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Thermal spraying — Classification method of adhesive strength by indentation

Projection thermique — Méthode de classification de la résistance adhésive par indentation

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Foreword

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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).

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For an explanation on 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.

The committee responsible for this document is ISO/TC 107, *Metallic and other inorganic coatings*.

Introduction

Adhesion of coatings specified here is required for three reasons:

— for selection of coating material and spray method;

— for quality assurance of thermal spraying;

— for product design and performance assessment of coatings.

This International Standard provides the classification method of adhesive strength for thermal spray coatings by using Vickers hardness testing machine generally and widely used. In the present test method, a pyramidal Vickers diamond indenter is indented with test force at the interface of coatings. When the test force exceeds the threshold value corresponding to the adhesive strength, an interfacial crack is generated. Accordingly, it is possible to evaluate the adhesive strength of coatings by the test force applied as discrete values when using conventional Vickers testing machine. This International Standard classifies the adhesive strength, called adhesion index, evaluated from the maximum indentation force without visible cracking and can be also applied to evaluate coatings with a high adhesive, such as cermet coatings. The present test method can be applied to sprayed coatings if the crack initiated by the indentation can be observed by using optical microscope.

The maximum indentation force does not therefore require much more effort in the experimental procedure and can describe the property of system combination (substrate chemistry, pre-preparation/roughness of substrate surface, coating chemistry and spraying related coating properties), provided the measurements are obtained carefully in line with the methods proposed in this International Standard. This International Standard recommends good practice to minimize levels of uncertainty in the measurement process. The procedure has been validated through underpinning technical work within the round robin test programme conducted by the Subcommittee on Standard Development, Japan Thermal Spray Society.

Thermal spraying — Classification method of adhesive strength by indentation

1 Scope

This International Standard specifies the classification method of adhesive strength for thermal spray coatings at room temperature by using Vickers hardness testing machine. This International Standard classifies the adhesive strength, called adhesion index, evaluated from the maximum indentation force without visible cracking, and it is applicable for relative dense metal coatings, ceramic coatings and cermet coatings. This International Standard can be used within comparison within each coating system.

The indentation method is not recommended for very thin and very porous coatings and also for the coating system with lower cohesion strength than adhesion strength. It is recommended that this test method can be applied for a coating of which porosity is less than approximately 15 %.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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*

ISO 14917, *Thermal spraying — Terminology, classification*

ISO 14923, *Thermal spraying — Characterization and testing of thermally sprayed coatings*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 14917, ISO 14923 and the following apply.

3.1
critical indentation force
maximum indentation force without visible cracking between substrate and coating

3.2
adhesion index
AI
index of adhesive strength of coatings defined by the critical indentation force

4 Symbols and units

For the purposes of this document, the following symbols and units apply (see Figures 1 and 2).

Table 1 — Symbols and designations

Symbol	Designation	Unit
a	Impression-diagonal half-length parallel to interface	mm
e	Visible crack length mean value	mm
e_1, e_2	Individual crack lengths at corner of an impression (lengths from crack tip to edge of impression diagonal, parallel with the macroscopic interface)	mm
F	Test force (indentation force)	N
F_{eq}	Equivalent indentation force when using the special conical indenter	N
H	Mean value in impression position	mm
H_1, H_2	Individual impression position	mm
l	Sample length	mm
T	Mean value in crack tip position	mm
T_1, T_2	Individual crack tip position	mm
t	Coating thickness	mm
t_s	Substrate thickness	mm
w	Sample width	mm
CF	Visible cracking frequency	%
N_c	Number of indentations with visible cracking	—
N_t	Total number of indentations	—

Key
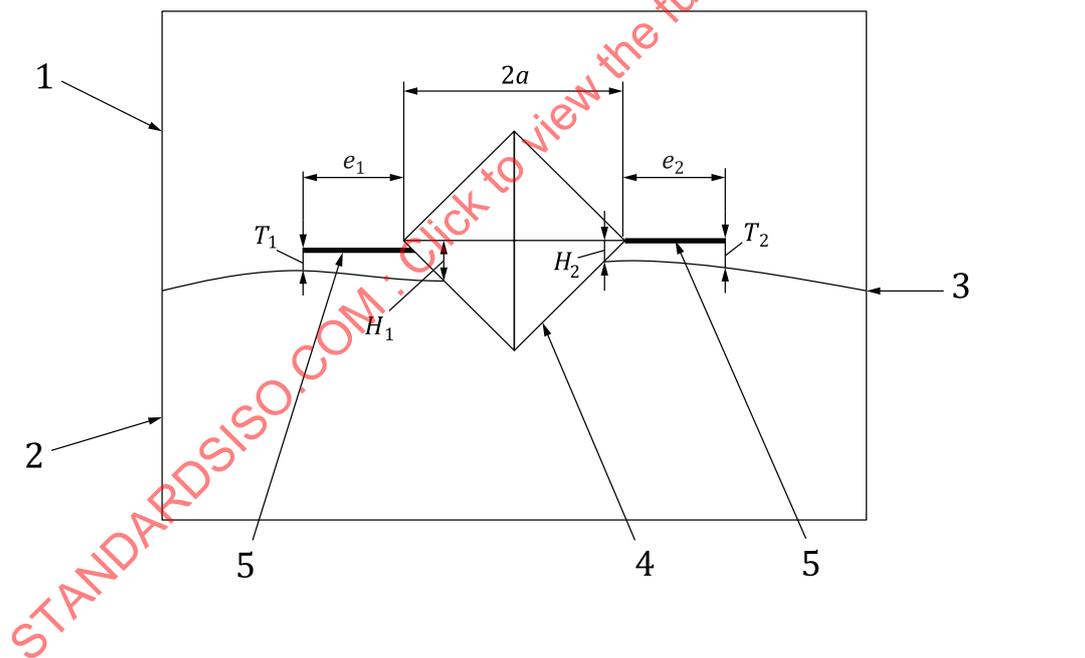
1   coating

2   substrate (or bond coat)

3   interface

4   impression

5   visible crack

**Figure 1 — Schematic diagram of indentation characteristics**

Key
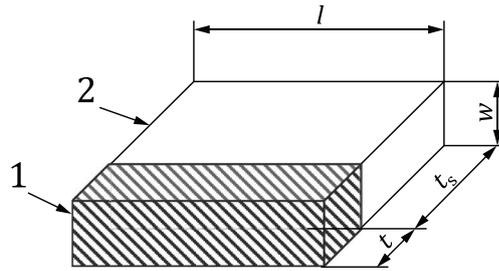
1   coating

2   substrate (with bond coat)

Figure 2 — Sample geometry

5 Principle

When a pyramidal Vickers diamond indenter is indented with the indentation force at the interface (see Figure 3), an interfacial crack opening force that acts perpendicularly to the interface is generated. This crack opening force is proportional to the test force. When the crack opening force exceeds, the adhesive strength of the test piece, an interfacial crack is generated. Accordingly, it is possible to classify the adhesive strength of the test piece by the indentation force applied using the Vickers indenter.


Key
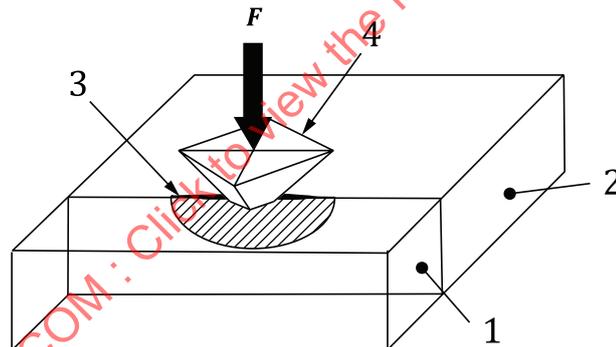
1   coating

2   substrate (with bond coat)

3   visible crack

4   Vickers diamond indenter

Figure 3 — Interfacial indentation test

6 Test pieces and sample preparation

The surface of the substrate to be coated by spraying shall be flat. After thermal spraying, samples are cut from the coated plate. Samples shall be coated of resin so as to obtain the specimen to be polished before proceeding to the test (see Figure 4).

In the samples, the thermal spray coating shall be adhered to the base material (or bond coating) and any cracking is not acceptable in the evaluation section before the test.
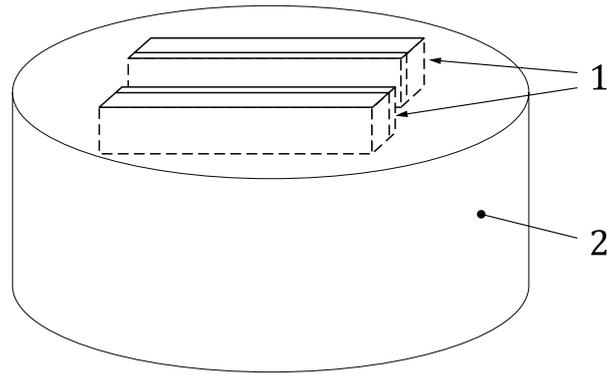
Key

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 in order to eliminate the effect of that in the experimental results. It is recommended that the thickness of substrate, t_s, (see Figure 2) is at least 5 mm.

The minimum thickness of the thermal spray coating, t, (see Figure 2) shall be at least twice the half-length of the impression in the case of high stiffness coatings, such as WC-12Co cermet coating. It is recommended that the thickness of coating is at least five times the half-length of the impression in the case of low stiffness coatings, such as thermal barrier coating which has very low elastic modulus.

6.2   Sample size

The width of the cut samples, w, (see Figure 2) shall be sufficiently greater than 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 longer than 20 mm.

6.3   Sample cutting

The appropriate cutting method and condition shall be selected in order to prevent the cutting to damage the sample. Wheel cutting or electro spark discharge machines are convenient to use for the cutting.

6.4   Mounting

The samples shall be embedded in resin without the gap between the resin and the samples. It is recommended that the sample is embedded in resin after it has been bonded to a rigid supporting jig when higher testing force is applied.

6.5   Surface preparation

The test surfaces shall be polished. The polished sample surface finish should be adequate enough to enable determination of the lengths of the impression diagonal and the crack. It is recommended that the test surface is buffed with the alumina or diamond powder to mirror surface as the final polishing.

The test shall be carried out on a surface which is free from the damaged layer induced by cutting and other specimen preparations.

7 Apparatus

7.1 Indentation testing machines

The indentation testing machine shall have the capability of applying predetermined indentation forces within the required scope and 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.

When no interfacial visible crack is generated using a standard Vickers indenter due to the high adhesive strength of coatings, a special conical diamond indenter with a point angle of 70° and a point radius of 0,05 mm can be used (see Figure 5).

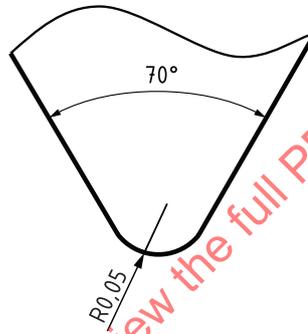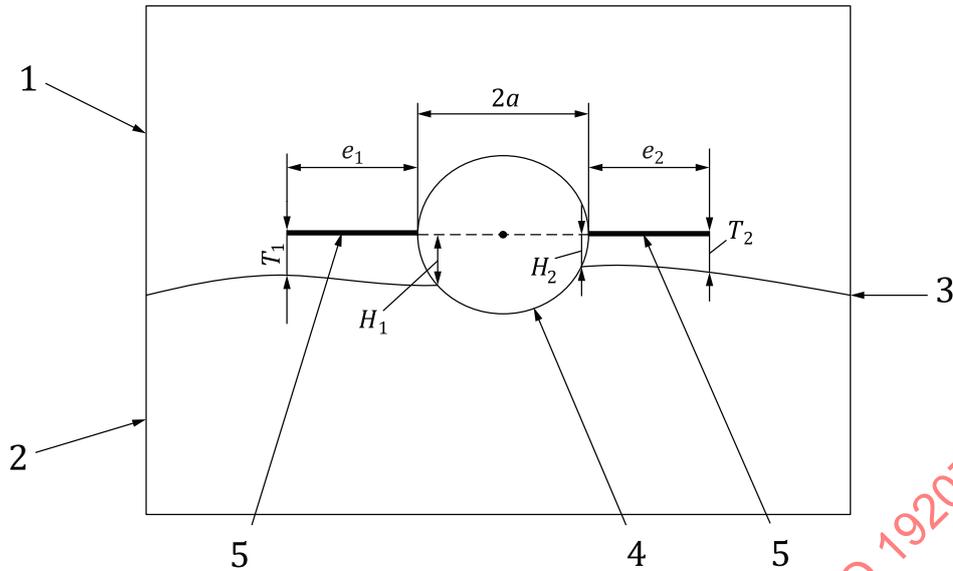When the special conical indenter is used, the dimensions are defined as shown in Figure 6.

Figure 5 — Shape of special conical indenter

Key

1   coating
2   substrate
3   interface
4   impression
5   visible crack

Figure 6 — Schematic diagram of indentation characteristics for special conical indenter

7.3   Impression and crack measurements

The diagonal and crack dimensions can be measured using an optical microscope attached or separated with the indentation testing machine, but it shall have been calibrated against a standard.

It is recommended that the lengths of impression diagonal and crack can be measured at a magnification satisfying the resolution of at least 10 μm. (The recommended magnification is 200.)

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 shall be carried out at ambient temperatures in a normal laboratory-air environment, not in corrosive environments, such as strong acids or seawater.

The test piece shall be placed on a rigid support and the testing surface should be perpendicular to the indentation axis of the testing machine. The support surfaces shall be clean and free from foreign matter (scale, oil, dirt and etc.). It is important that the test piece lies firmly on the support so that displacement cannot occur during the test.

The impression shall be applied at the interface by the indentation and the diagonal shall be parallel to the interface. If the centre of impression is misaligned with the interface due to the rigidity difference of coating and substrate (or bond coat), the deviation shall be estimated beforehand and taken into account as offset of the indentation position.

The distance between the centre of any impression and the edge of the sample shall be at least 3 mm.

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

8.2   Impression and crack length measurement

8.2.1   General

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

a)   if the distance from the centre of the impression to the tip of the interfacial crack is shorter than the half-length of the impression (for example, crack of right side in Figure 7), the crack length is defined as zero;

b)   if there are multiple cracks at an individual side of impression, the crack length shall be defined by the maximum length among those cracks;

c)   if the interfacial crack stops in a pore, the length of the interfacial crack is defined as the distance from the impression corner to the far edge of the pore (see Figure 8).

There are two methods for measuring the crack length. The results are independent of the method (see Reference [1]). Either of the following methods can be used.

8.2.2   Method A

Measure the distance from a crack tip to another crack tip in the direction parallel to the interface. The crack length, e, is the half of the value which is this value minus the impression diagonal length, $2a$ (see Figure 1).

NOTE   If the magnification is too high, this method is impractical because the crack tip to crack tip distance is usually too large to include in one image.

8.2.3   Method B

Measure individual crack lengths, e_1 and e_2, from the impression corner to the crack tip on both sides of the impression even if the crack root does not coincide with the tip of the impression diagonal. The crack length, e, is the average value of those.

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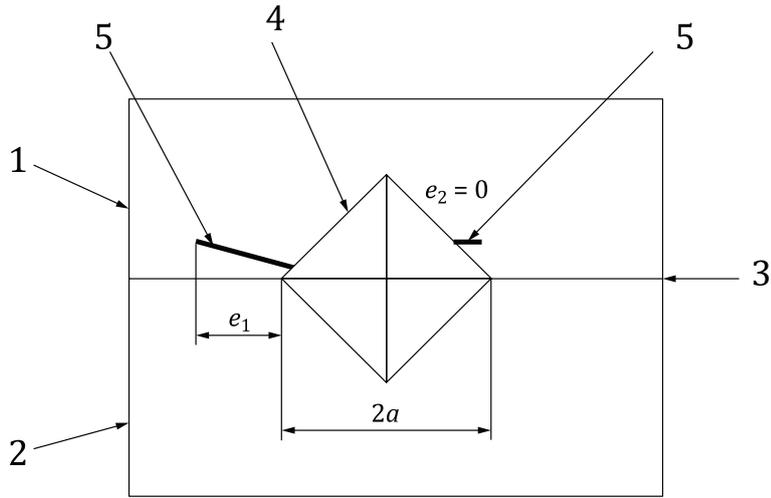
1 coating
2 substrate (or bond coat)
3 interface
4 impression
5 visible crack

Figure 7 — Example of crack length measurement

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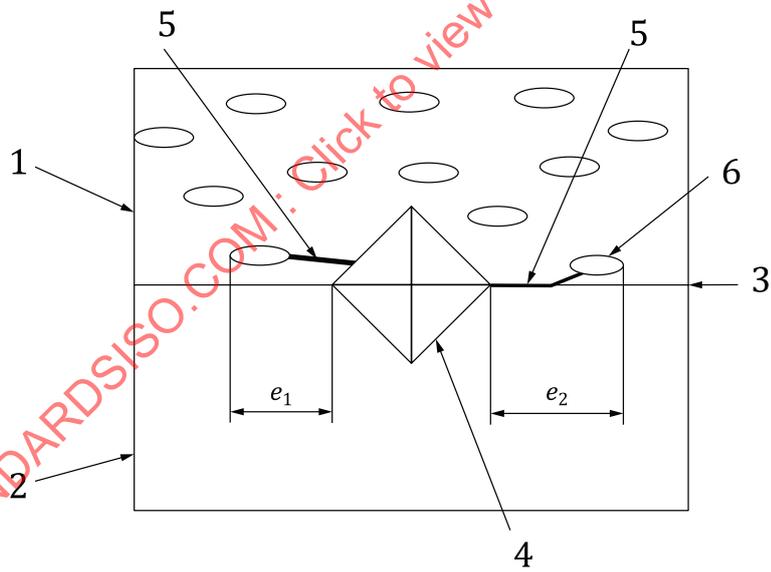
1 coating
2 substrate (or bond coat)
3 interface
4 impression
5 visible crack
6 pore

Figure 8 — Definition of crack length in the case that the crack stops in a pore

9 Evaluation of adhesion index

9.1 Criterions of interfacial crack initiation and critical indentation force

If the mean crack length, e, exceeds 50 μm, it should be defined as an interfacial crack initiation.

At each indentation force, valid measurements shall be conducted at no fewer than 10 points.

The maximum value of indentation forces which induces the interfacial crack initiation with the visible cracking frequency, CF less than 50 % in the valid measurement points is defined as the critical indentation force.

The visible cracking frequency, CF, is obtained by Formula (1):

$$CF = N_c / N_t \tag{1}$$

where

N_c is the number of indentations with visible cracking;

N_t is the total number of indentations.

At each indentation force, the number of all measurements and the number of measurements at which interfacial cracks are generated shall be reported.

9.2 Classification of adhesive strength and notation of adhesion index

The adhesive strength is classified by the critical indentation force defined in 9.1.

The following is an example of the designation of adhesion index.

EXAMPLE

AI 30

where

AI is the symbol of adhesion index;

30 is the approximate kilogram-force equivalent value of the critical indentation force, where (30 kgf = 294,2 N).

If using the special conical indenter in 7.2, the indentation force, F, shall be converted to the equivalent value used for the Vickers indenter, F_{eq}, by following Formula (2):

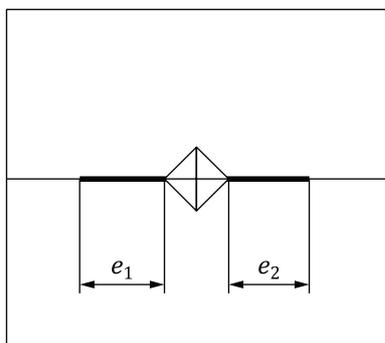$$F_{eq} = 3,2F \tag{2}$$
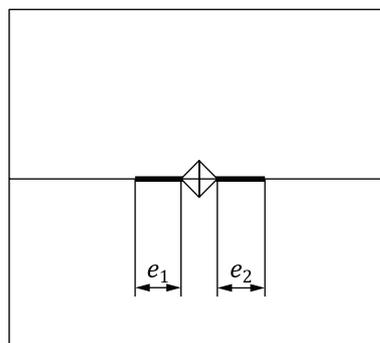
where

F_{eq} is the equivalent indentation force;

N_t is the indentation force.

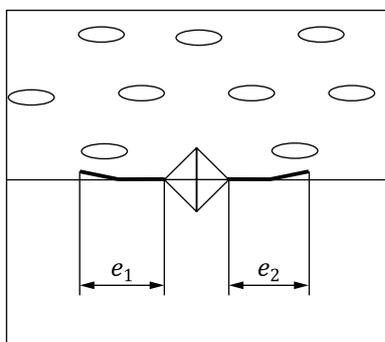Figure 9 is provided for typical examples of classification.

CF = 100 % at test force F = 29,42 N

→ Classified as "AI02"

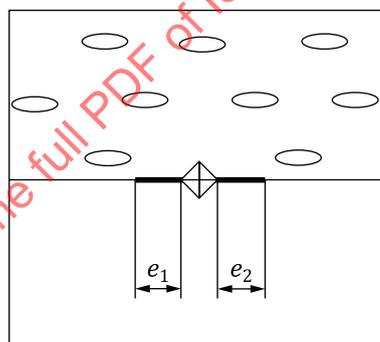**a) Typical case of metal coating**

CF = 28,6 % at test force F = 19,61 N

CF = 71,4 % at test force F = 49,03 N

→ Classified as "AI03"

b) Typical case of porous ceramic coating

CF = 14,3% at test force F = 29,42 N
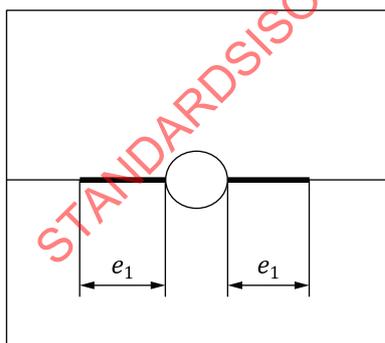
CF = 57,1% at test force F = 196,1 N
[Equivalent test force F_{eq} = 627,6 N]

→ Classified as "AI50"
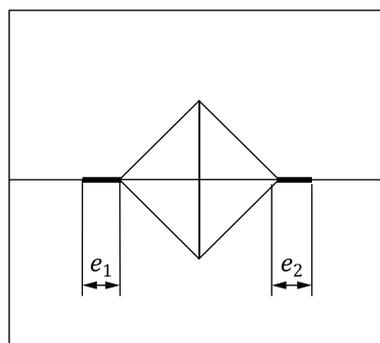
CF = 0 % at test force F = 490,3 N

c) Typical case of dense cermet coating

Figure 9 — Examples of adhesion index classification