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Corrosion of metals and alloys — Test method for thermal-cycling exposure testing under high-temperature corrosion conditions for metallic materials

Corrosion des métaux et alliages — Méthode pour essais de corrosion à haute température, avec exposition à des cycles thermiques, sur des matériaux métalliques

Reference number
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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.

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.

ISO 13573 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*, Working Group 13, *High Temperature Corrosion*.

Corrosion of metals and alloys — Test method for thermal-cycling exposure testing under high-temperature corrosion conditions for metallic materials

1 Scope

This International Standard describes the methodology for thermal cycling corrosion testing (known as cyclic oxidation testing) of metallic materials in gaseous environments between ambient and elevated temperatures (series of measurements on a single test piece with repeated, regular and controlled temperature cycles). It also may be applicable to other materials with some modifications. Tests with ultra short dwell times in the range of minutes or seconds are outside the scope of this International Standard.

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.

ANSI B74.12-92, *Specifications for the Size of Abrasive Grain – Grinding Wheels, Polishing and General Industrial Uses*

ASTM E1350-97, *Standard Test Methods for Testing Sheathed Thermocouples Prior to, During, and After Installation*

ASTM E220-02, *Standard Test Method for Calibration of Thermocouples By Comparison Techniques*

ASTM E230-03, *Standard Specification and Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples*

ASTM E3-01, *Standard Practice for Preparation of Metallographic Specimens*

ASTM E407-07e1, *Standard Practice for Microetching Metals and Alloys*

ASTM E633-00, *Standard Guide for Use of Thermocouples in Creep and Stress Rupture Testing to 1800°F (1000°C) in Air*

FEPA 43-1984 R:1993, *Grit Sizes for Coated Abrasives*

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

ISO 6344-3:1998, *Coated abrasives — Grain size analysis — Part 3: Determination of grain size distribution of microgrits P240 to P2500*

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

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

JIS R6001-87, *Bonded abrasive grain sizes*

3 Terms and definitions

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

3.1
scale
surface film and corrosion products produced on the surface of the test piece by high temperature corrosion

3.2
adherent scale
scale adhering to the test piece even after cooling

3.3
spalled scale
scale flaked from the test piece

3.4
delaminated scale
scale fully or partially detached from the surface but still in contact with the test piece

3.5
gross mass change
mass change of the test piece after cooling, including collected spalled scale

3.6
net mass change
mass change of the test piece after cooling, without including the mass of spalled scale

3.7
high temperature corrosion
corrosion occurring when the temperature is higher than the dew point of aqueous phases of the environment but at least 100 °C

3.8
breakaway
rapid increase in corrosion rate following a change from protective to non-protective scale growth

3.9
thermal cycle
sequence of temperatures that is repeated throughout the test. A single thermal cycle consists of the heating phase, the hot dwell time, the cooling time and the cold dwell time

4 Test method

4.1 Reagents and materials

4.1.1 Test pieces

The test pieces shall have the form of a rectangular plate, a disc or a cylinder with a surface area of 300 mm^2 at minimum and a thickness of 1,5 mm at minimum.

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

The test pieces shall be finished by machining so that the strata affected by cutting do not remain.

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

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

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 6344-3:1998, *Coated abrasives – Grain size analysis – Part 3: Determination of grain size distribution of microgrits P240 to P2500*	P1200	15,3 ± 1,0	Europe
JIS R6001-87, *Bonded abrasive grain sizes*	#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			

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

The surface of the test pieces shall not be deformed by marking, stamping or notching. Identification of the test pieces shall be solely on the basis of recording the relative position within the test chamber, however, holes for the test piece support (Figure 5) and or reference marking are permissible.

Where holes are used for the test piece support, they shall be drilled prior to final finishing or application of coatings. These have to be taken into account when calculating the surface area.

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 13385-1.

The test pieces shall be dried after degreasing by ultrasonic cleaning using iso-propanol or ethanol.

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 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.

It is recommend that duplicate test pieces are used each time.

4.1.2 Gas supply for closed system operation

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

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

The space between the humidifying regulator and the test piece 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 practicable to the inlet of the test piece chamber except where a humidifying regulator is used, in which case it shall be located upstream to the humidifier.

For testing in air, a specific humidity (mass fraction of water in air) of ~20 g/kg is recommended. This corresponds to a relative humidity of 100 % at 25 °C (dew point) and is easy to obtain by bubbling through a water bath of 25 °C.

If any other humidity is employed, it shall be agreed between the parties concerned.

In the case that the gas is humidified the water vapor content shall be measured. For example, this can be achieved by the use of a hygrometer before the test piece 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.

The formation of condensed phases from the test gas during the cooling cycle shall be avoided. This may be achieved by turning off the humidification or by switching to an inert gas.

4.2 Test apparatus

4.2.1 Design of apparatus

The apparatus shall be comprised of a set-up that will transition the test pieces between hot and cold environments in a controllable and reproducible manner. Ideally, the heating device should be equipped with a testing portion capable of separating the test piece from outside air (this assembly is referred to as a closed system) unless this is impracticable for the tests planned. When applicable, a humidifying regulator should be used to continuously supply the gas kept at a constant humidity which should be monitored with a hygrometer. The gas supply shall be controlled by a gas flow meter. A facility to accelerate cooling may also be included. Examples of basic designs are shown in Figures 1 and 2.

The heating device shall be constructed such that the test piece chamber is isolated from the external environment. It shall be ensured that a continuous gas flow within the prescribed range passes over the test pieces.

The test piece chamber shall not be composed of a material that reacts with the test atmosphere during the test to a degree that it changes the composition of the atmosphere.

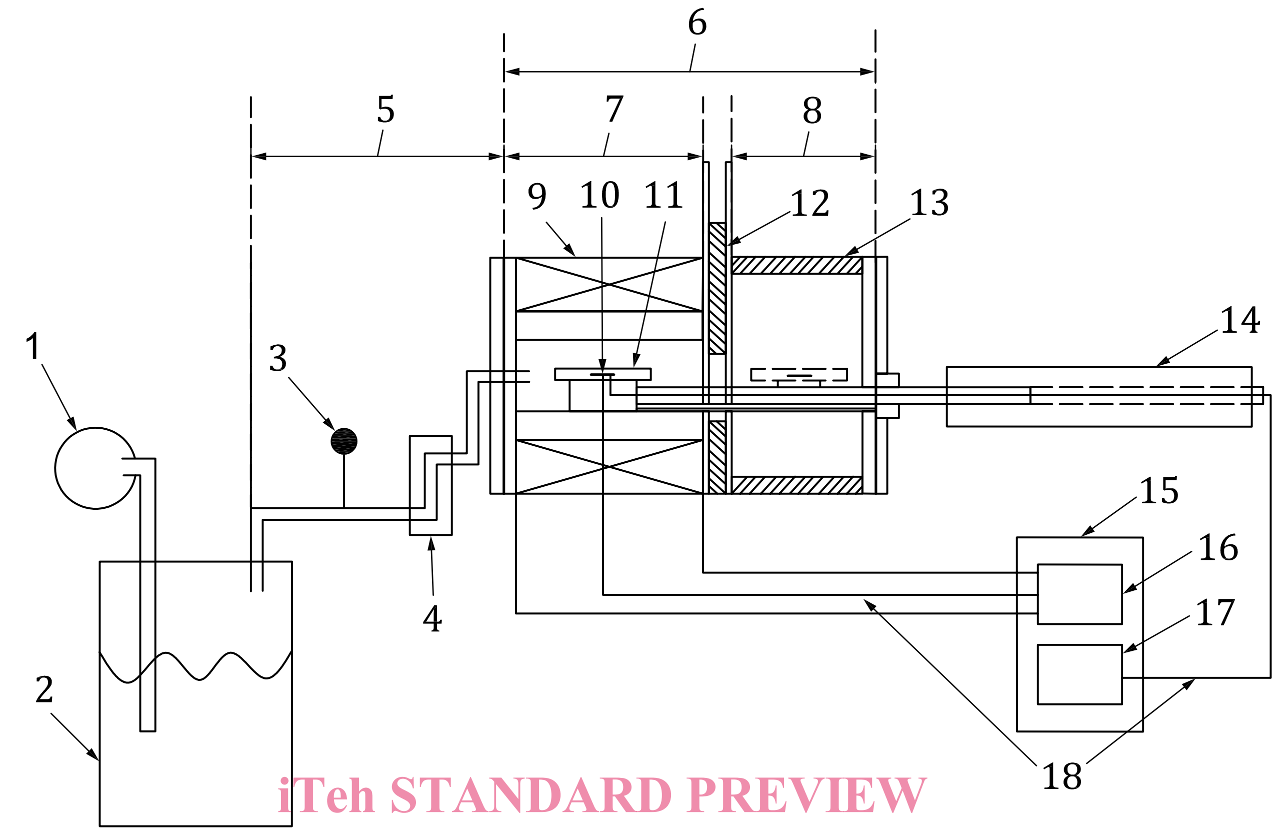
If a closed system with a test piece chamber cannot be used, then the tests may be performed in an open system with laboratory air. In this case the humidity of the air shall be recorded and the laboratories should be kept free from temperature changes and influences from weather conditions, as far as possible. Ideally, however, closed systems should be used.

The furnace shall be characterized at the exposure temperature prior to the testing to determine the length of the isothermal zone inside the furnace. A common method is by the use of an independent moveable thermocouple.

The time-dependent temperature response during the thermal cycling at a position at, or near to, the test piece shall be recorded prior to the testing in order to allow definition of the parameters of a thermal cycle, according to 4.3.3. This can be achieved by using dummy test pieces and appropriate thermometry.

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. The temperature of the furnace may vary or fluctuate due to movement of the furnace (less pronounced when the test piece supports are moved). The control system used shall ensure that the desired temperature inside the furnace is reached rapidly without being exceeded.

The heating device thermocouples for temperature control shall be as follows: The material for thermocouple shall fully withstand the test temperature. Moreover, the diameter of wire is recommended to be as small as possible, within the limit where the thermoelectric power does not change in service.



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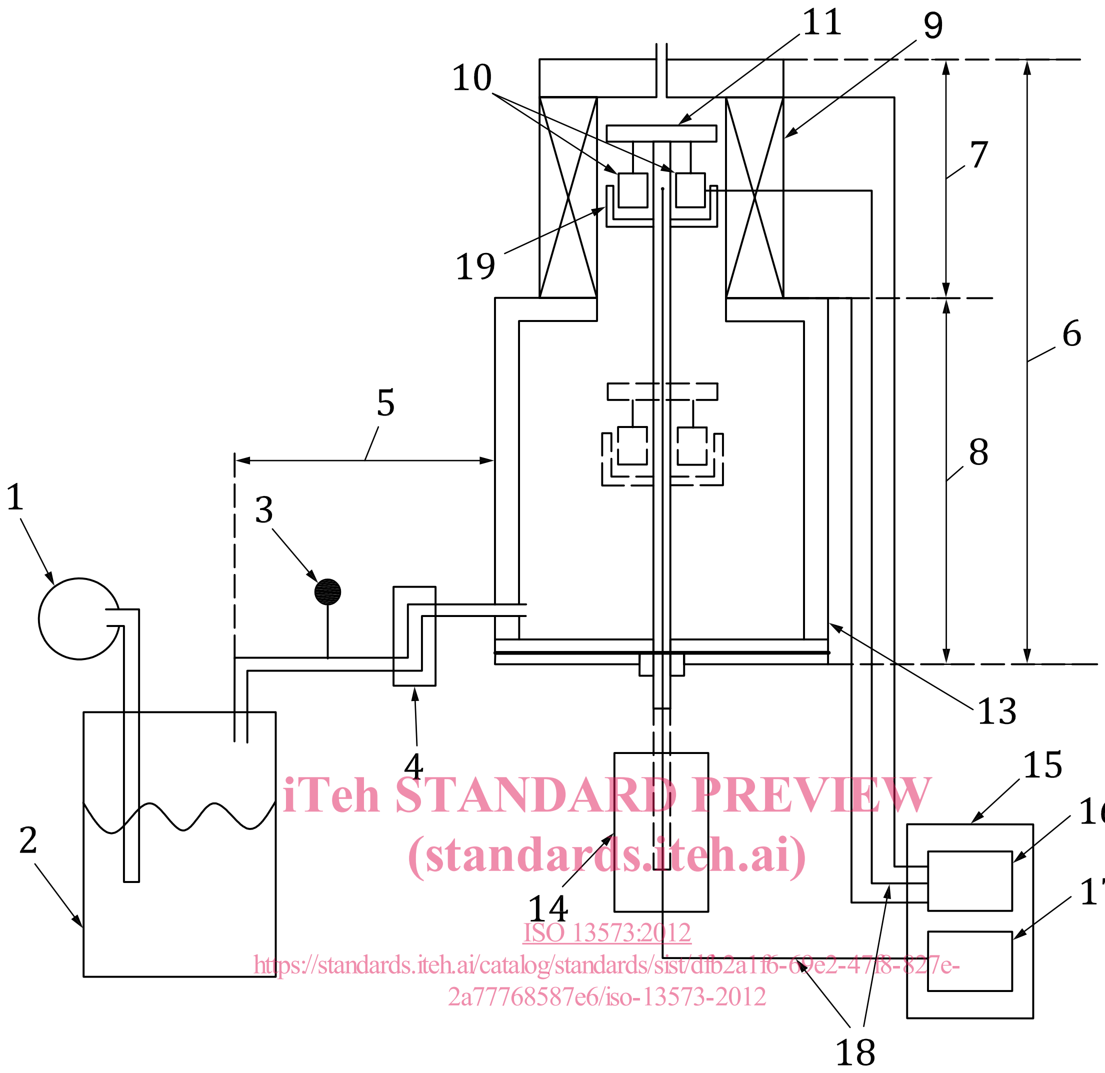
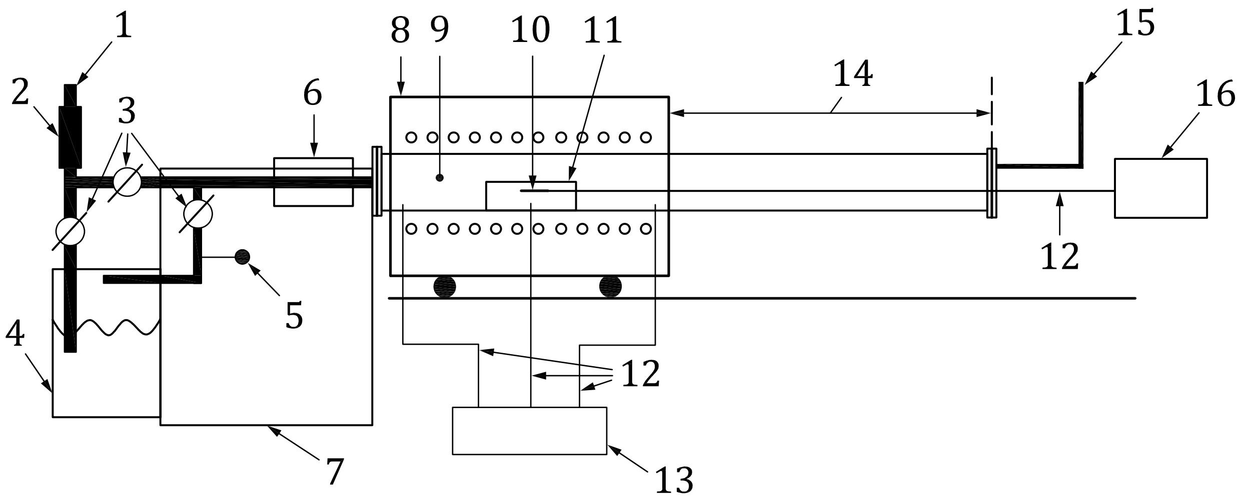


b)

Key

1 Blower
2 Humidifying regulator (electronic type)
3 Hygrometer
4 Air flow meter
5 Heating zone with ribbon heater
6 Testing portion
7 Heating zone
8 Cooling zone
9 Heating furnace
10 Test piece
11 Test piece support
12 Shielding plate (movable type)
13 Cooling chamber
14 Test piece carrier
15 Temperature regulating device
16 Power control device
17 Measuring instrument
18 Thermocouples
19 Crucible

Figure 1 — Basic design of a closed horizontal (a) and a vertical (b) apparatus, examples for setups with movable test piece support



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