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Corrosion of metals and alloys — Test method for high temperature corrosion testing of metallic materials by immersing in molten salt or other liquids under static conditions

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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.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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*.

<u>ISO 17245:2015</u> https://standards.iteh.ai/catalog/standards/sist/28c9dd01-60cb-41e9-a124e87e75ed1092/iso-17245-2015

Introduction

In contrast to high temperature corrosion occurring in gaseous environment covered in ISO 21608 and ISO 13573, this International Standard focuses on high temperature corrosion occurring on materials that are in direct contact with molten corrosive substances.

The present document considers the case of a metallic material immersed completely in a corrosive substance that either melts during high temperature exposure or that is liquid throughout the experiment.

The closely-related condition involving exposure in a compacted powder is described in ISO 17248 and that involving application of a surface deposit of salt, ash, or other substances in ISO 17224.

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Corrosion of metals and alloys — Test method for high temperature corrosion testing of metallic materials by immersing in molten salt or other liquids under static conditions

1 Scope

This International Standard specifies the method for high temperature corrosion testing of metallic materials by immersing in molten salt or other liquids which are under static conditions and exposing them to high temperature in a controlled gas environment to evaluate their corrosion resistance.

Two options are considered

- a) using a stagnant inert gas environment, and
- b) using a flowing reactive gas environment.

This International Standard does not cover methods where test pieces are fully or partially embedded in a corrosive powder made up of salt, ash, and/or other solids, or cases in which a surface deposit is applied to the specimens. These methods are covered in ISO 17248 and ISO 17224, respectively.

This International Standard is not applicable to the situation of test pieces in relative movement to their surrounding corrosive liquid.

NOTE This situation is intended to be dealed with in a future International Standard. https://standards.iteh.ai/catalog/standards/sist/28c9dd01-60cb-41e9-a124-

e87e75ed1092/iso-17245-2015

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

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

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

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

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 liquid

powder mixture made up of salt, ash, and/or other solids that will melt under the experimental temperature or liquid 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 liquid

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

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4.1 Principle

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The test shall be performed with at least three test pieces of each material to ensure reproducibility of the test results. These test pieces can be used for determination of mass change, dimensional changes, and/or for observation of the surface and/or cross sectionsist/28c9dd01-60cb-41e9-a124-

e87e75ed1092/iso-17245-2015

4.2 Reagents and materials

4.2.1 Test pieces

The test pieces shall have the form of a rectangular plate, a disc, or a cylinder with a minimum surface area of 300 mm² and a minimum thickness of 1,5 mm.

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 mean particle diameter of approximately 15 μ m. This can be achieved by the use of abrasives according to <u>Table 1</u>.

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	P1200	153+10	Europe
ISO 6344 Coated abrasives - Grain size analysis	11200	10,0 = 1,0	Lurope
JIS R6001–87	#1000	15,5 ± 1,0	Japan
a Federation of European Producers of Abrasives			

Table 1 (continued)

Standard	Designation	Mean diameter μm	Region
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 $\pm 0,02$ 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 are stored in a desiccator prior to weighing and exposure.

The mass of the test pieces shall be determined prior to exposure. At least two measurements shall be made for each test piece. The difference between the measurements shall not exceed 0,05 mg.

4.2.2 Corrosive substance

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The type of corrosive substance to be used in the test shall be selected in accordance with the environment for which the test is intended. It shall be prepared either by taking deposit from actual equipment or by mixing chemicals of reagent grade. ai/catalog/standards/sist/28c9dd01-60cb-41e9-a124-

The prepared substance shall be mixed well to make it homogeneous. The melting range of the substance shall be measured in advance if it is not known, or it shall be determined from phase diagrams in order to ensure that the liquid phase is the major component.

WARNING — When working with dangerous substances (such as certain ashes and molten salts), 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 (closed system).

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



Кеу

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

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Figure 1 — Closed 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 atmosphere45-2015

NOTE Condensation can be an issue, which can be addressed by heating flanges or ensuring that the ratio of total crucible volume to chamber volume is 1:10.

4.3.2 Stagnant inert gas environment

The test chamber shall be filled with a gas that is inert to the corrosive substance prior to the test and then closed. An overpressure valve should be used in order to maintain the pressure of the system at desired pressure.

4.3.3 Flowing reactive gas environment

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.

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.

4.3.4 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 to guarantee that the temperature of the test piece is kept within the permissible range given in <u>Table 2</u>.

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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. The thermocouple sheaths shall withstand fully the test temperature and environment.

A specimen thermocouple shall be positioned as close as possible to the crucibles. The temperature of the test piece shall be deduced from the furnace calibration using dummy test pieces in an appropriate environment under thermal equilibrium.

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 can be calibrated.

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

4.4 Procedure ISO 17245:2015 https://standards.iteh.ai/catalog/standards/sist/28c9dd01-60cb-41e9-a124-

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4.4.1 Preparation and placement of the test piece

The crucible shall be inert to the environment.

The configuration of the test piece in an alumina, silica, or zirconia crucible, depending on the acidic or basic character of the corrosive substance, is shown in Figure 2.

Crucibles shall be baked in air to remove volatile compounds before their first use. The recommended baking conditions are 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.

The amount of corrosive substance in each crucible shall be at least 20 ml per 1 cm^2 of the surface area of a test piece. The corrosive substance shall be placed in a manner that the depth between the surface of the corrosive substance and the surface of the test piece will be at least 5 mm in the liquid phase.

The volume shrinkage of the corrosive substance during melting shall be determined in a separate test using the same powder loading procedure as will be used in the corrosion test.