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Steel — Measurement method for the evaluation of hydrogen embrittlement resistance of high-strength steels —

Part 2:
Slow stain rate test

Acier — Méthode de mesure pour l'évaluation de la résistance à la fragilisation par l'hydrogène des aciers à haute résistance — Partie 2: Essai de décoloration lente

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Foreword

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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).

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This document was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 7, *Methods of testing (other than mechanical tests and chemical analysis)*.

A list of all parts in the ISO 16573 series shall be found on the ISO website.

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

The mechanical properties of high-strength steels, such as tensile strength, elongation and reduction of area would be degraded by the effect of hydrogen, known as hydrogen embrittlement, and the susceptibility of hydrogen embrittlement becomes greater by increasing the strength level of steels.

Steel — Measurement method for the evaluation of hydrogen embrittlement resistance of high-strength steels —

Part 2:
Slow stain rate test

1 Scope

This document provides an evaluation method of the resistance of high-strength steels to hydrogen embrittlement (i.e. hydrogen delayed fracture) using slow strain rate test with hydrogen pre-charged specimens. The amount of hydrogen absorbed in the specimens is analysed quantitatively by thermal desorption analysis such as gas chromatography, mass spectrometry and so on. This document includes testing methods for either smooth or notched specimens.

It is applicable to ferritic base steels.

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 6892-1, *Metallic materials — Tensile testing — Part 1: Method of test at room temperature*

ISO 6892-2, *Metallic materials — Tensile testing — Part 2: Method of test at elevated temperature*

ISO 7500-1, *Metallic materials — Calibration and verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Calibration and verification of the force-measuring system*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

Figure 1 shows schematic sequences for the overall testing method including hydrogen pre-charging (such as electrochemical method described in ISO 16573-1), mechanical testing and hydrogen analysis. Mechanical properties such as, yield strength, tensile strength, fracture strength, elongation to fracture and reduction of area are measured by applying tensile load at slow strain rate before and after hydrogen charging. Hydrogen contents in the specimen shall be measured by thermal desorption analysis, and the relationship between the diffusible hydrogen content and the degradation of mechanical properties shall be obtained. Thermal desorption analysis of pre-charged but not deformed samples allows the quantification of the initial diffusible hydrogen content. However, thermal desorption analysis of pre-charged and deformed samples is only valid when the slow strain rate test is carried out using the

plated samples. This method provides at least a qualitative comparison of the resistance to hydrogen embrittlement among several high-strength steels having different microstructures or compositions.

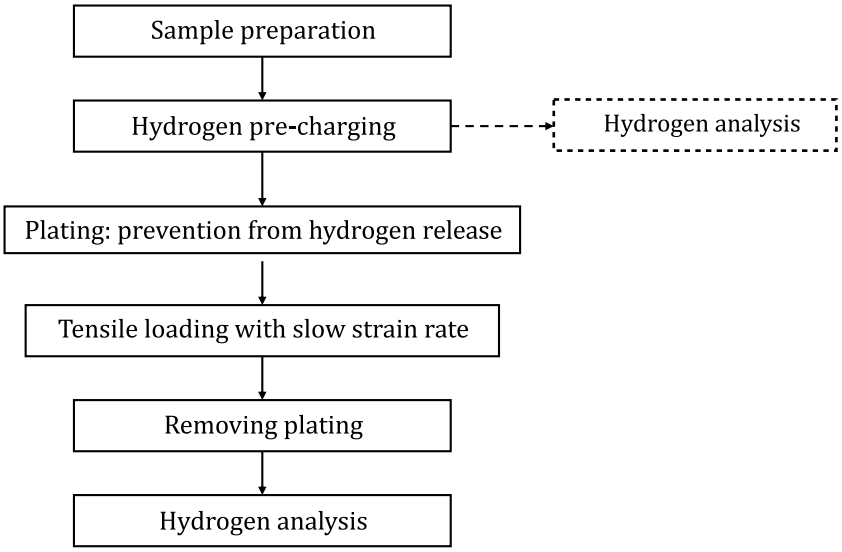


Figure 1 — Flow chart illustrating the test methods

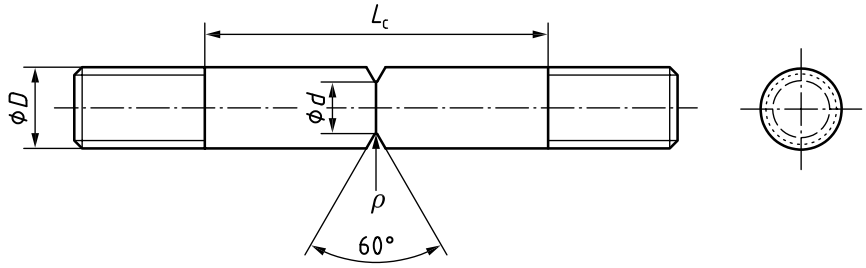
5 Specimen preparation

5.1 General

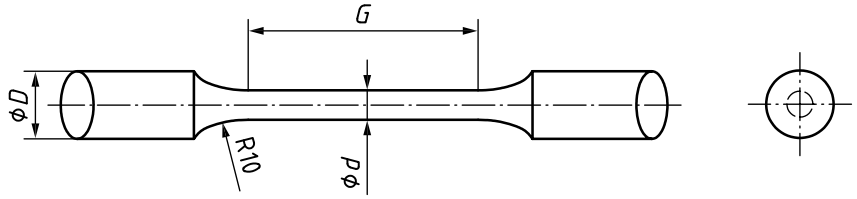
Tension specimens (bar type and flat type specimens) shall be used for evaluation of hydrogen embrittlement[3][5].

5.2 Cylindrical type specimen

The dimensions of the specimens shall be in accordance with Figure 2, and other configurations of the test specimen may be applied. Unless otherwise specified, diameter of the specimen shall be 10 mm. For samples with smaller diameter (i.e. D = 5 mm), ρ/D = 0,02 may be applied[2].



a) Notched specimen



b) Smooth specimen

Key

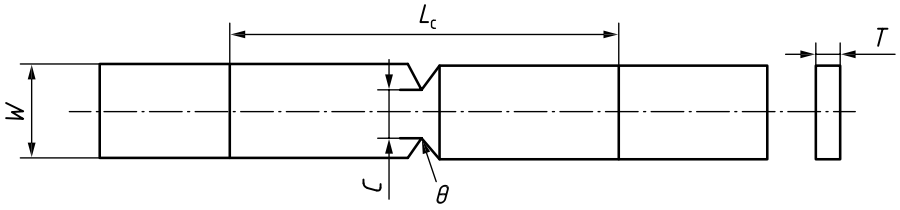
ρ	radius of the notch bottom
d/D	0,6
ρ/D	0,01 or 0,02
L_c/D 7	7
G/D 5	5

NOTE Some types of specimen do not have thread.

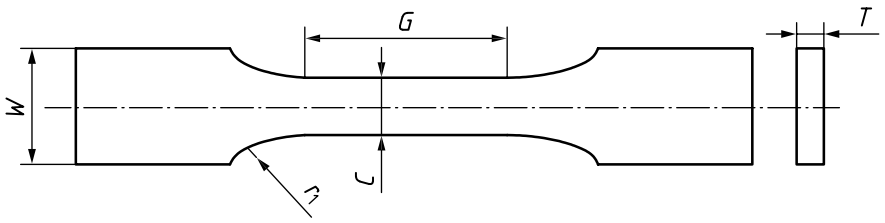
Figure 2 — Dimension of cylindrical type specimens

5.3 Flat type specimen

Flat type specimens shall also be used. Dimensions are shown in Figure 3. It is recommended to use specimens with 10 mm in grip width as a standard size. In case of a flat type specimen, refer to ISO 6892-2:2018, Figure A.1 for grip with bolt.



a) Notched specimen



b) Smooth specimen

Key

W	width of grip ends	1	$C/W \leq 0{,}6$
C	width of gauge	2	$G/W \geq 2{,}5$
G	length of gauge in smooth specimen	3	$L_c/W \geq 7$
θ	notch angle (degree)	4	$0{,}1 \leq T/C \leq 1$
r_1	radius of fillet (mm)	5	$\theta = 60$
T	thickness:(mm) and should be larger than 1 mm	6	$r_1 = 10$

T should be larger than 1 mm.

Figure 3 — Dimension of flat type specimens

6 Hydrogen charging methods

6.1 General

There are four hydrogen charging methods: cathodic charging, hydrogen absorption in aqueous solution at free corrosion potential, hydrogen absorption in atmospheric corrosion environments and hydrogen absorption in high pressure hydrogen gas. The examples of the condition of each method are as follows.

6.2 Cathodic charging

6.2.1 Hydrogen charging solution

To estimate the effect of hydrogen on the mechanical properties of steels, the hydrogen is forced to diffuse into the specimens by the cathodic charging method. For hydrogen pre-charging, the charging solution should be prepared in accordance with Table 1.

Two kinds of solutions may be used for hydrogen pre-charging. Solution 1 may be used for introducing a relatively large amount of hydrogen to the specimens and Solution 2 may be used for introducing a small amount of hydrogen.

Table 1 — Chemical composition of the solutions for hydrogen charging

Charging solution	Element	Content g/l	Mark
Solution 1	NaCl	30	Large amount of Hydrogen
	NH4SCN	3	
Solution 2	NaOH	4	Small amount of hydrogen

6.2.2 Hydrogen charging conditions

The electro-chemical cell for hydrogen pre-charging may be placed in a 200 ml to 1 000 ml beaker. It is recommended that the anode of the electrochemical cell be made of platinum wire of spiral type of 0,5 mm in diameter and 2 m in length (counter electrode), and the specimen works as the cathode (working electrode). After the Pt wire and the specimen are placed in the cell, apply the constant current of its current density in the range of 0 A/m2 to 20 A/m2 by using potentiometer/galvanostat for 48 h. A charging time of 48 h is recommended, but other charging times may be used as long as a total time of 72 h is reached for hydrogen charging and the homogenization treatment by room temperature exposure after cadmium (Cd) plating. For materials with low hydrogen diffusion coefficient, the hydrogen charging time and the total time may be increased. The specimen's surface area shall be calculated for proper current supply. The pre-charged hydrogen amount may be changed by varying the current density or pre-charging time. However, it is recommended to use fixed pre-charging time and current density to get reproducible test results.

6.3 Hydrogen absorption in aqueous solution at free corrosion potential

For hydrogen charging by corrosion in acid, HCl solutions or HCl with CH3COOH/CH3COONa buffered solutions are often used. For example, the specimen is immersed in 5 % HCl solution at room temperature. Immersion time shall be determined based on the specimen size and hydrogen diffusion coefficient of the tested material.

6.4 Hydrogen absorption in atmospheric corrosion environments

For hydrogen charging by atmospheric corrosion, the salt spray test (SST) or cyclic corrosion tests (CCT) including salt spraying, drying and humidifying are carried out. Examples of CCT processes are listed in Table 2.

Table 2 — Example of CCT processes

Process	Conditions	Time
Salt spray	5 % NaCl, 35 °C	2 h
Dry	20 % to 30 % humidity, 60 °C	2 h
Wet	≥95 % humidity, 50 °C	2 h

6.5 Hydrogen absorption in high pressure hydrogen gas

For hydrogen charging by hydrogen gas, the specimens are exposed directly to gaseous hydrogen up to 140 MPa at above room temperature. Exposure time is determined based on the specimen size and hydrogen diffusion coefficient of the tested material. However, extreme care is necessary, and it is not recommended due to the danger of experiment.

7 Preparation of electroplating solution and electroplating condition

7.1 General

The plating process is applied for the hydrogen pre-charging method in order to prevent hydrogen release during the loading test.

7.2 Electroplating solution

After hydrogen pre-charging, plating shall be conducted to prevent the release of hydrogen from the specimens during constant loading test. Cd is a well-known toxic material but the hydrogen diffusivity in Cd is known to be nearly zero. Instead of using toxic Cd, other appropriate plating materials, for example, Zn may be applied. The elements for Cd plating solution and the amounts of each element are listed in Table 3. The pH of the solution shall be 3 pH to 5 pH. The pH of the solution can be adjusted by adding H3BO4 or ammonium solution (see Table 3).

IMPORTANT — When Cd coating is applied, the solution should be handled with special care.

Table 3 — Elements for Cd and Zn plating solutions and the amounts of each element

Solution	Element	Type	Amount
Cd plating solution	Cd(BF4)2	solution [(C) = 50 %]	427 g
	NH4BF4	solute	48 g
	H3BO4	solute	21,6 g
	H2O (distilled)	solvent	460 ml
	pH	-	3 to 5