INTERNATIONAL STANDARD

ISO 15724

First edition
2001-12-01

Metallic and other inorganic coatings — Electrochemical measurement of diffusible hydrogen in steels — Barnacle electrode method

Revêtements métalliques et autres revêtements inorganiques — Mesurage électrochimique de l'hydrogène diffusible dans les aciers — Méthode par électrode anatife

Reference number
ISO 15724:2001(E)

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.ch
Web www.iso.ch

Printed in Switzerland

Contents

Page

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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15724 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 2, *Test methods*.

Introduction

When atomic hydrogen enters steels and certain other alloys, it can cause loss of ductility or of load-carrying ability, cracking (usually submicroscopic cracks) or catastrophic brittle failure at applied stresses well below the yield strength or even the normal design strength of the alloys. This phenomenon often occurs in alloys that show no significant loss in ductility when measured by conventional tensile tests and is frequently referred to as “hydrogen-induced delayed brittle failure”, “hydrogen stress cracking” or “hydrogen embrittlement”. The hydrogen can be introduced during fabrication, cleaning, pickling, phosphating, electroplating, autocatalytic processes and in the service environment as a result of cathodic protection reactions or corrosion reactions. Parts that have been machined, ground, cold-formed or cold-straightened subsequent to any heat treatment are especially susceptible to hydrogen embrittlement damage.

Metallic and other inorganic coatings — Electrochemical measurement of diffusible hydrogen in steels — Barnacle electrode method

WARNING — This International Standard does not address any health hazards and safety matters associated with its use. It is the responsibility of the user of this International Standard to establish appropriate health and safety practices and take suitable actions for any national regulations.

1 Scope

This International Standard describes a method that uses an electronic hydrogen detection instrument for measuring relative, diffusible hydrogen concentrations in bare steels or in plated steels after the coating has been removed. It is assumed that the hydrogen is uniformly distributed throughout a part.

The method does not measure actual hydrogen concentration. However, oxidation current densities measured against time provide a useful indication of relative hydrogen concentrations and, therefore, the measurements can be used for comparison purposes. The method may be used as a quality control procedure as it does provide a quick means of measuring the effectiveness of pre- and/or post-plating heat treatments or of monitoring hydrogen uptake during plating or due to corrosion.

It is important to note that the absence of failure in a particular test does not provide confirmation of complete elimination of hydrogen embrittlement because no one test method can provide all the data necessary to evaluate the degree of hydrogen degradation.

For unplated parts the method is non-destructive; however, for plated parts the coating has to be removed prior to measurement by a means proven not to damage the steel or to introduce hydrogen.

This test method is limited to:

— carbon and alloy steels, excluding austenitic stainless steels (see note 1);

— flat specimens to which the cell can be attached (see note 2);

— measurements at room temperature (25 °C ± 1 °C).

NOTE 1 If this method is used for austenitic stainless steels and other face centred cubic (FCC) alloys, measurement times and interpretation of results will have to be determined because of the different kinetics involved.

NOTE 2 For slightly curved surfaces it is essential to define an area that is reproducible. The area calculation will be different from that described in this International Standard.

NOTE 3 The method can be applied to small parts, however, this necessitates some modification of the technique, procedure and interpretation of results.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For

undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 9587, *Metallic and other inorganic coatings — Pretreatments of iron or steel to reduce the risk of hydrogen embrittlement*

ISO 9588, *Metallic and other inorganic coatings — Post-coating treatments of iron or steel to reduce the risk of hydrogen embrittlement*

3 Principle

This method is an adaptation of the electrochemical permeation method for hydrogen diffusion.

A hydrogen-containing steel part is made the anode in an electrochemical cell containing an alkaline solution as the electrolyte, with a nickel-nickel oxide (Ni/NiO) electrode forming the cathode. The cell maintains zero hydrogen concentration at the surface of the steel by oxidizing the hydrogen atoms to water. As there is no corrosion reaction taking place at the steel surface, the anodic current measured as a function of time is attributed to the concentration of mobile hydrogen.

In this method, the actual hydrogen concentration is not determined. This is because the calculation of hydrogen concentration requires knowledge of the hydrogen diffusion coefficient in the alloy and knowledge of the decay curve, both of which are not always known, especially at very low hydrogen concentrations. However, comparative current densities can be used to indicate relative hydrogen concentrations.

In this method, it is assumed that the hydrogen is uniformly distributed throughout the steel part and that hydrogen is not appreciably lost due to diffusion during the course of the measurement. Furthermore, as the measurement is relatively rapid, it is probable that trapped hydrogen that is not easily released without stress but that could contribute to delayed failure, will not be detected.

This method is based on measurement of current that decays over a fixed period, in this case for 30 min. The current density will depend on the material as well as on the hydrogen concentration. Each material will have a background current density (see 7.4). A measurement higher than background indicates the presence of hydrogen. Higher current densities indicate higher hydrogen concentrations; the higher the current densities the higher the likelihood of hydrogen embrittlement. A background correction of the actual measurement, however, is not made (see note to 7.4).

4 Reagents

Reagent grade chemicals of sufficiently high purity to ensure the accuracy of the determination shall be used in all tests.

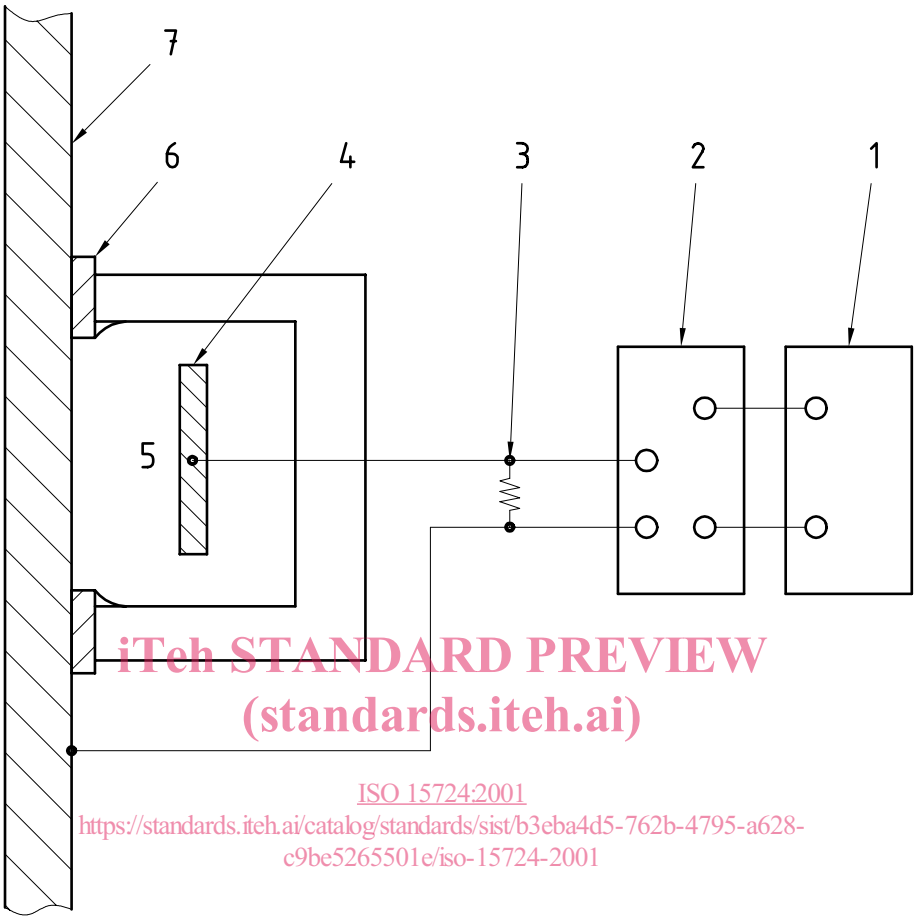
4.1 Electrolyte (0,2 M sodium hydroxide solution), dissolve 8 g of sodium hydroxide (NaOH) pellets in distilled or de-ionized water and dilute to 1 l.

4.2 Methyl alcohol (CH_3OH).

4.3 Ethyl alcohol (C_2H_5OH).

5 Apparatus

See Figures 1 and 2.



Key

1	Strip chart recorder
2	Electrometer
3	Known resistance
4	Driving electrode, Ni/NiO
5	0,2 M NaOH
6	Gasket
7	Hydrogen-containing part

Figure 1 — Schematic diagram of a barnacle electrode system

5.1 The barnacle electrode system.

This consists of

5.1.1 cell, left open to the atmosphere without any purging (see Figure 2), and made of a non-metallic inert material that will not react with or contaminate the electrolyte, and the side opening of which has a recess to accommodate a silicon rubber gasket.

5.1.2 gasket, made of silicon rubber, that shall provide a reproducible contact area between the solution and the specimen, preferably 1 cm^2.

5.1.3 cell holder, a cradle-like C-clamp.