

7.2 Indenter.

The indenter for use with testing machines shall be a standard Vickers indenter in the shape of a right pyramid with a square base specified in ISO 6507-2.

7.3 Optical microscope, attached to or separated from the indentation testing machine, to measure the diagonal and crack dimensions.

It is recommended that the lengths of the impression's diagonal and crack be measured at a magnification satisfying the needs for a resolution of at least 10 μm. (The recommended magnification is 200 or more.)

8 Procedure and conditions of testing

8.1 Indentation

Indentation shall be carried out on a Vickers hardness testing machine in accordance with the method in ISO 6507-1 for the Vickers hardness test.

The test piece shall be placed on a rigid support to prevent the deformation of the mount due to the indentation loading, and the testing surface should be perpendicular to the indentation axis of the testing machine. Support surfaces shall be clean and free from foreign matter (scale, oil, dirt, etc.).

The indentation shall be made at the interface between the ceramic coating and substrate (or bond coat) to measure the interfacial toughness, and the diagonal of the impression shall be parallel to the interface.

The distance between the centres of any indentation shall be at least 5 times as large as the interfacial crack in order to prevent the influence of the adjacent interfacial crack. If two adjacent interfacial cracks differ in size, the spacing shall be based on the length of the larger crack. It is recommended that the distance between the centres of any indentation is at least 5 mm.

The distance between the centre of indentation and the edge of the test piece shall be at least 5 mm to prevent the edge effect of the test piece.

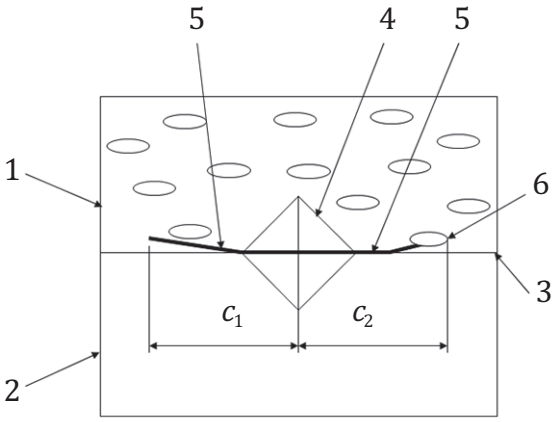
The duration of the test force shall be 10 s to 15 s.

8.2 Indentation and crack length measurement

Measure the lengths of the diagonal parallel to the interface. The readings shall be taken for the validation.

There are two methods for measuring the crack length. The results are independent of the method (see Reference [6]). Either of the following methods can be used.

NOTE If the interfacial crack stops in a pore, the length of the interfacial crack, *c*, is defined as the distance from the indentation centre to the edge of the pore (see Figure 5).



Key

1 ceramic coating
2 substrate (or bond coat)
3 interface
4 impression
5 visible crack
6 pore

Figure 5 — Definition of crack length in the case that the crack stops in a pore

a) Method A:

Measure the distance from crack tip to crack tip in the direction parallel to the interface. The half-length of the crack, c, is the half of this value (see Figure 1).

b) Method B:

Measure individual crack half-lengths, c_1 and c_2, from the impression centre line to crack tip for each of the two cracks even if the crack root does not coincide with the tip of the indentation diagonal.

If there are multiple cracks at an individual side of impression, the maximum length of crack shall be taken as the measured value.

9 Evaluation of indentation interfacial toughness

9.1 Analysis

The indentation interfacial toughness, K_{IFC}, shall be calculated from the diagonal half-length of an impression and the mean value of half crack length for an interfacial crack. The indentation interfacial toughness, K_{IFC}, is given by Formula (1)[7]:

$$K_{IFC} = \frac{\eta}{\sqrt{2\pi}} \frac{F}{a\sqrt{c}} \cosh\left(\pi\varepsilon\right) \tag{1}$$

where

$$\varepsilon = \frac{1}{2\pi} \ln\left(\frac{(3-\nu_1)E_2 + (1+\nu_2)E_1}{(3-\nu_2)E_1 + (1+\nu_1)E_2} \right) \tag{2}$$

The mean value of the K_{IFC} shall be obtained from more than 5 measurements and rounded to 2 significant figures.

It is recommended that the Young's modulus and Poisson's ratio for both the coating and substrate are measured according to ISO 17561, ISO 14577-4 or ISO 19477. If the Poisson's ratio is difficult to evaluate, a value of 0,3 may be used as the value of Poisson's ratio in this calculation. In this case, it should be reported that an assumed value of 0,3 was used for Poisson's ratio.

9.2 Test validity

No spalling and no vertical cracking shall occur at measurement points. When this document is applied to the multilayer coatings, no interfacial cracking shall occur at another interface; for instance, in the case of thermal barrier coatings, no interfacial cracking shall occur at the interface between the metal bond coat and the substrate (see Figure 6).

The mean value of half crack lengths for an interfacial crack, c, shall be at least 3 times the diagonal half-length of an impression. If the mean value of half crack lengths for an interfacial crack does not meet this requirement, the test should be repeated with a larger test force in order to generate an interfacial crack with enough length for the evaluation.

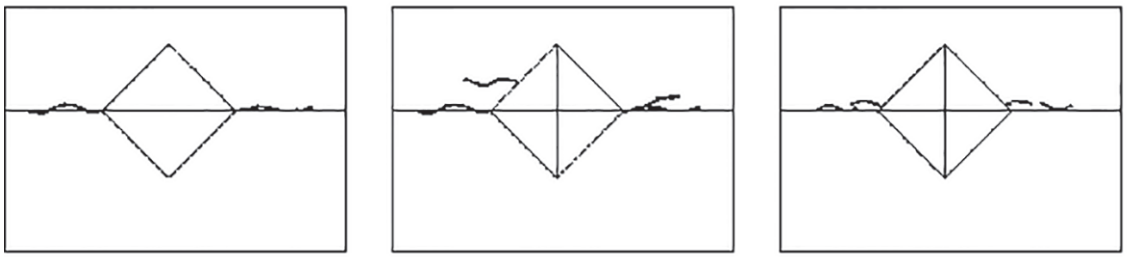
The distance from the centre of the impression to the interface, H, shall be less than the diagonal half-length for an impression.

The following conditions are recommended:

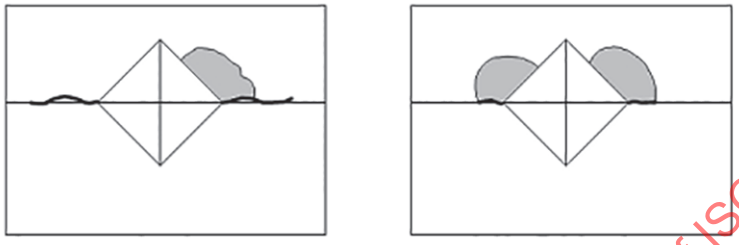
a) the distance between the centre of the impression and the interface, H, is less than 15 % of the diagonal half-length for an impression, $H < 0{,}15a$;

b) the difference between the right and left half-lengths of an interfacial crack is less than half of the mean value of half crack lengths for an interfacial crack, $|c_1 - c_2| < c/2$;

c) the individual distance between the crack tip and the interface, T_1, T_2, is less than the half-length of the impression, $T_1 < a/2$ and $T_2 < a/2$.



a) Acceptable impressions



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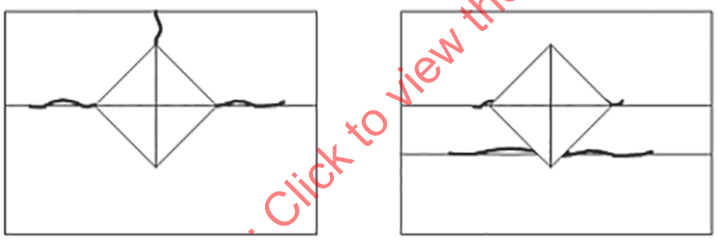

2 3

b) Unacceptable impressions

Key

1 spalling
2 vertical cracking
3 cracking at another interface in multilayer coating system

Figure 6 — Guidelines for the acceptability of impressions

10 Test report

10.1 Items to be reported

The report shall contain the following items.