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Fine ceramics (advanced ceramics, advanced technical ceramics) — Rockwell indentation test for evaluation of adhesion of ceramic coatings

Céramiques techniques — Évaluation de l'adhérence des revêtements céramiques par l'essai de pénétration Rockwell

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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 document 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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This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 184, *Advanced technical ceramics,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 26443:2008), of which it constitutes a minor revision. The changes are as follows:

— table of contents was added;

— normative reference titles were updated;

— units for loads in kgf were deleted.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Rockwell indentation test for evaluation of adhesion of ceramic coatings

1 Scope

This document specifies a method for the qualitative evaluation of the adhesion of ceramic coatings up to 20 µm thick by indentation with a Rockwell diamond indenter. The formation of cracks after indentation can also reveal cohesive failure. The indentations are made with a Rockwell hardness test instrument.

The method described in this document can also be suitable for evaluating the adhesion of metallic coatings.

The test is not suitable for elastic coatings on hard substrates.

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 6508-1, *Metallic materials — Rockwell hardness test — Part 1: Test method*

ISO 6508-2, *Metallic materials — Rockwell hardness test — Part 2: Verification and calibration of testing machines and indenters*

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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

4 Principle

An indentation is made into the coated surface of the specimen to be tested, whereby the coating near the indent can be damaged. The indentation and surrounding area are examined for cracks and/or flaking with the aid of an optical microscope.

5 Apparatus

The indentations shall be made in accordance with ISO 6508-1, following the procedure for a Rockwell hardness indentation.

The Rockwell hardness testing machine shall conform with the requirements of ISO 6508-2.

The contour of the diamond indenter shall be checked regularly by optical means (magnifying glass, optical microscope, stereomicroscope or projection screen). This check shall be made for at least four different axial sections. The indenter shall be replaced if this examination reveals any damage to the

indenter (e.g. chipping). A magnification of at least ×200 is recommended to detect ring cracks or microwear.

Although a research project to evaluate the effect of indentation parameters showed no major influence of load rate or holding time on the results (see Reference [1]), they should preferably be kept constant for reasons of repeatability. To conform with ISO 6508-1, it is necessary to keep the loading time between 1 s and 8 s and the hold time at (4 ± 2) s. Neither loading time nor holding time need to be recorded.

6 Sampling and preparation of test specimens

Select a representative test specimen from the coating to be tested. Clean the specimen so that it is free from dust and other particles, and also from oil or other surface films.

7 Procedure

The indentation shall be made in a direction perpendicular to the specimen surface. Therefore, specimens shall be prepared plane parallel and/or levelled before indentation.

Depending on the coating/substrate combination, a suitable load range shall be selected.

The following rules shall apply:

— for metallic substrates harder than 54 HRC, a load of 1 471,5 N shall be used (Rockwell C scale);

— for metallic substrates softer than 54 HRC and for medium case-hardened steel substrates, a load of 981 N shall be used (Rockwell D scale);

— for all other substrates, e.g. shallow case-hardened steel, thin substrates, cemented carbides, solid ceramics and cermets, a load of 588,6 N shall be used (Rockwell A scale).

Using an optical microscope (magnification ×100), relate the indentation to the classification given in Table 1. A pictorial representation and sample photographs of these classes can be found in Annex A.

Table 1 — Classification of test results

Class	Observation
Class 0	No cracking and no adhesive delamination (see Figure A.1)
Class 1	Cracking without adhesive delamination of the coating (see Figure A.2)
Class 2	Partial adhesive delamination, with or without cracking (see Figure A.3)
Class 3	Complete adhesive delamination (see Figure A.4)

Class 0 reveals acceptable adhesion. However, the absence of any visual failure can be due to the test not being suitable for the substrate/coating system under investigation. Class 1 shows no adhesive delamination; adhesion is acceptable. In the cases of class 2 and class 3, adhesion is unacceptable.

The test may also reveal cohesive failure of the coating, e.g. cracking. The observation of cracks can be facilitated by using optical contrasting techniques, e.g. Nomarski interference contrast microscopy.

Delamination can be due to adhesive as well as cohesive failure of the coating.

— Adhesive delamination is defined as a removal of the coating, whereby the underlying substrate can be clearly seen, or a removal of one or more sublayers in a multilayer coating, whereby the substrate or an underlying sublayer can be clearly distinguished.

— Cohesive delamination is defined as a partial removal of the coating, whereby the underlying substrate stays covered by the coating, or a removal of one or more sublayers in a multilayer coating, whereby the substrate and none of the underlying sublayers can be clearly distinguished.

Complete delamination is defined as an uninterrupted removal of the coating along the circumference of the indent.

— When a class 2 failure is observed, an estimate of the percentage of delamination in relation to the surface area of the indent shall be given. This estimate shall be based on the sum of the calculated areas of each individual delamination, determined from its dimensions. A micrograph of a typical class 2 failure can be found in Annex A.

— When a class 3 failure is observed, the size of the adhesive delamination shall be described by the ratio (r/a) of the radius of adhesive delamination r to the radius of the indent a.

For a class 3 failure, the radius of adhesive delamination is defined as the maximum radius of the delamination related to the centre of the indent, excluding any needle-like delaminations away from the indent.

lt is recommended that at least three measurements be made at representative locations.

8 Limits

Results shall only be compared when a similar substrate/coating combination and coating thickness are used.

When comparing results, class designations shall be linked to the load used. Only indents made at the same load shall be compared.

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, a unique identification of the test report and of each page, the name and address of the customer and the signature of the responsible individual(s);

c) a reference to this document, i.e. ISO 26443:2023;

d) the type of test equipment used, the manufacturer and the date of the last calibration;

e) a description of the test material: type of substrate, type of coating and date of receipt;

f) the method of test (i.e. the load) used, and details of sampling and specimen preparation;

g) the results of at least three tests for the load used, including descriptions for any class 2 and class 3 failures;

h) any comments about the test or the test results (e.g. the observation of cohesive failure).

Annex A
(informative)

Pictorial representations and sample photographs of the classes defined in Table 1

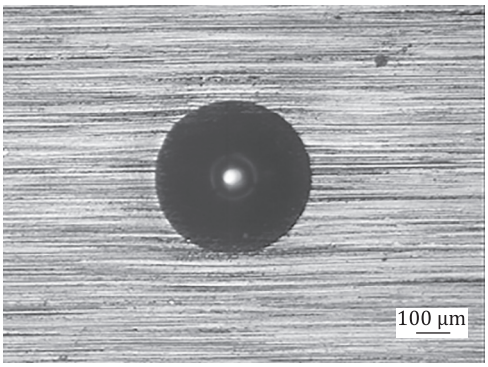
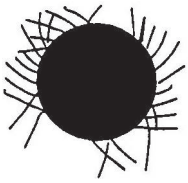


Figure A.1 — Class 0



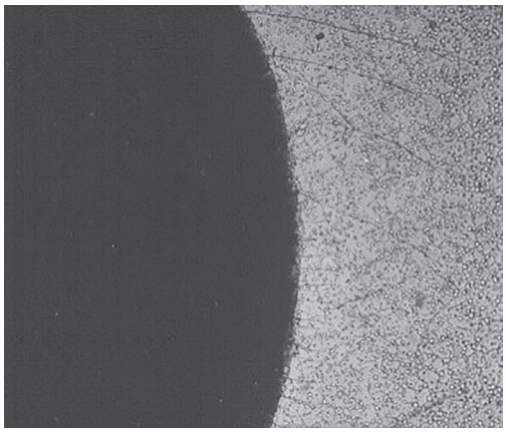
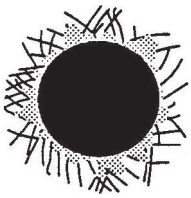


Figure A.2 — Class 1



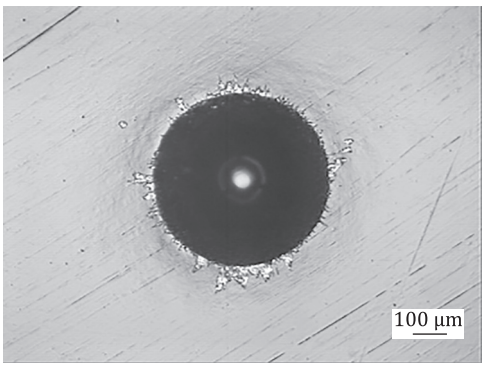
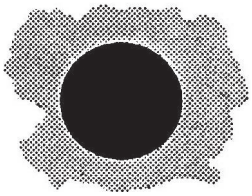


Figure A.3 — Class 2



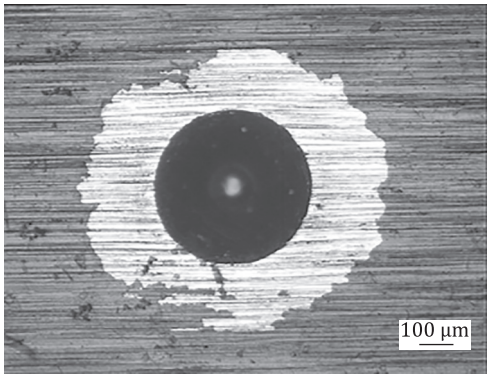


Figure A.4 — Class 3

Bibliography

[1] ISENORM research project 1998-2000, supported by the DWTC (Belgium), partners VITO, WTCM, Free University of Brussels (ULB)

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