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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

金属和合金的腐蚀 金属材料在静态浸入熔盐或其他液体条件下的高温腐蚀试验方法

(ISO 17245:2015, MOD)

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1 Scope

This document specifies a test method for evaluating the corrosion resistance of metallic materials when they are immersed in static molten salts or other liquids under static conditions and exposed to high-temperature controlled gas environments.

This document uses two types of controlled gas environments: stagnant inert gas environment and flowing reactive gas environment.

This document is applicable to high-temperature corrosion tests of metallic materials under static immersion conditions in molten salt or other liquids.

This document does not apply to situations where there is relative motion between the specimen and the surrounding corrosive liquid.

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.

GB/T 10123, *Corrosion of metals and alloys -- Vocabulary* (GB/T 10123-2022, ISO 8044:2020, IDT)

GB/T 16545-2015, *Corrosion of metals and alloys -- Removal of corrosion products from corrosion test specimens* (ISO 8407:2009, IDT)

GB/T 16701, *Methods for measuring the thermoelectric force of noble metal and base metal thermocouple wires*

GB/T 16839.1, *Thermocouples -- Part 1: EMF specifications and tolerances*

GB/T 41654, *Corrosion of metals and alloys - Method for metallographic examination of samples after exposure to high-temperature corrosive environments* (GB/T 41654-2022, ISO 26146:2012, IDT)

JJG 141, *Verification Regulation of Working Noble Metal Thermocouples*

3 Terms and definitions

For the purposes of this document, the terms and definitions defined in GB/T 10123 as well as the followings apply.

3.1 corrosive liquid

A powder mixture consisting of salts, ash and/or other solids that melts at the experimental temperature, or a liquid containing compounds that react with metals at high temperatures.

3.2 descaling

Before measuring the mass of the remaining metal, remove corrosion products and corrosion phases from the specimen surface.

4 Test method

4.1 Principle

The number of specimens for each material should be no less than 3, so as to ensure reproducibility of test results. These specimens can be used for measurement of mass changes, dimensional changes and/or observation of surface and/or cross-sectional changes.

4.2 Test materials

4.2.1 Specimen

The specimen shall be in the form of a rectangular plate, disc or cylinder. The surface area is not less than 300 mm^2. The thickness is not less than 1.5 mm.

If the specimen cannot be produced in accordance with the above specifications, other specimen shapes and sizes shall be determined by negotiation between the supplier and the purchaser.

The specimens shall be machined to remove the affected layer resulting from cutting.

The final surface of the specimen shall be polished using an abrasive with an average particle diameter of approximately 15 μm. P1200 abrasives that meet the requirements of GB/T 9258.3 can be used.

If the relevant parties require other surface grinding and polishing methods, the surface grinding and polishing conditions should be described.

The dimensions of the specimen should be measured at at-least 3 locations on the specimen before the exposure test. The accuracy of the measuring instrument is ±0.02 mm.

Specimens that have been ultrasonically cleaned in isopropyl alcohol or ethanol should be dried in hot air or in a desiccator.

If the specimen absorbs a large amount of water vapor in the environment, it is recommended that the cleaned specimen be stored in a desiccator before weighing and exposure testing.

The mass of the specimen should be measured prior to exposure. At least two measurements shall be made on each specimen. The mass difference between the two weighings should not exceed 0.05 mg.

4.2.2 Corrosive substances

WARNING: When working with hazardous materials such as certain ashes and molten salts, all necessary safety measures should be taken.

The type of corrosive substance should be selected according to the test environment. Corrosive substances should be collected from sediments in actual equipment or formulated with reagent-grade chemicals.

Corrosive substances should be mixed evenly. If the melting range of the substance is not known, it should be measured in advance or determined based on the phase diagram to ensure that the liquid phase is the main compound of the corrosive substance.

4.3 Test device

4.3.1 Device design

The test device shall include a temperature regulating device capable of uniformly heating the specimen at a constant temperature. The heating device should be equipped with a test unit (closed system) that isolates the specimen from the outside air.

A schematic diagram of a horizontal closed test device is shown in Figure 1. Devices placed vertically can also be used. The complete heating furnace device design is shown in Figure 1 of GB/T 38430-2019.

sample chamber should be maintained above the dew point. Condensation can have serious effects on test results.

Gas flow meters should be used to monitor gas flow. The flow meter should be placed close to the entrance to the sample chamber, but when a humidifier is used, the flow meter should be placed in front of the humidistat.

After the gas has been humidified, the water vapor content should be measured. Measurement methods include measuring with a hygrometer in front of the sample chamber, or measuring the water content after the exhaust gas is condensed, or measuring the water consumption in the humidifier during the test, etc.

4.3.4 Temperature monitoring

Before the test, a movable thermocouple should be used to determine the temperature distribution within the heating device at the test temperature, as well as the width of the isothermal zone.

The temperature regulating device should be able to ensure that the temperature of the sample is maintained within the allowable range shown in Table 1.

Thermocouple wires should be protected with thermowells. Thermowells shall be capable of use at the test temperature and test environment.

The thermocouple should be placed close to the specimen. If the test conditions do not allow the use of thermocouples, the furnace should be calibrated with simulated specimens under appropriate circumstances. Calculate the temperature of the specimen.

The calibration of thermocouples should be carried out in accordance with the provisions of GB/T 16701, GB/T 16839.1 or JJG 141. One thermocouple should be selected from each batch for calibration.

If the stability of the thermocouple cannot be determined, it should be recalibrated annually or before and at the end of each test.

4.4 Program

4.4.1 Specimen preparation and placement

A crucible that is inert relative to the test environment should be used.

The test gas should be preheated to allow the gas phase temperature to reach equilibrium. When necessary, appropriate catalysts should be used.

4.4.3 Heating method

The specimen should be placed in the heating furnace at room temperature before the test gas is heated. The time it takes for the heating furnace to reach the test temperature should be recorded.

For safety reasons, if the test gas cannot be heated, a simulated gas that does not contain hazardous components should be heated until the specified temperature is reached.

It should be heated as soon as possible until the test temperature reaches 30°C lower than the melting temperature of corrosive substances. After that, it should be heated slowly (such as 2°C/min) to a temperature of 30°C above the melting temperature of the corrosive substance. It can then be heated to quickly reach the test temperature.

NOTE: Rapidly heating elements such as heating elements and reaction tubes cannot be used in some devices because rapid heating rates can cause damage to ceramic components.

When testing in a humidified environment, humidification should be performed only after the temperature of all parts of the sample chamber exceeds the dew point temperature.

The heating process should ensure that the temperature of the specimen does not exceed the upper limit of the allowable temperature listed in Table 1.

4.4.4 Test period

When the specimen temperature exceeds 97% of the set test temperature T_{dwell} (unit: K), it is defined as the start of the test.

When the specimen temperature is lower than 97% of the set test temperature T_{dwell} (unit: K), the test is defined as terminated.

The test period is related to the intended application and should be determined by negotiation between the supplier and the purchaser. Since phenomena observed in short-term exposure may not continue to occur in long-term tests, the exposure time for tests related to long-term service conditions should be no less than 300 h.

4.4.5 Specimen cooling

Safety measures: When handling hot corrosive liquids, all necessary health and safety requirements should be followed.

If necessary, the specimen can be cooled in the heating furnace. The corrosive liquid can also be poured out of the crucible and allowed to cool. In the latter case, the specimen should be thoroughly purged with dry gas before removing it from the furnace to avoid any harmful reactions with air at high temperatures. If the specimen is being cooled in the heating furnace, ventilation should be continued to lower the temperature to below 200°C. Record the time it takes for the specimen to cool to 50°C. If it is a humidified environment, humidification should be stopped before the temperature of the coldest part of the sample chamber drops below 200°C.

After cooling, the solidified material attached to the surface of the specimen should be removed using a solvent. Normally, concentrated hydrochloric acid can be used.

For safety reasons, if cooling in the test gas is not possible, cooling should be performed in a simulated gas that does not contain hazardous components.

4.5 Determination of mass changes

4.5.1 Principle

To determine the oxidation kinetics of specimen corrosion, the changes in specimen mass need to be measured. Parallel specimens should be used for each material. Specimens should be weighed as soon as possible, or should be stored in a desiccator immediately after removal from the oven until before weighing. Use forceps to handle the specimen. Samples should not be touched directly with hands to avoid contamination (grease, salt). Care should be taken when using gloves as contamination from isolates on the gloves may lead to errors in mass determination. If repeated measurements are inconsistent, record the temperature and relative humidity of the weighing environment.

4.5.2 Measurement before testing

The mass of the specimen [$m_T(t_0)$, as shown in Figure 5 of GB/T 38430-2019] should be measured before exposure. Each specimen shall be measured at least twice. The accuracy should be no less than 0.02 mg. The maximum difference between each measurement result should not exceed ±0.05 mg.

4.5.3 Descaling before mass measurement

If descaling is prior to mass determination, all corrosion products should be removed from the specimen with minimal loss of base metal. This process should be carried out in accordance with GB/T 16545-2015.

The detailed process is shown in Annex B (see Table B.1 and Table B.2).

After descaling, the specimens should not be used for further corrosion test.

These procedures are not suitable for the removal of internal corrosion products defined in GB/T 41654.

4.5.4 Determination of mass loss due to corrosion

b) Characteristic heating and cooling curves of the specimen in corrosive substances;

c) Test cycle;

d) Sample chamber volume;

e) Stationary inert gas environment (4.3.2) or flowing active gas environment (4.3.3);

f) Composition of the test gas, including humidity;

g) Volume flow rate of test gas (m^3/s);

h) Chemical composition, melting point, moisture, preparation method and source of corrosive substances;

i) The amount of corrosive substance used in each crucible;

j) Size, type and material of crucible used.

6.1.4 Test results include the following data:

a) After descaling, the mass loss of the specimen;

b) After the corrosion test, the mass loss of the specimen;

c) In determining the corrosion and oxidation kinetics of the specimen, divide the specimen mass loss Δ_m by the surface area A as a function of time;

d) Appearance image after test;

e) Cross-sectional image after the test, including the surface layer of the metallographic cross-section of the specimen after the test (the selected magnification should be able to clearly show the extent of corrosion on a single micrograph);

f) Metallographic test results;

g) If testing is performed in accordance with 4.2.2, record the results of corrosive substance analysis after long-term exposure.

6.2 Additional information

The test results report includes the following items:

a) Mechanical properties of raw materials;

b) Microstructure and sampling conditions of raw materials;

c) Details of the test device;

Annex B

(informative)

Chemical and electrolytic procedures for removal of corrosion products

B.1 General conditions

In the development of this appendix, extensive literature is consulted to determine procedures for chemical and electrolytic descaling. In addition to the test methods listed in GB/T 16545-2015, the following procedures specifically designed for the test purposes described in this document can be used. This appendix summarizes the findings of the survey.

Before adopting these procedures, blank specimens should be tested to ensure the validity of the chosen method. Excessive rust removal will not only lead to the dissolution of corrosion products, but also the dissolution of the metal matrix.

B.2 Procedures

WARNING: All necessary safety measures should be taken when working with hazardous substances (such as sodium hydroxide, potassium permanganate, hydrochloric acid).

Tables B.1 and B.2 summarize various chemical and electrolytic procedures for the removal of corrosion products. The specific process selection for a particular material depends on many factors, including previous experience and the various treatments the specimen has undergone previously.

For all procedures listed, it is advisable to keep the specimen surface vertical during cleaning. This will minimize the release of any gases during the descaling process.

The time recommended in Figure 1 of GB/T 16545-2015 is suitable for the test of mass loss.

When using electrolysis procedures, it is recommended to select appropriate electrolysis parameters and use blank specimens to ensure accurate removal of corrosion products. A typical setup is shown in Figure B.1.

------ The End ------