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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 dynamic conditions
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Corrosion des métaux et alliages — [to be translated]
Corrosion des métaux et alliages — [to be translated]
Corrosion des métaux et alliages — [to be translated]

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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.

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

This second/third/... edition cancels and replaces the first/second/... edition (), [clause(s) / subclause(s) / table(s) / figure(s) / annex(es)] of which [has / have] been technically revised.

Introduction

In contrast to high temperature corrosion occurring in gaseous environment, which is covered in the international standards 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, ~~commonly referred to as hot corrosion, where the molten salts fluid is in relative motion to materials.~~

The present document considers the case of a metallic material ~~embedded~~immersed, partially or completely, in a corrosive ~~molten substance that either melts during high temperature exposure or that is liquid throughout the experiment~~salts, where the ~~molten salt liquid can be~~is in motion. ~~Tests with samples immersed under static conditions are covered by ISO 17245.~~

The closely-related condition involving ~~exposure in a compacted powder is described in ISO 17248 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.

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1 Scope

This international standard specifies the method for high temperature corrosion testing of metallic materials by ~~immersing embedding~~ in molten salts ~~or other liquids~~, ~~under dynamic conditions~~, and exposing them to high temperature in a controlled gas environment to evaluate their corrosion resistance.

Two options exist: ~~fully embedding immersing test pieces in an open system, and in a closed test loop.~~

~~Fully embedding is used when not closed loop test is available.~~

~~Closed Test loops, in which liquid is circulating, test are used to investigate many different phenomena: impact of hydrodynamics on corrosion, liquid chemistry effect on corrosion (study of down/upstream effect), temperature effect on corrosion (with two tests sections in the loop), mass transfer in a closed system... tubes interaction zones or different fluid boundary layer regimens (i.e. different adimensionalless numbers, such as Reynold's number).~~

~~The procedures used for corrosion tests are inherent to the corrosive liquid and then no specific procedure is given in this document. This document does not cover methods where specimens are fully or partially embedded in a corrosive powder made up of salt, ash, and/or other solids immersed in a liquid, or cases in which a surface deposit is applied to the specimens. These methods are covered in ISO 17245 and ISO 17224, respectively.~~

~~This document does not cover the situation of test pieces immersed under static conditions, which is the subject of ISO 17245.~~^[RF21]

2 Normative references

The following referenced documents are indispensable for the application 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.

ASTM E220-02, *Standard method for calibration of thermocouples by comparison techniques*

ASTM E230-02, *Standard temperature-electromotive forces tables for standardized thermocouples*

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ASTM E1350-97, *Standard test method for testing sheathed thermocouples prior to, during and after installation*

ISO 3611:1978, *Micrometer callipers for external measurement*

ISO 6906:1984, *Vernier callipers reading to 0,02 mm*

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*

3 Terms and definitions

For the purposes of this document, [ISO 8044 and](#) the following terms and definitions 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/liquid

~~Either powder mixture made up of salt, ash, metal, glass 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 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 and be melted in contact with metallic materials.~~

3.3

controlled gas environment

~~flowing~~ gas mixture of defined composition and flow rate which may affect the behavior of the corrosive powder/liquid

3.4

descaling

removal of corrosion products and corrosive phases from the specimen surface before measurement of the mass of the remaining metal

4 Test method

4.1 Principle

Mass change measurements may be performed with ~~fully embedded sample~~ test pieces that are suspended in a removable sample holder. 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 specimens test pieces shall be used for determination of mass change, and one shall be used for observation of the surface and/or cross section. This procedure will shall be applied when a dynamic closed test dynamic is conducted in a closed loop will be used, where a holding sample test piece holder will be placed inside the flow.

~~When a rotating device test piece holder is will be used, only metallographic analysis before and after exposure will shall be used performed in all cases.~~

When a dynamic test is conducted in a closed loop, the sample holder position has to be chosen to least disturb the flow. In this case the test shall be performed with at least two test pieces of each material to ensure reproducibility of the test results.

When a rotating test piece holder is used, it can be difficult to have two samples having exactly the same hydrodynamic conditions and then one sample can be used.

In both cases if pure oxidation of samples occurs, different materials can be used during the same test. However, in case of corrosion by dissolution of the sample, release of corrosion products can occur and only one material grade can be used per test; in order to avoid that corrosion of a given grade interfere with the corrosion of another.

In case of corrosion by dissolution and in case of absence of a purification loop of the corrosive liquid, the volume of liquid has to be sufficient in comparison to the total surface of the samples that solubility limits of samples elements are not reached during the whole duration of the test and concentrations of these elements are can be assumed as constant during the corrosion test of the sample. If measure of dissolved elements concentration is possible, it would be highly beneficial for qualification of the materials. If measurement is not possible, it must be assumed or evaluated.

4.2 Reagents and materials

4.2.1 Test pieces for closed loop testing and rotating system

- a) Test pieces for immersing in a closed loop testing fully embedding shall have the form of a rectangular plate, a disc or a cylinder with a surface area of 300 mm² at minimum and a thickness of 1,5 mm at minimum.
- b) Test pieces for immersing in a rotating system testing fully embedding shall have the form of a cylindrical shape, a disc or a cylinder with a surface minimum dimensions of 7 mm diameter per and 15 mm length. [RF28]

[L9] 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. In this case the dimensions shall be chosen in such a way that bubbling [L10] does not occur during the test.

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 µm. [L11] 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.

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Sharp edges of test pieces may give anomalous behavior. These shall be slightly rounded during the final stages of test piece preparation.^[L12]

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.^[L13]~~

If it is suspected that specimens may adsorb significant amounts of atmospheric contaminants such as water it is recommended that the cleaned test pieces are stored ~~in a desiccator^[L14] under vacuum conditions or under inert gas~~ prior to weighing and exposure.

4.2.2 Corrosive substance powder

~~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.~~

~~The type of corrosive substance 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 testing temperature must be according to the powder melting point.~~

~~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. 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, liquid metals), 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 and a container for the corrosive liquid, 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 container can be an open system or a closed loop. When applicable, in a closed system a humidifying regulator^[L15] should be used to continuously supply the gas kept at a constant humidity which should be monitored with a hygrometer^[L16]. The gas supply shall be controlled by a gas flow meter.~~

- a) ~~For a closed testing loop option, the next figure allows to give a schematic of the principal parts: pump, pipes, testing chamber is shown in Figure 1 where the testing materials where places. As corrosion kinetics can depend on the mass transfer, hydrodynamics has to be characterized for the performed experiments. Determination of flow rate is not sufficient as, for the same flow velocity, when geometry changes, hydrodynamic at contact with sample also changes. The design of the equipment shall ensure that the temperature is uniform to ± 5 °C between the reservoir and the test chamber. The linear velocity across the samples must be adjusted, in terms of more accurate laboratory conditions flow rate of the salt shall be determined in order to achieve the linear velocity agreed between the parties involved to reproduce the real operation conditions of the device.^[L17]~~

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

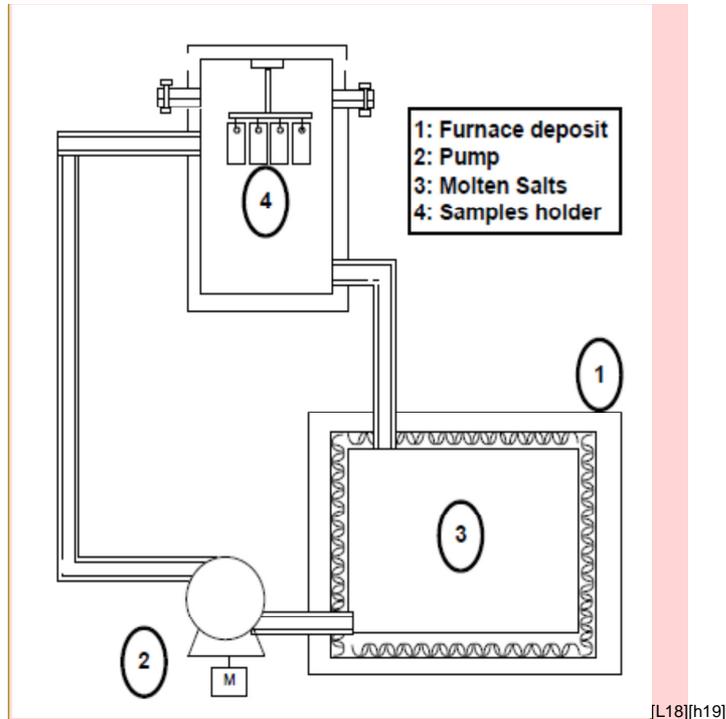


Figure 1 – Apparatus for closing test loop

For each experiment, flow must be at least characterized by the Reynolds Number, the Schmidt number and a description of the system geometry. If mass transfer coefficient can be calculated or measured, it is better. Presence of samples has to disrupt at minimum the flow. The number of the samples in the flow has not to modify the flow. If it modify it, the sample number has to be kept constant during the whole experiment (when one sample is removed, another one is immersed).

- b) For a rotating testing apparatus, a schematic the next figure allows to give a schema of the principal parts: Furnace, testing reactor, crucible, and rotater device (is shown in Figure 2) ing stirring where four cylindrical samples where the testing materials where placed. The rate of rotation of the test pieces shall be determined in order to achieve the linear velocity. The mass transfer coefficient agreed between the parties involved linear velocity across the samples must be adjusted, in terms of more accurate laboratory conditions to reproduce the real operation conditions of the device.

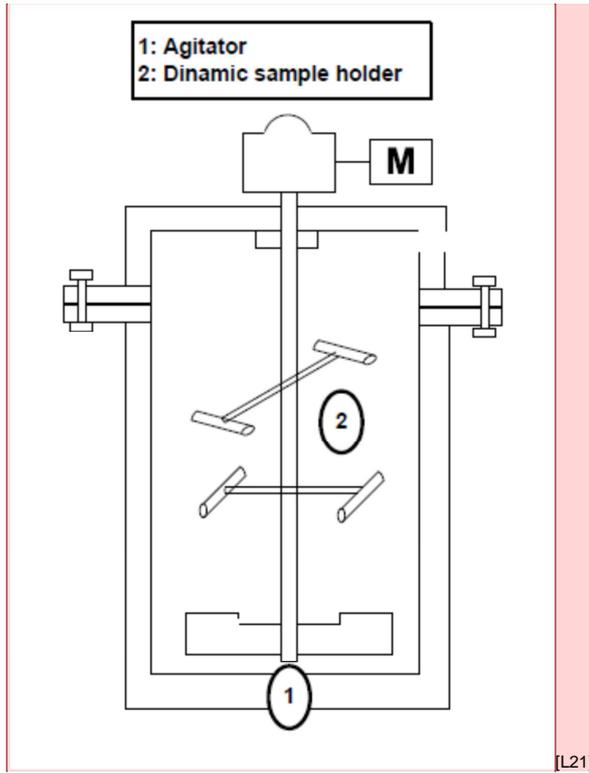


Figure 2 – closed apparatus for rotating test pieces samples Apparatus [RF222]

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

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

A specimen thermocouple shall be positioned immersed in the molten salt corrosive liquid. If the experimental condition does not allow the use of such a thermocouple, the temperature of the test piece must be measured, protecting the thermocouple inside of a ceramic tube. [L23]

Calibration of thermocouples shall be performed in accordance with ASTM E220-02, ASTM E230-02, and ASTM E1350-97. A representative thermocouple taken from the batch of wire may be calibrated.

Thermocouples immersed in the liquid shall be recalibrated annually or at the beginning and the end of each experiment if there is uncertainty about thermocouple stability. [L24].

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 4.4.3.1.

When a humidifying regulator is used it shall be capable of adjusting to the desired humidity. Deionized water of electrical conductivity less than $1 \mu\text{S cm}^{-1}$ 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 vapor 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.

On the other hand, experiments can be conducted in a closed reactor atmosphere in the two types of possible test.

4.4 Procedure

4.4.1 Preparation and placement of the sample

- a) For a closed testing loop option, the samples will be placed in the test chamber. A minimum separation of 8 mm [RF225] among samples is recommended. The samples rack must be placed when the salt corrosive substance is totally molten if it is possible. [L26]
- b) For a rotating test and if it is possible, the stirring samples holder must be placed inside of the salt corrosive substance when it is totally molten.

4.4.2 Test environment [RF227]

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

The flow rate of the test gas shall be so high as to fill the test chamber at least three times an hour [RF228]. This is to ensure that the corrosion rate is not determined by the supply of the reactants. If a gas phase supplies a reactant, the gas flow rate must be adapted to the corrosion rate in order to avoid that the corrosion rate is controlled by reactant supply in the gas phase.

The gas flow shall be preheated to reach equilibrium in the gas phase [RF229]. An appropriate catalysts shall be used if necessary. [L30]

The test environment can be adjusted only in the rotating test system.

4.4.3 Heating method

The test pieces in their crucibles shall be placed in the furnace ~~either at room temperature, and heating shall be carried out in the test gas, or at the test temperature, by agreement between the parties. In the former case the~~ time to reach the test temperature shall be recorded. ~~Heating can also be done under inert gas (Ar, He, ...) to avoid oxidation at lower temperature than the test temperature. Moreover, if the chemistry of the liquid is not modified by the furnace opening and if the experimental device allows it, samples can be directly immersed at high temperature.~~

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 at which point the safety concerns have been resolved.

Where the ~~salt~~corrosive substances are loaded into a hot furnace a flow of inert gas ~~or the gas chosen for the tests~~ may be used during the loading procedure, and during the molten procedure.

~~NOTE In certain set-ups a fast heating rate is not possible because the heating rate can be harmful to ceramic components, such as heating elements and reaction tubes.~~

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.

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 %^[RF231] 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 between the parties concerned. The materials behavior observed in short-term exposures may not continue at long durations, therefore for testing of relevance to long-term service conditions the ~~longest~~^[RF232] exposure time shall be at least 300 h^[L33].

4.4.5 Cooling of test pieces

The test pieces ~~can be removed at the tested temperature. If it is not possible, the tests pieces must shall~~ be removed from the testing apparatus after cooling to lower temperature according to the possibilities of the experiment limiting pollution of the environment if experiment is led to continue with the same liquid, below 50 °C. The time to cool to 50 °C ~~the removing temperature~~ shall be recorded. Cooling shall be carried out in the test atmosphere or in inert atmosphere or in a gas similar in composition to the test gas but with the critical components absent down to room temperature.

~~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.~~

SAFETY PRECAUTIONS — All necessary health and safety requirements should be met when handling hot corrosive liquids.

~~The solidified substance attached to the test piece surface after cooling shall be removed using a stripping solution. Normally HCl (concentrated) solution may be used for this purpose~~^{[RF234], [L35]}

4.4.6—Discontinuous testing

For discontinuous exposure the same heating and cooling method 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.

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 closed loop testing samples [RF236].~~ 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 ~~or under inert gas (in a glove box for instance)~~ after removal from the furnace until immediately before weighing. ~~When handling the specimens tweezers shall be used have to be done with the greatest precaution. Test pieces shall never be touched with the hands to avoid contamination (grease, salts). [L37]~~ 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 ($m_T(t_0)$, Figure 5 in ISO 21608:2012). 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 specimens with a minimum removal of sound metal. This shall be done according to ISO 8407:2009.

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

After descaling the samples 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

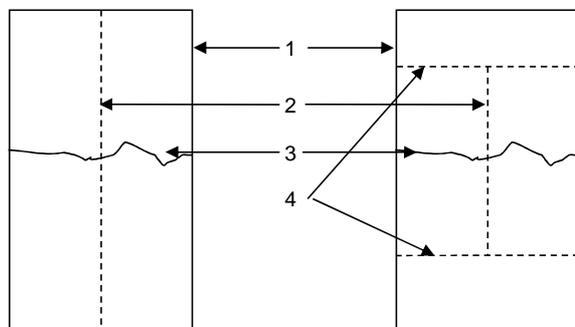
The mass loss of the test specimens due to corrosion shall be determined according to ISO 8407:2009 (2.1.3.3).

5 Examination of surface and microstructure of corroded test pieces

Metallographic examination shall be made on the surface and cross section of fully ~~embedded-immersed~~ samples.

The metallographic examination of partially ~~embedded-immersed~~ samples serves the purpose of characterizing three potentially different zones on a single test piece, i.e. the embedded part, the non-~~embedded-immersed~~ part, and the gas/powder-liquid interface region. Metallographic examination shall be made of the surface in plan view, of a longitudinal section, and, if required, of at least two transverse sections representing the embedded and the non-~~embedded-immersed~~ zones (Figure 3). [GS38]

Detailed procedures for examination are provided in ISO 26146.



Key

- 1 Test piece
- 2 Longitudinal section
- 3 Gas/powder interface
- 4 Transverse sections

Figure GS39 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 E3-01 and etching according to ASTM E407-00

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 specimen in the molten salt corrosive substance
- 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 meters per second, if it is the case
- g) Chemical composition, melting point, water content, preparation method, and origin and average particle size of the corrosive powder substance
- h) Linear velocity in the closed loop description of the geometry, Mass transfer coefficient or if not determined at least Re , Sc numbers or rotating speed in rotating apparatus if no obstacle are present in the crucible (be sure that flow, at sample interface, is not disrupted by a vortex, or put an immersed cover at the fluid surface to avoid the formation of a vortex)
- i) Open or closed system according to 4.3.1
- j) In the case of the use of open systems the humidity of the laboratory air

6.1.4 Test results

- a) Mass loss of samples (caused by descaling procedures according to 4.5.3.4) or thickness of the corroded layer if a closed loop system is used: caused by descaling procedures according to 4.5.3.4
- b) In the above case of determination of corrosion kinetics: mass loss of samples 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 must clearly show the extent of the total attack in a single micrograph.
- f) Thickness profile of unaffected metal along the longitudinal section of samples. [L40]
- g) Minimum thickness of unaffected metal in each zone of partially embedded samples. [L41]
- h) Results of any other metallographic investigations performed according to 5.
- i) Results of analyses of corrosive powder environment after long-term exposure, if performed according to 4.2.2.

6.1.5 Supplementary note

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

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- 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 salt substance determined by differential thermal analysis and thermogravimetry~~DTA and TGA~~, if available

Bibliography

- [1] ISO 17224, *Corrosion of Metals and Alloys – Test method for high-temperature corrosion testing of metallic materials by application of a deposit of salt, ash, or other substances*
- [2] ISO 17245, *Corrosion of Metals and Alloys – Test method for high-temperature corrosion testing of metallic materials by immersing in molten salt or other liquids*
- [3] JIS Z2290-2004, *General rules for high-temperature corrosion test of metallic materials*
- [4] JIS Z2293-2004, *Methods for high-temperature corrosion test of metallic materials by dipping and embedding in molten salts*

Annex A (informative)

Chemical and electrolytic procedures for removal of corrosion products from samples

A.1 General

In the development of this International Standard, a number of sources were consulted to identify chemical and electrolytic descaling procedures. They may be used in addition to those listed in ISO 8407:2009, and they are specifically designed for the purpose of the tests described in this standard. This annex summarizes the results of the survey.

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

A.2 Procedures

Tables A.1 and 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 Figure 1 in ISO 8407:2009.

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.

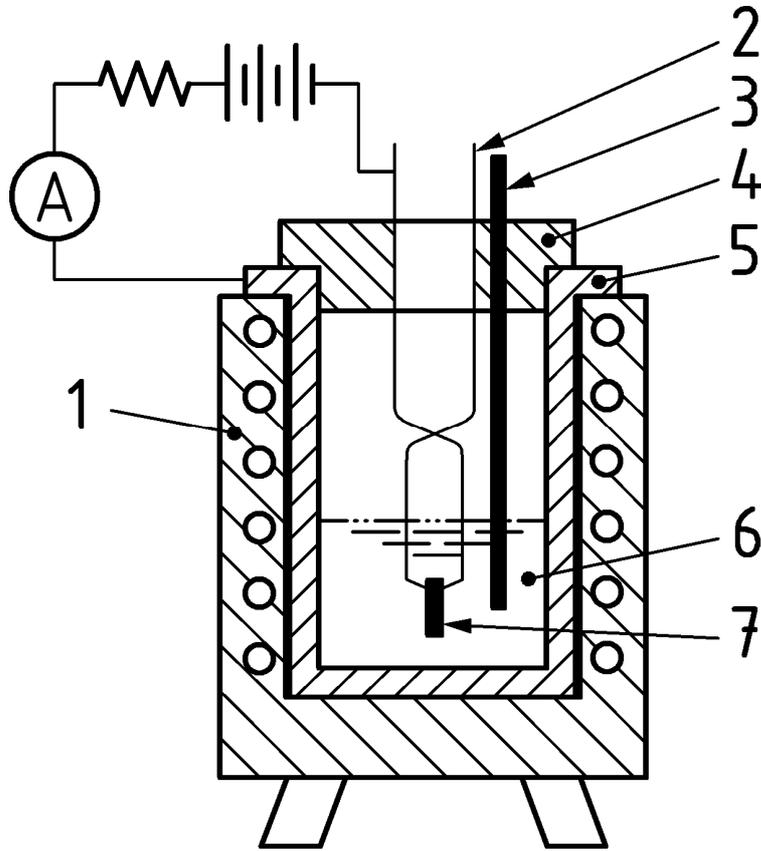
WARNING — When working with dangerous substances (such as sodium hydroxide, potassium permanganate, hydrochloric acid, and molten salts), all necessary safety precautions shall be taken.

Table A.A.A-1 — Chemical procedures for removal of corrosion products

Designation	Material	Chemical agents	Total time	Temperature	Remarks
C.1.1	Heat-resistant stainless steels and superalloys	Step 1: 180 g of sodium hydroxide (NaOH) 30 g of potassium permanganate (KMnO ₄) Distilled water to make 1 000 ml	30 min to 40 min	boiling	Repetition of step 1 / step-2 / washing is effective for sticky corrosion products
		Step 2: 100 g of ammonium citrate ((NH ₄) ₂ C ₆ H ₆ O ₇) Distilled water to make 1 000 ml	30 min to 40 min	boiling	Brushing by soft brush in the middle of treatment is effective
C.2.1	Carbon steels and low-alloy steels	50 ml of hydrochloric acid (HCl, $\zeta = 1,19$ g/ml) 0,5 g of hexamethylenetetramine or of 1,3-di- <i>n</i> -butyl-2-thiourea Distilled water to make 1 000 ml	30 min to 40 min	60 °C	Repetition of acid cleaning / washing is effective for sticky corrosion products

Table A.A.A-2 — Electrolytic procedures for removal of corrosion products

Designation	Material	Chemical agents	Total time	Temperature	Remarks
E.1.1	Heat-resistant stainless steels and superalloys	Molten salt containing mass fractions of 40 % of sodium hydroxide (NaOH) and 60 % of sodium carbonate (Na ₂ CO ₃)	2 min to 5 min	500 °C to 650 °C	Current density of 0,3 to 1,0 A/cm ² Repetition of electrolysis and washing is effective for sticky scale Brushing by soft brush in the middle of washing is effective Optimization of electrolysis is important



Key

- 1 Electric Furnace
- 2 Specimen holder
- 3 Thermocouple
- 4 Refractory Material
- 5 Electrolytic bath
- 6 Molten salt
- 7 Specimen

Figure A.1 – Typical equipment for the method E.1.1