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Corrosion of metals and alloys — Electrochemical test methods — Guideline for electrochemical measurements in high temperature molten salts

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Foreword

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This document was prepared by Technical Committee ISO/TC 156, Corrosion of metal and alloys.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Corrosion of metals and alloys in high temperature molten salts is generally an electrochemical phenomenon. Therefore, one can evaluate the corrosion resistance and corrosion mechanism by a variety of electrochemical techniques. This international standard describes the apparatus and procedures for electrochemical measurements in high temperature molten salts. The closely related guidelines for potentiostatic and potentiodynamic polarization measurement in aqueous solutions are described in ISO 17475.

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Corrosion of metals and alloys — Electrochemical test methods — Guideline for electrochemical measurements in high temperature molten salts

1 Scope

This international standard describes the general procedure of electrochemical measurements such as potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) in high temperature molten salts with the apparatus.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

ISO 8044:1999, *Corrosion of metals and alloys - Basic terms and definitions*

ISO 17474, *Corrosion of metals and alloys — Conventions applicable to electrochemical measurements in corrosion testing*

ISO 17475, *Corrosion of metals and alloys — Electrochemical test methods — Guidelines for conducting potentiostatic and potentiodynamic polarization measurements*

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 under static conditions*

3 Terms and definitions

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

4 Apparatus

The test apparatus should consist of heating unit, gas feeding unit, electrochemical measurement unit, and electrochemical cell. A basic design of a closed, vertical, apparatus is shown in [Figure 1](#).

4.1 Heating unit

4.1.1 The apparatus shall be composed of a heating unit heating all the test pieces at a uniform temperature. The heating unit shall be equipped with a testing portion capable of separating the test pieces from outside air.

4.1.2 The temperature distribution of the heating unit shall be characterized at the exposure temperature prior to the testing to determine the width of the isothermal zone by the use of a calibrated movable thermocouple. The heating unit or furnace shall guarantee that the temperature of the electrochemical cell is kept within the permissible range given in [Table 1](#).

4.1.3 Calibration of thermocouples shall be performed in accordance with ASTM E220, ASTM E230, and ASTM E1350. A representative thermocouple taken from a 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 the thermocouple stability.

Table 1 — 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

4.2 Gas feeding unit

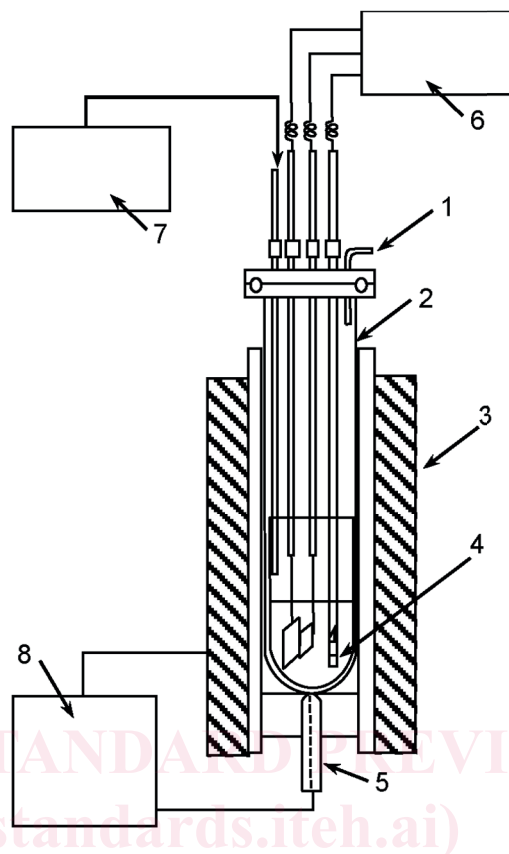
4.2.1 The gas supply system shall be capable of supplying the test gases at a constant flow rate to the test piece chamber described in Figure 1. The gas flow shall be monitored by a gas flow meter.

4.2.2 When a humidifying regulator is used, it shall be capable of adjusting to the desired humidity. The gas line between humidifying regulator and test chamber shall be kept above the dew point in order to avoid condensation. 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.

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Key

- 1 Gas exhaust
- 2 Test piece chamber
- 3 Furnace
- 4 Electrochemical cell
- 5 Thermocouple
- 6 Electrochemical measurement unit
- 7 Gas feeding unit
- 8 Power/temperature control device

Figure 1 — Schematic illustration of whole setup for electrochemical measurement unit in high temperature molten salt

4.3 Electrochemical measurement unit

4.3.1 The potentiostat should be capable of controlling the electrode potential in the test to within $\pm 1\text{mV}$ of a preset value. A scanning potentiostat is used for potentiodynamic measurements. For such measurement, the potentiostat shall be capable of automatically scanning the potential at a constant rate between preset potentials. For the EIS measurement, a potentiostat equipped with an electrochemical impedance measurement unit is used.

4.3.2 The electrode potential-measuring instruments should have a high input impedance of the order of $10^{11}\ \Omega$ to $10^{14}\ \Omega$, to minimize the current drawn from the system during measurement. The sensitivity and accuracy of the instrument should be sufficient to detect a change of $1.0\ \text{mV}$.

4.3.3 Use appropriate current-measuring instruments with maximum error 0.5 %.

4.4 Electrochemical cell

4.4.1 The three-electrode system consisting of a working electrode (the metal to be polarized), a reference electrode for measuring the electrode potential, and a counter (auxiliary) electrode shall be used. The test cell should incorporate inlet and outlet gas ports and a port for insertion of a temperature-measuring device. For the EIS measurement, the two-electrode system consisting of two identical working electrodes may be used instead. Examples of the three-electrode and two-electrode cells are shown in [Figure 2\(a\) and \(b\)](#), respectively.

4.4.2 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. The crucibles used to contain the molten salt should not react with salt; for example, a high purity alumina crucible shall be used for sulfate or chloride bath.

4.4.3 The working electrode is prepared from the test material of interest, usually in the form of a rod or sheet. The working electrode surface other than test area shall be protected by an insulating coating that does not react with the test salt nor gas. The area immersed below the molten salt/gas interface shall be considered as the effective electrode surface area, if an appropriate insulating coating is not identified. In order to minimize the influence of the electrochemical reaction that proceeds at the three-phase interface of gas/electrode/molten salt, the shape of the working electrode is recommended to be flag-shaped. The flag-shaped electrode can be manufactured by punching or wire electric discharge machining. An example of the flag-shaped electrode is shown in Figure 3. The level of the molten salt should not drop below the upper edge of the 'flag' section of the sample. 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 2. If another surface finish is required by the parties involved, the surface finish condition shall be agreed and described. After ultrasonically degreasing in an appropriate solvent such as isopropanol or ethanol etc., the test pieces shall be dried in hot air.

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Table 2 — 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	15,6 ± 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			

4.4.4 The reference electrode may be inserted directly into the molten salt. The type of reference electrode used will depend on the application, e.g. molten salt and temperature. An example of the reference electrode for molten chlorides, molten sulfates and molten carbonates is given in [Annex B](#).

4.4.5 The counter electrode should be sufficiently corrosion resistant in the test environment. If the reaction at the counter electrode is likely to influence the measurement, the counter electrode shall be separated in a compartment.

NOTE Pt, Au or carbon may be used as a counter electrode.