
Method of measurement of hydrogen permeation and determination of hydrogen uptake and transport in metals by an electrochemical technique

Méthode de mesure de la perméation de l'hydrogène et détermination de l'absorption d'hydrogène et de son transport dans les métaux à l'aide d'une technique électrochimique

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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).

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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*.

This second edition cancels and replaces the first edition (ISO 17081:2004), of which it constitutes a minor revision. [Figure 1](#) has been corrected and [Figure 2](#) made language independent.

Method of measurement of hydrogen permeation and determination of hydrogen uptake and transport in metals by an electrochemical technique

1 Scope

1.1 This International Standard specifies a laboratory method for the measurement of hydrogen permeation and for the determination of hydrogen atom uptake and transport in metals, using an electrochemical technique. The term “metal” as used in this International Standard includes alloys.

1.2 This International Standard describes a method for evaluating hydrogen uptake in metals, based on measurement of steady-state hydrogen flux. It also describes a method for determining effective diffusivity of hydrogen atoms in a metal and for distinguishing reversible and irreversible trapping.

1.3 This International Standard gives requirements for the preparation of specimens, control and monitoring of the environmental variables, test procedures and analysis of results.

1.4 This International Standard may be applied, in principle, to all metals for which hydrogen permeation is measurable and the method can be used to rank the relative aggressivity of different environments in terms of the hydrogen uptake of the exposed metal.

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

3 Terms and definitions

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

3.1

charging

method of introducing atomic hydrogen into the metal by exposure to an aqueous environment under galvanostatic control (constant charging current), potentiostatic control (constant electrode potential), free corrosion or by gaseous exposure

3.2

charging cell

compartment in which hydrogen atoms are generated on the sample surface, including both aqueous and gaseous charging

3.3

decay current

decay of the hydrogen atom oxidation current, after attainment of steady state, following a decrease in charging current

3.4

Fick's second law

second-order differential equation describing, in this case, the concentration of atomic hydrogen in the sample as a function of position and time

Note 1 to entry: The equation is of the form $\partial C(x, t)/t = D \partial^2 C(x, t)/\partial x^2$ for lattice diffusion in one dimension where diffusivity is independent of concentration. See [Table 1](#) for an explanation of the symbols.

3.5

hydrogen flux

amount of hydrogen passing through the metal sample per unit area per unit time

3.6

hydrogen uptake

atomic hydrogen absorbed into the metal as a result of charging

3.7

irreversible trap

microstructural site at which the residence time for a hydrogen atom is infinite or extremely long compared to the time-scale for permeation testing at the relevant temperature

3.8

mobile hydrogen atoms

hydrogen atoms in interstitial sites in the lattice (lattice sites) and reversible trap sites

3.9

oxidation cell

compartment in which hydrogen atoms exiting from the metal sample are oxidized

3.10

permeation current

current measured in oxidation cell associated with oxidation of hydrogen atoms

3.11

permeation flux

hydrogen flux exiting the test sample in the oxidation cell

3.12

permeation transient

variation of the permeation current with time, from commencement of charging to the attainment of steady state, or modification of charging conditions

3.13

recombination poison

chemical within the test environment in the charging cell which enhances hydrogen absorption by retarding the recombination of hydrogen atoms on the metal surface

3.14

reversible trap

microstructural site at which the residence time for a hydrogen atom is greater than that for the lattice site but is small in relation to the time to attain steady-state permeation

4 Symbols

[Table 1](#) gives a list of symbols and their designations.

Table 1 — Symbols and their designations and units

Symbol	Designation	Unit
A	Exposed area of sample in the oxidation cell	m^2
$C(x, t)$	Lattice concentration of hydrogen as a function of position and time	$\text{mol} \cdot \text{m}^{-3}$
C_0	Sub-surface concentration of atomic hydrogen in interstitial lattice sites on the charging side of the sample	$\text{mol} \cdot \text{m}^{-3}$
C_{0R}	Summation of the sub-surface concentration of hydrogen in interstitial lattice sites and reversible trap sites on the charging side of the sample	$\text{mol} \cdot \text{m}^{-3}$
D_l	Lattice diffusion coefficient of atomic hydrogen	$\text{m}^2 \cdot \text{s}^{-1}$
D_{eff}	Effective diffusion coefficient of atomic hydrogen based on elapsed time corresponding to $J(t)/J_{ss} = 0,63$	$\text{m}^2 \cdot \text{s}^{-1}$
F	Faraday's constant ($F = 96\,485 \text{ C} \cdot \text{mol}^{-1}$)	$\text{C} \cdot \text{mol}^{-1}$
$J(t)$	Time-dependent atomic hydrogen permeation flux as measured on the oxidation side of the sample	$\text{mol} \cdot \text{m}^{-2} \text{ s}^{-1}$
J_{ss}	Atomic hydrogen permeation flux at steady-state as measured on the oxidation side of the sample	$\text{mol} \cdot \text{m}^{-2} \text{ s}^{-1}$
$J(t)/J_{ss}$	Normalized flux of atomic hydrogen	1
$I(t)$	Time-dependent atomic hydrogen permeation current	$\text{A} \cdot \text{m}^{-2}$
I_{ss}	Steady-state atomic hydrogen permeation current	$\text{A} \cdot \text{m}^{-2}$
L	Sample thickness	m
t	Time elapsed from commencement of hydrogen charging	s
t_b	Elapsed time measured by extrapolating the linear portion of the rising permeation current transient	s
t_{lag}	Time to achieve a value of $J(t)/J_{ss} = 0,63$	s
x	Distance in sample measured in the thickness direction	m
τ	Normalized time ($D_l t/L^2$)	1
τ_{lag}	Normalized time to achieve a value of $J(t)/J_{ss} = 0,63$	1

5 Principle

5.1 The technique involves locating the metal sample of interest between the charging and oxidation cells, where the charging cell contains the environment of interest. Hydrogen atoms are generated on the sample surface exposed to this environment.

5.2 In gaseous environments, the hydrogen atoms are generated by adsorption and dissociation of the gaseous species. In aqueous environments, hydrogen atoms are produced by electrochemical reactions. In both cases, some of the hydrogen atoms diffuse through the metal sample and are then oxidized to hydrogen cations on exiting from the other side of the metal in the oxidation cell.

A palladium coating is sometimes applied to one or both sides of the membrane following initial removal of oxide films. A palladium coating on the charging face of the membrane affects the sub-surface hydrogen concentration in the substrate and the measured permeation current. It is important to verify that the calculated diffusivity is not influenced by the coating. Palladium coating is particularly useful for gaseous charging.

5.3 The environment and the electrode potential on the oxidation side of the membrane are selected so that the metal is either passive or immune to corrosion. The background current established prior to hydrogen transport is steady, and small compared to the hydrogen atom oxidation current.