Designation: G 148 – 97

Standard Practice for
Evaluation of Hydrogen Uptake, Permeation, and Transport in Metals by an Electrochemical Technique[1]

This standard is issued under the fixed designation G 148; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice gives a procedure for the evaluation of hydrogen uptake, permeation, and transport in metals using an electrochemical technique which was developed by Devanathan and Stachurski.[2] While this practice is primarily intended for laboratory use, such measurements have been conducted in field or plant applications. Therefore, with proper adaptations, this practice can also be applied to such situations.

1.2 This practice describes calculation of an effective diffusivity of hydrogen atoms in a metal and for distinguishing reversible and irreversible trapping.

1.3 This practice specifies the method for evaluating hydrogen uptake in metals based on the steady-state hydrogen flux.

1.4 This practice gives guidance on preparation of specimens, control and monitoring of the environmental variables, test procedures, and possible analyses of results.

1.5 This practice can be applied in principle to all metals and alloys which have a high solubility for hydrogen, and for which the hydrogen permeation is measurable. This method can be used to rank the relative aggressivity of different environments in terms of the hydrogen uptake of the exposed metal.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*
G 96 Guide for Online Monitoring of Corrosion in Plant Equipment (Electrical and Electrochemical Methods)[3]

3. Terminology

3.1 *Definitions:*
3.1.1 *charging,*, *n*—method of introducing atomic hydrogen into the metal by galvanostatic charging (constant charging current), potentiostatic charging (constant electrode potential), free corrosion, or gaseous exposure.

3.1.2 *charging cell,*, *n*—compartment in which hydrogen atoms are generated on the specimen surface. This includes both aqueous and gaseous charging.

3.1.3 *decay current,*, *n*—decay of the hydrogen atom oxidation current due to a decrease in charging current.

3.1.4 *Fick's second law,*, *n*—second order differential equation describing the concentration of diffusing specie as a function of position and time. The equation is of the form $\partial C(x,t)/\partial t = \partial/\partial x D_1 \partial/\partial x [C(x,t)]$ for lattice diffusion in one dimension where diffusivity is independent of concentration. See 3.2 for symbols.

3.1.5 *hydrogen flux,*, *n*—the amount of hydrogen passing through the metal specimen per unit area as a function of time. The units are typically concentration per unit area per unit time.

3.1.6 *hydrogen uptake,*, *n*—the concentration of hydrogen absorbed into the metal (for example, g/cm^3 or mol/cm^3).

3.1.7 *irreversible trap,*, *n*—microstructural site at which a hydrogen atom has a infinite or extremely long residence time compared to the time-scale for permeation testing at the relevant temperature, as a result of a binding energy which is large relative to the migration energy for diffusion.

3.1.8 *reversible trap,*, *n*—microstructural site at which a hydrogen atom has a residence time which is greater than that for the lattice site but is small in relation to the time to attain steady-state permeation, as a result of low binding energy.

3.1.9 *mobile hydrogen atoms,*, *n*—hydrogen atoms that are associated with sites within the lattice.

3.1.10 *oxidation cell,*, *n*—compartment in which hydrogen atoms exiting from the metal specimen are oxidized.

3.1.11 *permeation current,*, *n*—current measured in oxidation cell associated with oxidation of hydrogen atoms.

3.1.12 *permeation transient,*, *n*—the increase of the permeation current with time from commencement of charging to the attainment of steady state, or modification of charging conditions (that is, rise transient). The decrease of the permeation current with time resulting from a decrease in charging current (that is, decay transient).

3.1.13 *recombination poison*, *n*—chemical specie present

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within the test environment in the charging cell which enhances hydrogen absorption by retarding the recombination of hydrogen atoms adsorbed onto the metal surface into hydrogen gas.

3.2 *Symbols*:

3.2.1 For the purposes of this practice the following symbols apply:

A	= exposed area of specimen in the oxidation cell (cm^2)
$C(x,t)$	= lattice concentration of hydrogen as a function of position and time (mol/cm^3)
C_O	= sub-surface concentration of atomic hydrogen at the charging side of the specimen (mol/cm^3)
D_{eff}	= effective diffusivity of atomic hydrogen, taking into account the presence of reversible and irreversible trapping (cm^2/s)
D_l	= lattice diffusion coefficient of atomic hydrogen (cm^2/s)
F	= faraday's constant (9.6485×10^4 coulombs/mol)
$I(t)$	= time dependent atomic hydrogen permeation current (μA)
I_{ss}	= steady-state atomic hydrogen permeation current (μA)
$J(t)$	= time-dependent atomic hydrogen permeation flux as measured on the oxidation side of the specimen ($mol/s/cm^2$)
J_{ss}	= atomic hydrogen permeation flux at steady-state ($mol/s/cm^2$)
$J(t)/J_{ss}$	= normalized flux of atomic hydrogen
L	= specimen thickness (cm)
t	= time elapsed from commencement of hydrogen charging (s)
t_b	= elapsed time measured extrapolating the linear portion of the rising permeation current transient to $J(t) = O$ (s)
t_{lag}	= time to achieve a value of $J(t)/J_{ss} = 0.63$ (s)
x	= distance into specimen from the charging surface measured in the thickness direction (cm^2).
τ	= normalized time ($D_1 t/L^2$)
τ_{lag}	= Normalized time to achieve a value of $j(t)/J_{ss} = 0.63$ (s)

4. Summary of Practice

4.1 The technique involves locating the metal membrane (that is, specimen) of interest between the hydrogen charging and oxidation cells. In the laboratory, the charging cell contains the environment of interest. Hydrogen atoms are generated on the membrane surface exposed to this environment. In field or plant measurements, the wall of the pipe or vessel can be used as the membrane through which measurement of hydrogen flux are made. The actual process environment is on the charging side of the membrane which eliminates the need for a charging cell. See 7.1 for guidance on various specimen configurations.

4.2 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 membrane and are then oxidized on

exiting from the other side of the metal in the oxidation cell.

4.3 The conditions (for example, environment and the electrode potential) on the oxidation side of the membrane are controlled so that the metal surface is either passive or immune to corrosion. The background current established under these conditions prior to hydrogen transport should be relatively constant and small compared to that of the hydrogen atom oxidation current.

4.4 The electrode potential of the specimen in the oxidation cell is controlled at a value sufficiently positive to ensure that the kinetics of oxidation of hydrogen atoms are limited by the flux of hydrogen atoms, that is, the oxidation current density is diffusion limited.

4.5 The total oxidation current is monitored as a function of time. The total oxidation current comprises the background current and the current resulting from oxidation of hydrogen atoms. The latter is the permeation current.

4.6 The thickness of the specimen is selected usually to ensure that the measured flux reflects volume (bulk) controlled hydrogen atom transport. Thin specimens may be used for evaluation of the effect of surface processes on hydrogen entry or exit (absorption kinetics or transport in oxide films).

4.7 In reasonably pure, defect-free metals (for example, single crystals) with a sufficiently low density of microstructural trap sites, atomic hydrogen transport through the material is controlled by lattice diffusion.

4.8 Alloying and microstructural features such as dislocations, grain boundaries, inclusions, and precipitate particles may act as trap sites for hydrogen thus delaying hydrogen transport. These traps may be reversible or irreversible depending on the binding energy associated with the particular trap sites compared to the energy associated with migration for hydrogen in the metal lattice.

4.9 The rate of hydrogen atom transport through the metal during the first permeation may be affected by both irreversible and reversible trapping as well as by the reduction of any oxides present on the charging surface. At steady state all of the irreversible traps are occupied. If the mobile hydrogen atoms are then removed and a subsequent permeation test conducted on the specimen the difference between the first and second permeation transients can be used to evaluate the influence of irreversible trapping on transport, assuming a negligible role of oxide reduction.

4.10 For some environments, the conditions on the charging side of the specimen may be suitably altered to induce a decay of the oxidation current after attainment of steady state. The rate of decay will be determined by diffusion and reversible trapping only and, hence, can also be used to evaluate the effect of irreversible trapping on transport during the first transient.

4.11 Comparison of repeated permeation transients with those obtained for the pure metal can be used in principle to evaluate the effect of reversible trapping on atomic hydrogen transport.

4.12 This practice is suitable for systems in which hydrogen atoms are generated uniformly over the charging surface of the membrane. It is not usually applicable for evaluation of corroding systems in which pitting attack occurs unless the charging cell environment is designed to simulate the localized