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Corrosion of metals and alloys — Test method for high temperature corrosion testing of metallic materials by embedding in salt, ash, or other solids

Corrosion des métaux et alliages — Méthode d'essai pour essais de corrosion à haute température de matériaux métalliques par enfouissement dans du sel, des cendres ou d'autres solides

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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 WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

The committee responsible for this document is ISO/TC 156, *Corrosion of metals and alloys*.

Introduction

In contrast to high temperature corrosion occurring in a gaseous environment, which is covered in ISO 21608 and ISO 13573, this International Standard focuses on high temperature corrosion occurring on materials that are in direct contact with solid corrosive substances, commonly referred to as under-deposit corrosion.

This International Standard considers the case of a metallic material embedded, partially or completely, in a corrosive powder that is present in abundance and remains completely or partially in the solid state throughout high temperature exposure. Partially embedded test pieces offer the advantage of allowing investigation of corrosion at three different positions, i.e. under the powder, at the powder/gas phase interface, and in the gas phase above the corrosive powder.

The closely-related condition involving immersion in a molten salt or other liquid is described in ISO 17245, and that involving application of a surface deposit of salt, ash, or other substances in ISO 17224.

Corrosion of metals and alloys — Test method for high temperature corrosion testing of metallic materials by embedding in salt, ash, or other solids

1 Scope

This International Standard specifies the method for high temperature corrosion testing of metallic materials by embedding in a corrosive powder made up of salt, ash, and/or other solids and exposing them to high temperature in a controlled gas environment to evaluate their corrosion resistance.

Two options exist: fully embedding and partially embedding.

Fully embedding is used in cases in which a mass change measurement is required.

Partially embedding is used to investigate three potentially different interaction zones on a single test piece, i.e. the embedded part, the non-embedded part, and the gas/powder interface region. Therefore, gravimetric measurements are not appropriate and metallographic investigations are necessary instead.

This International Standard does not cover methods where test pieces are immersed in a liquid or cases in which a surface deposit is applied to the test pieces. These methods are covered in ISO 17245 and ISO 17224, respectively.

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 6906, *Vernier callipers reading to 0,02 mm*

ISO 8044, *Corrosion of metals and alloys — Basic terms and definitions*

ISO 8407:2009, *Corrosion of metals and alloys — Removal of corrosion products from corrosion test specimens*

ISO 21608:2012, *Corrosion of metals and alloys — Test method for isothermal-exposure oxidation testing under high-temperature corrosion conditions for metallic materials*

ISO 26146, *Corrosion of metals and alloys — Method for metallographic examination of samples after exposure to high-temperature corrosive environments*

ASTM E3, *Standard guide for preparation of metallographic specimens*

ASTM E220, *Standard method for calibration of thermocouples by comparison techniques*

ASTM E230, *Standard temperature-electromotive forces tables for standardized thermocouples*

ASTM E407, *Standard practice for microetching metals and alloys*

ASTM E1350, *Standard test method for testing sheathed thermocouples prior to, during and after installation*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8044 and the following apply.

3.1
affected layer
layer beneath the test piece surface that is affected in its composition and/or structure due to corrosion

3.2
corrosive powder
powder mixture made up of salt, ash, and/or other solids that contains compounds that are expected to react with the metal at high temperature

3.3
controlled gas environment
flowing gas mixture of defined composition and flow rate which can affect the behaviour of the corrosive powder

3.4
descaling
removal of corrosion products and corrosive phases from the test piece surface before measurement of the mass of the remaining metal

4 Test method

4.1 Principle

Mass change measurements can be performed with fully embedded test pieces. In this case, the test shall be performed with at least three test pieces of each material to ensure reproducibility of the test results. Two test pieces shall be used for determination of mass change or dimensional change, and one shall be used for observation of the surface and/or cross section.

4.2 Reagents and materials

4.2.1 Test pieces

Test pieces for fully embedding shall have the form of a rectangular plate, a disc, or a cylinder with a minimum surface area of 300 mm^2 and a minimum thickness of 1,5 mm.

Test pieces for partially embedding shall have the form of a rectangular plate with a minimum length of 60 mm and a minimum thickness of 1,5 mm. The rectangular geometry allows accurate metallographic examinations of longitudinal sections as well as transverse sections, which is not possible with a cylindrical test piece.

If the test pieces cannot be made according to these specifications, the shape and dimensions of the test pieces shall be in accordance with the agreement between the parties involved.

The test pieces shall be machined to remove the strata affected by cutting.

The final finishing of the surface of the test pieces shall be performed with abrasives with a mean particle diameter of approximately 15 µm. This can be achieved by the use of abrasives according to Table 1.

Table 1 — Designation and mean diameter of particles of coated abrasives according to regional standards

Standard	Designation	Mean diameter µm	Region
FEPA[a] 43–1984 R 1993: Grit Sizes for Coated Abrasives ISO 6344 Coated abrasives — Grain size analysis	P1200	15,3 ± 1,0	Europe
JIS R6001–87	#1000	15,5 ± 1,0	Japan
ANSI B74.12–92 — Specifications for the Size of Abrasive Grain — Grinding Wheels, Polishing and General Industrial Uses	600	16,0	America

[a] Federation of European Producers of Abrasives.

If another surface finish is required by the parties involved, the surface finish condition shall be described.

Sharp edges of test pieces might give anomalous behaviour. These shall be slightly rounded during the final stages of test piece preparation.

The dimensions of the test pieces shall be measured prior to exposure at a minimum of three positions for each dimension with a precision of ±0,02 mm by means of the measuring instruments specified in ISO 3611 and ISO 6906.

After ultrasonically degreasing in isopropanol or ethanol, the test pieces shall be dried in hot air or in a desiccator.

If it is suspected that test pieces might absorb significant amounts of atmospheric contaminants such as water, it is recommended that the cleaned test pieces be stored in a desiccator prior to weighing and exposure.

4.2.2 Corrosive powder

The type of powder to be used in the test shall be selected in accordance with the environment for which the test is intended. The powder shall be prepared either by taking ash or deposit from actual equipment or by mixing chemicals of reagent grade.

The particle size of the powder shall not exceed 100 µm. When using reagent grade chemicals, the particle size (according to the supplier's specification) shall be recorded.

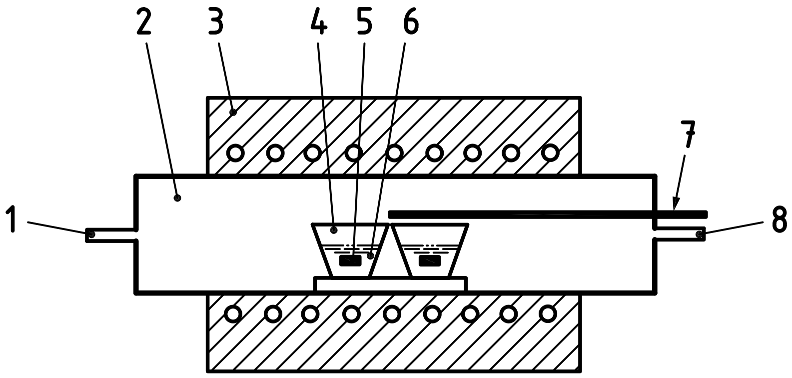
WARNING — When working with dangerous substances (such as certain ashes, molten salts, heavy metals, and organic materials), all necessary safety precautions shall be taken.

4.3 Test apparatus

4.3.1 Design of apparatus

The apparatus shall be composed as a whole, of the temperature-regulating device for heating all test pieces at a uniform temperature. The heating device shall be equipped with a testing portion capable of separating the test pieces from outside air. The gas supply shall be controlled by a gas flow meter.

A basic design of a closed, horizontal, apparatus is shown in Figure 1. Other designs can use vertical orientation. A design of the complete furnace setup is shown in ISO 21608:2012, Figure 1.



Key

1	test gas inlet
2	test chamber
3	heating unit
4	crucible
5	test piece
6	corrosive substance
7	thermocouple
8	gas exit

Figure 1 — Apparatus using a horizontal furnace

The test chamber shall not be composed of materials that react significantly with the gas environment or the corrosive substance during the test. If the reaction is not avoidable, the reaction shall be small enough to neglect a change in composition of the atmosphere.

4.3.2 Temperature monitoring

The temperature distribution of the furnace shall be characterized at the exposure temperature prior to the testing to determine the width of the isothermal zone by the use of a movable thermocouple.

The temperature-regulating device shall be capable of guaranteeing that the temperature of the test piece is kept within the permissible range given in Table 2.

Table 2 — Permissible tolerance of temperature of test pieces

Temperature range, °C	≤300	300 to 600	600 to 800	800 to 1 000	1 000 to 1 200	>1 200
Temperature tolerance, °C	±2	±3	±4	±5	±7	By agreement

Thermocouple sheaths shall be used to protect the thermocouple wires and shall fully withstand the test temperature and environment.

A specimen thermocouple shall be positioned as close as possible to the test piece. If the experimental condition does not allow the use of such a thermocouple, the temperature of the test piece has to be deduced from the furnace calibration using dummy test pieces in an appropriate environment.

Calibration of thermocouples shall be performed in accordance with ASTM E220, ASTM E230, and ASTM E1350. A representative thermocouple taken from the batch of wire may be calibrated.

Thermocouples shall be recalibrated annually or at the beginning and the end of each experiment if there is uncertainty about thermocouple stability.

4.3.3 Gas supply

The gas supply system shall be capable of supplying the test gases at a constant flow rate to the test piece chamber described in 4.3.1.

When a humidifying regulator is used, it shall be capable of adjusting to the desired humidity. Deionized water of electrical conductivity less than 1 μS cm^{-1} shall be used.

The gas line between humidifying regulator and test chamber shall be kept above the dew point in order to avoid condensation. Condensation might have a severe impact on the result.

The gas flow shall be monitored by a gas flow meter. The flow meter shall be located as close as possible to the inlet of the test chamber except where a humidifying regulator is used, in which case, it shall be located upstream from the humidifier.

In the case that the gas is humidified, the water vapour content shall be measured. This can be achieved by e.g., the use of a hygrometer before the test chamber or by measuring the amount of water after condensation of the exhaust gases or by measuring the water consumption of the humidifier over the course of the experiment.

4.4 Procedure

4.4.1 Preparation and placement of the test piece

The crucibles shall be inert to the environment. Each crucible shall contain only one test piece.

Crucibles shall be baked in air to remove volatile compounds before their first use. The recommended baking condition is at least 24 h at a temperature of 1 000 °C. If water absorption is suspected to have occurred, used crucibles shall be dried at significantly above 100 °C.

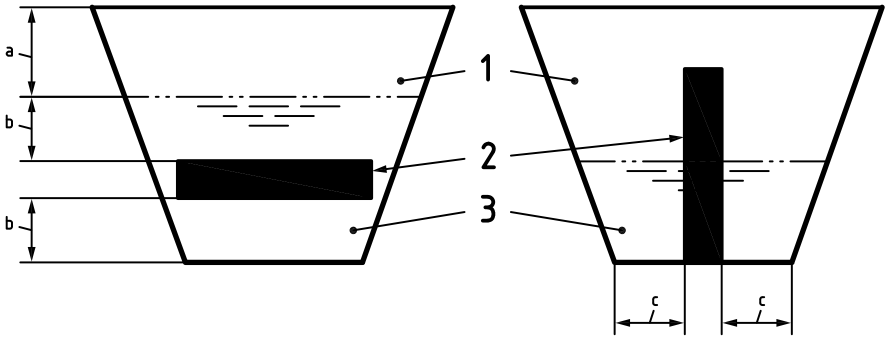
For fully embedding tests, a layer of at least 3 mm of corrosive powder shall be put in the crucible and the top of the powder shall be flattened by applying a uniform pressure (about 0,04 MPa = 40 g/cm^2).

NOTE This can be achieved by applying a flat metal piece with the appropriate mass to area ratio to the entire surface area of the powder in one or more applications.

After the test piece is set on the bed of powder, the additional powder shall be put on top of the test piece to cover it with a powder layer of at least 3 mm in thickness. Finally, the top layer shall be flattened by the procedure described above. The distance between the top of the powder and the top of the crucible shall not exceed 3 mm.

The test pieces for partially embedding tests shall be placed upright in crucibles with a height of at least the length of the test pieces and a diameter that leaves at least 5 mm on each edge of the test pieces. Corrosive powder shall be filled into the crucibles up to half the height of the test pieces and its top layer shall be flattened by the procedure described above.

The configuration of fully embedded and partially embedded test pieces in crucibles is shown in Figure 2.



Key

1 crucible
2 test piece
3 corrosive powder
a Max. 3 mm.
b Min. 3 mm.
c Min. 5 mm.

NOTE Left = fully embedding tests; right = partially embedding tests.

Figure 2 — Test piece fully embedded and partially embedded in corrosive powder

4.4.2 Test environment

The composition of the test gas shall be chosen in a way that it keeps the corrosive activity of the corrosive powder stable.

The flow rate of the test gas shall be sufficient as to fill the test chamber at least three times per hour. This is to ensure that the corrosion rate is not determined by the supply of the reactants.

The gas flow shall be preheated to reach equilibrium in the gas phase. An appropriate catalyst shall be used, if necessary.

4.4.3 Heating method

The test pieces in their crucibles shall be placed in the furnace either at room temperature or at the test temperature, by agreement between the parties. In the former case, the time to reach the test temperature shall be recorded.

Where test pieces are loaded into a cold furnace, heating shall be carried out in the test gas. In the case of humidified atmospheres, the humidification shall not be started until the temperature in the coldest part of the test chamber exceeds the dew point of possible condensing phases.

If heating in the test gas is not acceptable from a safety perspective, heating shall be performed in a gas similar in composition to the test gas but with the critical components absent until a temperature has been reached and at which point, the safety concerns have been resolved. Where test pieces are loaded into a hot furnace, a flow of inert gas can be used during the loading procedure.

The heating shall be carried out in a manner that the temperature of test pieces does not exceed the upper limit of permissible range listed in Table 2.

4.4.4 Test duration

The test is defined to commence when the test piece temperature exceeds 97 % of the desired test temperature, T_{dwell} (measured in K).

The test ends when the temperature of the test piece falls below 97 % of the desired test temperature, T_{dwell} (measured in K).

The test duration shall be relevant to the intended application and agreed upon between the parties concerned. The materials' behaviour, observed in short-term exposures, might not continue at long durations. Therefore, for testing of relevance to long-term service conditions, the entire exposure time shall be at least 300 h.

4.4.5 Cooling of test pieces

If furnace cooling is used, it shall be carried out in the test gas down to 200 °C and the time to cool down to 50 °C shall be recorded. The test pieces can also be removed from the furnace at test temperature. A dry purge gas atmosphere shall be applied for a sufficient duration before removing the test pieces from the furnace. In the case of humidified atmospheres, the humidification shall be stopped before the temperature in the coldest part of the test chamber falls below 200 °C.

If cooling in the test gas is not acceptable from a safety perspective, cooling shall be performed in a gas similar in composition to the test gas but with the critical components absent.

4.4.6 Discontinuous testing

For discontinuous exposure, the same heating methods (see 4.4.3) and cooling methods (see 4.4.5) shall be applied each time the test pieces are reloaded for continued exposure.

Each time before the test pieces are reloaded, the corrosive powder shall be completely replaced. If the corrosive powder cannot be completely replaced without damage to the samples, replacement shall be in accordance with the agreement between the parties involved.

4.5 Determination of mass change

4.5.1 Principle

One method to determine corrosion kinetics is to measure mass change. This is possible only with fully embedded test pieces, and only if the corrosive powder can be removed completely after testing. Duplicate test pieces of each material shall be used. Test pieces should be weighed as soon as possible or shall be stored in a desiccator after removal from the furnace until immediately before weighing. When handling test pieces, tweezers shall be used. Test pieces shall never be touched with the hands to avoid contamination (e.g., grease, salts). Care has to be taken when using gloves as the contamination with the separating agent of the gloves leads to falsification in mass determination. If repeated measurements are not consistent, the temperature and humidity of the weighing environment has to be controlled.

4.5.2 Measurements prior to testing

The mass of the test pieces shall be determined prior to exposure [$m_{\mathrm{T}}(t_0)$, in ISO 21608:2012, Figure 5]. At least two individual measurements shall be made for each test piece with a precision of 0,02 mg. The maximum difference between the measurements shall not exceed ±0,05 mg.

4.5.3 Descaling prior to mass determination

If descaling prior to mass determination is intended, all corrosion products shall be removed from test pieces with a minimum removal of sound metal. This shall be done according to ISO 8407.

Additional procedures are given in Annex A (see Table A.1 and Table A.2).

After descaling, the test pieces shall not be used for further corrosion testing.

NOTE These procedures are not suitable to remove internal corrosion products as defined in ISO 26146.

4.5.4 Measurement of corrosion mass loss

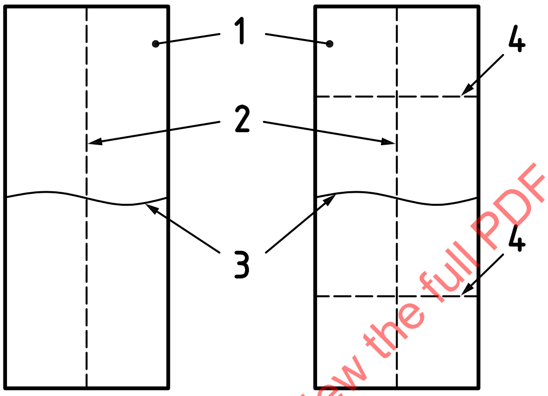
The mass loss of the test pieces due to corrosion shall be determined according to ISO 8407.

5 Examination of surface and microstructure of corroded test pieces

Metallographic examination shall be made on the surface and cross section of fully embedded test pieces.

The metallographic examination of partially embedded test pieces serves the purpose of characterizing three potentially different zones on a single test piece (i.e. the embedded part, the non-embedded part, and the gas/powder interface region). Metallographic examination shall be made of the surface in plain view, of a longitudinal section, and, if required, of at least two transverse sections representing the embedded and the non-embedded zones (see Figure 3).

Detailed procedures for examination are provided in ISO 26146.



Key

1 test piece
2 longitudinal section
3 gas/powder interface
4 transverse sections

Figure 3 — Two examples of preparation of test pieces for metallographic examination

6 Report

6.1 Matters to be described

The following data, where known, shall be included in the report on the test results.

6.1.1 Test material

a) Manufacturer

b) Name of material (Manufacturer designation; ASTM, DIN, etc.)

c) Grade or symbol

d) Heat number/Batch number

e) Chemical composition (analysis technique)

f) Processing condition

g) Heat treatment condition

h) Microstructure of the material determined according to ASTM E3 and etching according to ASTM E407

6.1.2 Test piece

a) Designation of test piece

b) Sampling conditions of the test piece from raw material (Crystallographic orientation, rolling direction, etc.)

c) Dimensions (in mm) and surface area, A, (in cm^2) of test piece

d) Surface finish condition of test piece

e) Degreasing method of test piece

f) Initial mass of fully embedded test piece for mass change measurement

6.1.3 Testing environments

a) Test temperature

b) Characteristic heating and cooling curves of the test piece in the powder

c) Test duration

d) Volume of test chamber

e) Composition of test gas, including humidity

f) Volumetric flow rate of test gas in normal cubic metres per second

g) Chemical composition, particle size distribution (if available), and origin of the corrosive powder

h) Amount of corrosive powder used per crucible

i) Size, type, and material of the crucibles used

6.1.4 Test results

a) Mass loss of fully embedded test pieces caused by descaling procedures, according to 4.5.3

b) Mass loss of fully embedded test pieces caused by the corrosion experiment, according to 4.5.4

c) In case of determination of corrosion kinetics: mass loss of fully embedded test pieces, Δm, divided by surface area, A, as a function of time

d) Image of appearance after testing

e) Image of cross sections, including the surface layer of the metallographic section of test piece after testing (the chosen magnifications shall clearly show the extent of the total attack in a single micrograph)

f) Thickness profile of unaffected metal along the longitudinal section of partially embedded test pieces

g) Minimum thickness of unaffected metal in each zone of partially embedded test pieces

h) Results of any other metallographic investigations performed according to Clause 5

i) Results of analysis of corrosive powder after long-term exposure, if performed, according to 4.2.2

6.2 Supplementary note

It is desirable to additionally describe the following matters in the report on the test results:

a) mechanical properties of the initial material;

b) microstructure of the initial material and sampling conditions;

c) further details of the test apparatus;

d) characteristics of the corrosive powder determined by differential thermal analysis and thermogravimetry, if available.

Annex A
(informative)

Chemical and electrolytic procedures for removal of corrosion products from fully embedded test pieces

A.1 General

In the development of this International Standard, a number of sources were consulted to identify chemical and electrolytic descaling procedures. They can be used in addition to those listed in ISO 8407, and they are specifically designed for the purpose of the tests described in this International Standard. This Annex summarizes the results of the survey.

Prior to adopting these procedures, the user should conduct a test on control test pieces to ensure the efficiency of the chosen method. Excess descaling might result in dissolution not only of corrosion products but also of metal substrates.

A.2 Procedures

Table A.1 and Table A.2 summarize various chemical and electrolytic procedures for removal of corrosion products. The specific choice of procedure for a given material will depend on many factors, including previous experience.

For all the procedures listed, it is recommended that the surfaces should be maintained vertical during cleaning. This will minimize retention of any gases released during the procedure.

The times specified represent recommendations appropriate for mass-loss studies in the context of ISO 8407:2009, Figure 1.

When using electrolytic procedures, selecting adequate electrolysis parameters and using blank specimens is recommended, in order to ensure precise removal of corrosion products. A typical setup is shown in Figure A.1.

WARNING — When working with dangerous substances (such as sodium hydroxide, potassium permanganate, hydrochloric acid, and molten salts), all necessary safety precautions shall be taken.