
**Corrosion of metals and alloys —
Method for metallographic examination
of samples after exposure to high-
temperature corrosive environments**

*Corrosion des métaux et alliages — Méthode d'étude
métallographique d'échantillons soumis à des environnements
corrosifs à haute température*

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

ISO 26146 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

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Corrosion of metals and alloys — Method for metallographic examination of samples after exposure to high-temperature corrosive environments

1 Scope

This International Standard covers examination of samples that have previously been exposed to corrosive environments at high temperatures.

This International Standard covers the classification, identification, and thickness measurement of any corrosion layer forming during the exposure.

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 3611, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

ISO 13385-1, *Geometrical product specifications (GPS) — Dimensional measuring equipment — Part 1: Callipers; Design and metrological characteristics*

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3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

NOTE [Figure 1](#) provides a diagrammatic representation of appropriate parameters.

3.1

original metal surface

surface of the metallic sample before coating or exposure to the corrosive environment

3.2

original coating surface

surface of the coating after final processing treatment but before exposure to the corrosive environment

3.3

interdiffusion zone

region around the original interface between coating and substrate that, during exposure, has changed composition through diffusion processes between the coating and the substrate

3.4

deposit

chemically active liquid or solid species that are placed in contact with or are deposited on the test piece before and/or during exposure

EXAMPLE Salts, fly ashes, chars, and molten metals.

3.5

deposit thickness

distance, normal to the test piece surface, between the outer extent of corrosion product and the surface of the deposit layer

3.6

coating

layer of material applied to a metal surface to provide protection

3.7

coating thickness

distance, normal to the test piece surface, between the original metal surface and the surface of the coating

3.8

outward growing corrosion scale

corrosion scale that grows outwards from the original metal or coating surface

3.9

inward growing corrosion scale

corrosion scale that grows inwards from the original metal or coating surface

3.10

external scale

total of outward and inward growing continuous corrosion scales

3.11

internal corrosion

corrosion products that form beneath any external scale

Note 1 to entry: These usually appear as discrete particles.

3.12

grain boundary corrosion

corrosion product that grows along metal grain boundaries as a particular form of internal corrosion

3.13

de-alloyed zone

zone beneath the corrosion scale(s) that exhibits a decrease in the concentration of scale-forming alloy elements

Note 1 to entry: This may be manifested as the dissolution of precipitates originally present in the microstructure.

3.14

metal loss

distance between the original test piece surface and the boundary with unaffected alloy

3.15

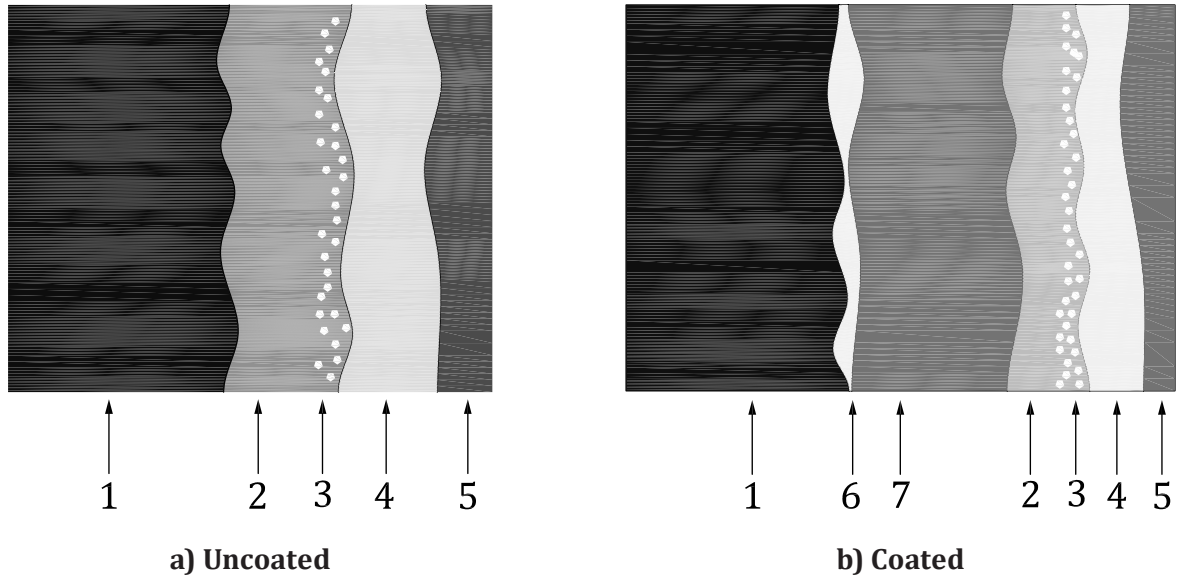
remaining sound metal

section of metal that has not been affected by corrosion

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**Key**

- 1 substrate
- 2 de-alloyed zone
- 3 internal corrosion
- 4 external scale
- 5 deposit
- 6 interdiffusion zone
- 7 coating

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Figure 1 — Identification of individual layers to be measured in exposed test pieces

4 Symbols and abbreviated terms

4.1 Symbols

- x thickness of individual layer
- t metal thickness

4.2 Abbreviated terms used as subscripts

The identification of individual layers is through the use of the following subscripts:

0	initial
og	outward growing part of the external corrosion scale
ig	inward growing part of the external corrosion scale
tot	total thickness of external scale ($x_{tot} = x_{og} + x_{ig}$)
in	internal corrosion
gb	grain boundary corrosion
d	deposit
c	coating
da	de-alloyed
ml	metal loss compared to original dimensions
rm	remaining sound metal

5 Requirements

The minimum requirement for measurement accuracy is to achieve an uncertainty at the 95 % confidence limit of $\pm 5 \mu\text{m}$ or 5 % of the measured material loss, whichever is the lesser for all errors, i.e. calibration, misalignment, both in the vertical and horizontal directions, and measurement.

Measurements shall be carried out under an optical microscope with an X-Y moving stage or with graticule and cross wires. The precision of the measurement system shall be to a precision of $\pm 1 \mu\text{m}$.

The measurement system shall be fully calibrated to include orthogonality, traceable to certified length standards at intervals no greater than 12 months. The system shall be checked against secondary standards at the beginning and the end of each series of measurements.

6 Test method

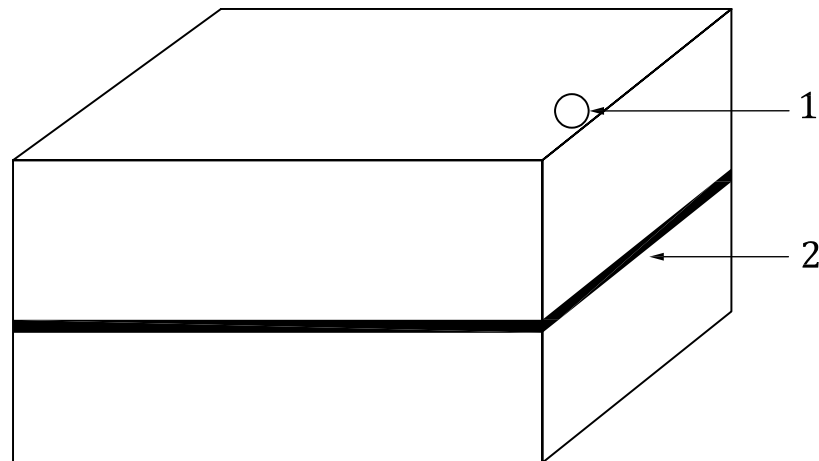
6.1 Test pieces

The size(s) and shape(s) of the test pieces adopted are governed by the type and form of material received from the various suppliers, e.g. wrought products (bar, rod, plate, strip) or cast products (sticks of varying shapes).

Three basic forms of test piece are considered appropriate, i.e. rod, disc, or block. These simple geometries are easier to measure and hence less prone to errors.

Machine tolerances should preferably be better than $\pm 0,05 \text{ mm}$; however, on larger test pieces, this requirement may be relaxed.

Reference marks are not usually made on test pieces exposed in the laboratory. However, for special cases or other exposures, e.g. probes in plant, test pieces may contain reference positions sufficient to identify a specific point within the measurement plane, both pre- and post-exposure. The reference marks serve as a datum, thus ensuring that repeat measurements are made at identical positions on the test piece. This can be achieved by use of a reference mark and reference groove. These reference marks and grooves vary with test piece geometry. [Figure 2](#) shows the reference mark and groove for a block sample; similar marks and grooves may be placed on other sample geometries.

**Key**

- 1 reference mark
- 2 reference groove

Figure 2 — Example of reference mark on block sample

6.2 Procedure

6.2.1 Examination prior to exposure

The dimensions of the uncorroded sample shall be measured prior to exposure. The dimensions of the test piece shall be measured with a precision of $\pm 0,02$ mm by means of the measuring instruments specified in ISO 3611 and ISO 13385-1. Measurements should be taken at a minimum of eight equidistant positions across the intended section for the post-test measurements.

This forms the basis of the definition of the original metal surface.

6.2.2 Preparation of cross-sections

Dimensional changes of the test piece after exposure shall be determined by preparing a metallographic cross-section. Particular care should be taken in preparation of this cross-section to ensure that the test piece is mounted and polished parallel to the intended plane of the cross-section and all corrosion products and deposits are retained. The procedure recommended is as follows.

In order to retain the corrosion product and deposit, the test piece shall be protected with an appropriate coating. A deposited metallic coating or thermoplastic resin may be suitable depending on the system to be investigated.

Specimen supports may be included in the mount to ensure that the section surface remains planar. The artefacts described below may fulfil this purpose.

The test piece may be sectioned before or after mounting. Rod and tube test pieces should be sectioned to a circular cross-section normal to the primary axis. Disc test pieces should be cut parallel to the primary axis. Block test pieces should be cut parallel to the longest side. Sectioning details are shown in [Figure 3](#).

If there is a requirement to reveal a cross-section at a specific plane relative to the surface by a reference mark, the cut should be made at a sufficient distance from that plane to allow for removal of material during grinding and polishing (see [Figure 3](#)).