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

**Part 2:
Slow strain 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 à déformation lente

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: 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).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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.

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

Part 2: Slow strain 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.

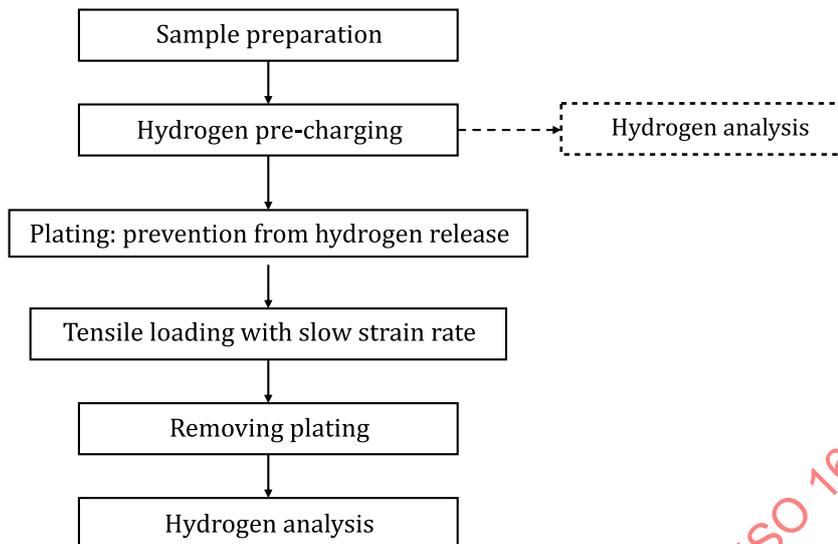


Figure 1 — Flow chart illustrating the test methods

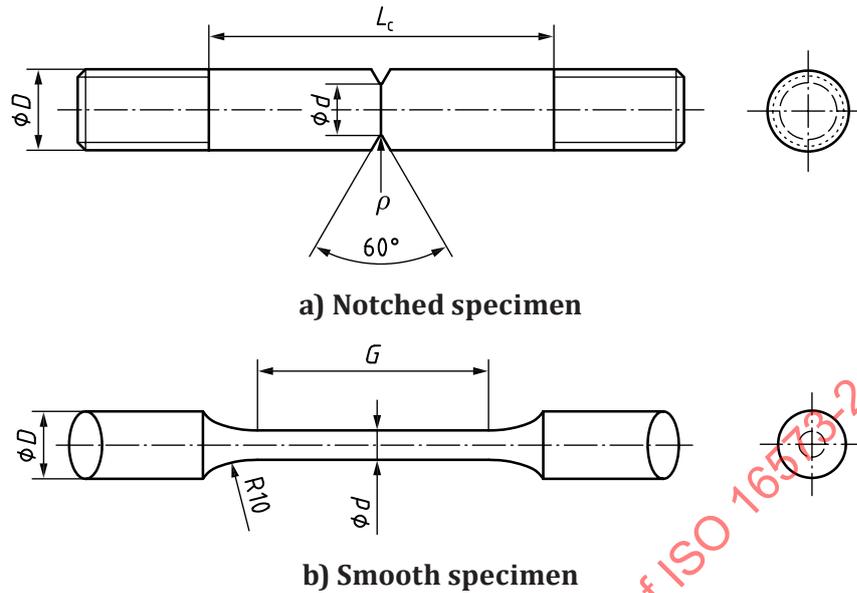
5 Specimen preparation

5.1 General

Tension specimens (bar type and flat type specimens) shall be used for evaluation of hydrogen embrittlement^{[2][3]}.

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), $\rho/D = 0,02$ may be applied^[4].



Key

ρ radius of the notch bottom

d/D 0,6

ρ/D 0,01 or 0,02

L_c/D 7 7

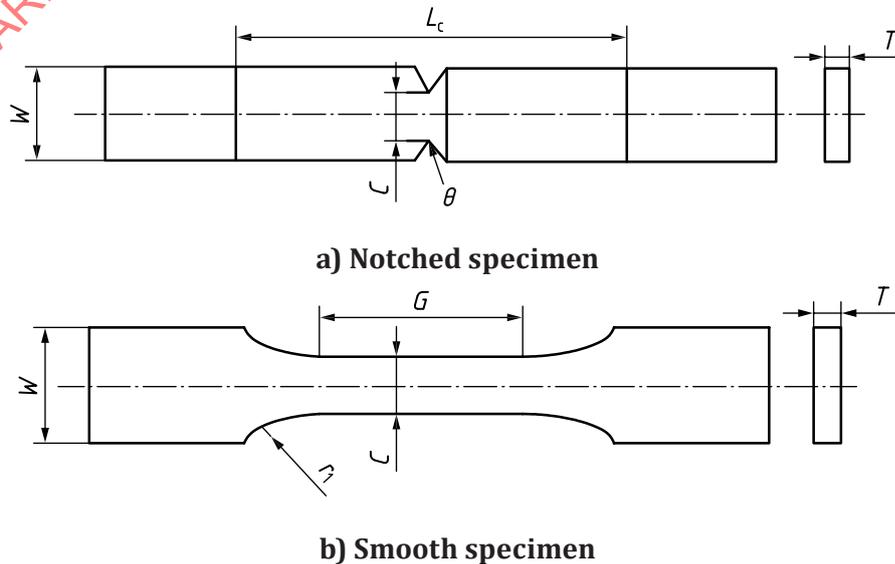
G/D 5 5

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.



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	Mark
		g/l	
Solution 1	NaCl	30	Large amount of Hydrogen
	NH ₄ SCN	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 $\text{CH}_3\text{COOH}/\text{CH}_3\text{COONa}$ 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 H_3BO_4 or ammonium solution (see [Table 3](#)).

WARNING — 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	$\text{Cd}(\text{BF}_4)_2$	solution [(C) = 50 %]	427 g
	NH_4BF_4	solute	48 g
	H_3BO_4	solute	21,6 g
	H_2O (distilled)	solvent	460 ml
	pH	-	3 to 5

Table 3 (continued)

Solution	Element	Type	Amount
Zn plating solution	ZnCl ₂	solute	45 g
	KCl	solute	234 g
	H ₃ BO ₄	solute	21,6 g
	Sodium Dodecyl Sulfate	solute	0,5 g
	H ₂ O (distilled)	solvent	700 ml
	pH	-	5 to 6

7.3 Electroplating conditions

An electrochemical reaction is used for Cd plating. Immerse the metal platinum (Pt) anode into the prepared Cd solution, and immerse the specimen in order for it to work as a cathode. After hydrogen pre-charging, clean the specimens by mechanical grinding (polishing) or with 0,1 N HCl and 0,1 N NaOH by ultrasonic cleaner for 30 s, respectively. Then, immerse the specimens in Cd plating solution and apply a constant current of 0,5 A for 300 s. Other conditions of current and time may also be used for Cd plating. Using the potential difference, Cd is adhered to the surface of the specimen.

The thickness of Cd plating should be at least 15 µm or higher to prevent hydrogen release. Extreme care is needed to handle the solutions containing Cd during the entire Cd plating procedures.

8 Slow strain rate test

8.1 General

This test measures the loss of material strength and ductility in the hydrogen charged specimen as compared to the uncharged one and determines which composition or microstructure reveals higher resistance to hydrogen embrittlement.

Slow strain rate test shall be performed by use of general tensile testing machines in accordance with the requirements of ISO 7500-1, ISO 6892-1 and ISO 6892-2.

8.2 Procedure

- a) Use adequate jig and tensile test system to apply uniaxial loading to the specimens. Chamber with environmental controller shall be used in SSRT if the behaviour of hydrogen pre-charged specimen at various environment should be considered. Generally, the temperature of the chamber should be in the range (-40 °C to 85 °C) in accordance with the criterion accepted for all hydrogen fuel stations.

NOTE Here, "environment" represents temperature, humidity, etc.

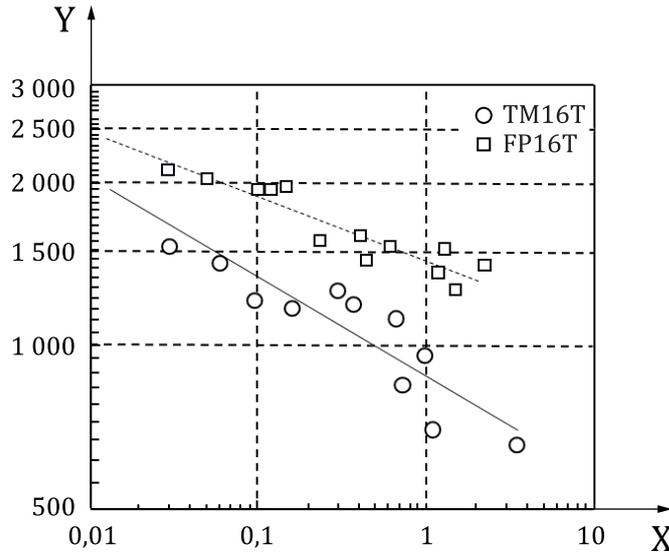
- b) Measure fracture strength of specimens containing various diffusible hydrogen contents. In the case of hydrogen continuous charging, various current densities should be applied to measure the degradation of fracture strength.
- c) Use a constant strain rate to pull the test specimen. For both smooth and notched tension specimens, use a strain rate of 10⁻⁵/s (with the tolerance of ±30 %) based on 25 mm of the gage length of the specimen. An extensometer shall be used to measure the strain rate. For full characterization of the hydrogen embrittlement behaviour or the material, it can be necessary to conduct a wide variety of tests at strain rates either higher or lower than the values stated herein.
- d) From the relation between test force (or stress) and extension (or strain) of smooth specimen, measure tensile properties such as yield strength (proof strength), tensile strength, fracture strength (calculated from load at fracture/fractured area), elongation to fracture and reduction

area. In the case of notched specimen, measure notch tensile strength and notch fracture strength (calculated from the load at fracture/fractured area).

- e) To assure the reliability, at least 3 specimens shall be tested with the same cross head speed.
- f) After the slow strain rate test, measure diffusible hydrogen contents of the specimen immediately. If it is impossible to measure diffusible hydrogen content just after the test, specimens should be preserved below $-50\text{ }^{\circ}\text{C}$, preferably in liquid nitrogen, to prevent hydrogen release from the fractured surface. Freezer or dry ice box where the temperature is controlled under $-50\text{ }^{\circ}\text{C}$ shall be used to preserve the specimens. In such cases, maximum preservation period should be one week. Alternatively, the thermal desorption analysis of pre-charged but not stressed samples allows the quantification of the initial diffusible hydrogen content.

8.3 Presentation of the results

- a) Several examples of the results obtained by the slow strain rate test are shown in [Figures 4, 5 and 6](#). Results shall be displayed in the form of curves in which the horizontal axis represents the diffusible hydrogen content and the vertical axis represents the susceptibility to hydrogen embrittlement through the property chosen for representing it. Figures shall be drawn in log-log coordinates. Results shall also be expressed as coefficients a and b where $\log(Y) = a \log_{10}(X) + b$. Curves generally contain at least 5 measured values. Those measured data shall be regularly distributed. The ratio of diffusible hydrogen contents of maximum value and minimum value should be at least 10.
- b) In [Figure 4](#), the horizontal axis indicates the diffusible hydrogen content of the specimens and the vertical axis represents either fracture strength in the smooth specimen or notched fracture strength in the notched specimen. In [Figure 5](#), the horizontal axis should be the diffusible hydrogen content, and the vertical axis represents notch tensile strength. In the case of smooth specimen, the vertical axis may represent ductility such as reduction of area or elongation to fracture as shown in [Figure 6](#).
- c) In [Figures 4 to 6](#), the vertical axis shall be replaced by the parameter such as fracture strength/tensile strength, and/or the horizontal axis shall be replaced by current density (mA/mm^2).
- d) It shall be concluded that the susceptibility of hydrogen embrittlement is severe if the vertical axis parameter of a material is lower than that of the competitive material at the specific diffusible hydrogen content, or at the specific current density.
- e) As an additional evaluation method, local hydrogen concentration and local stress may be calculated by numerical method for comparing the results of specimens with different stress concentration factors^[2].



Key

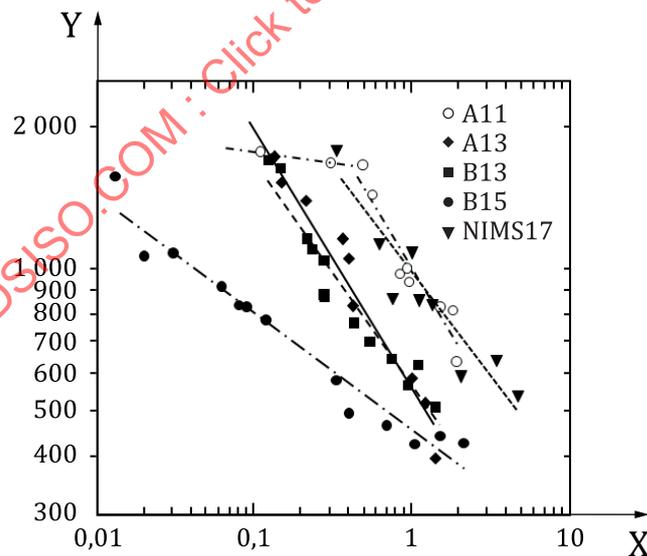
X diffusible hydrogen content, % (mass fraction or mass ppm^a)

Y fracture strength, MPa

FP16T and TM16T denote full pearlite microstructure and tempered martensite microstructure, respectively, having the same tensile strength of 1 600 MPa^[5].

^a 0,01 % is the equivalent of 100 ppm, ppm is a deprecated unit.

Figure 4 — Example of the results of the slow strain rate test (diffusible hydrogen contents vs fracture strength or notched fracture strength)



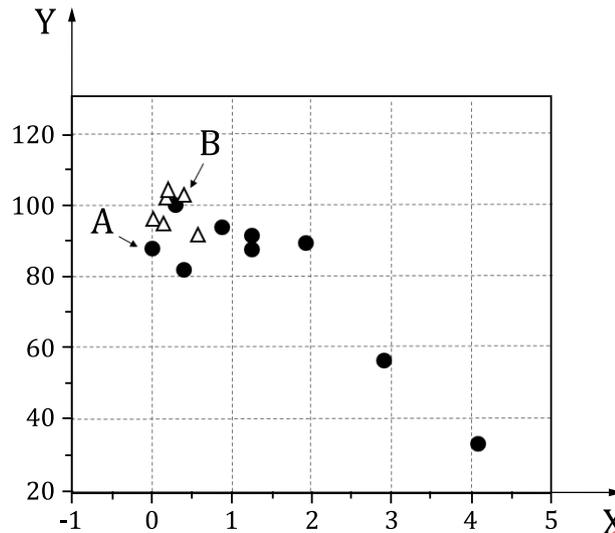
Key

X diffusible hydrogen content, % (mass fraction or mass ppm)

Y notch tensile strength, MPa

A11, A13, B13, B15 and NIMS17 denote tempered martensitic microstructure having the tensile strength of 1 050 MPa, 1 305 MPa, 1 320 MPa, 1 450 MPa, and 1 760 MPa, respectively^[3].

Figure 5 — Example of the results of the slow strain rate test (diffusible hydrogen contents vs notch tensile strength)



Key

- X diffusible hydrogen content, % (mass fraction or mass ppm)
 Y reduction of area, %
 A pre-charged at 30 °C of cold-drawn pearlitic steel
 B pre-charged at 200 °C of cold-drawn pearlitic steel

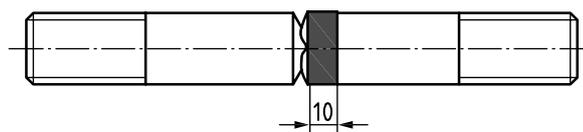
Figure 6 — Example of the results of the slow strain rate test result (diffusible hydrogen contents vs reduction area (%) of smooth specimen)^[6]

9 Post-test specimen treatment

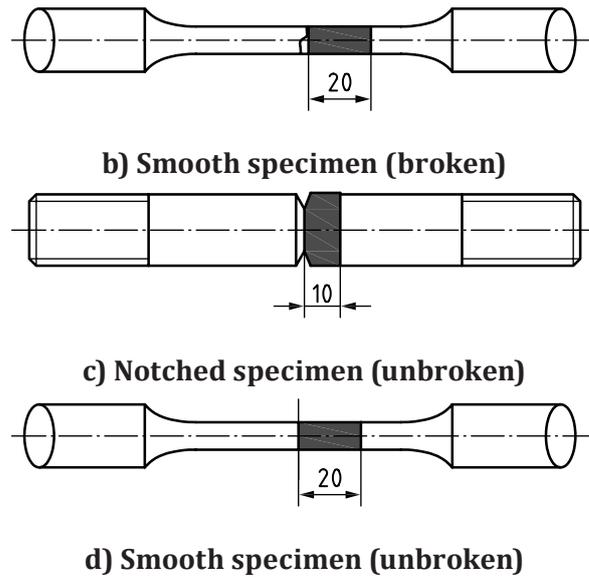
The following process shall be applied after the specimens are fractured. To prevent hydrogen desorption, keep fractured specimens or specimens in liquid nitrogen or freezer or cooler box containing dry ice below -50 °C. For the specimens preserved in freezer or cooler box containing dry ice where the temperature shall be controlled below -50 °C, maximum preservation period should be one week. For the Cd-coated (or Zn-coated) specimens, removing the Cd layer (or Zn layer) is necessary before analysing diffusible hydrogen content.

For fractured specimens or specimens not fractured after 100 h or more have elapsed, the following process shall be applied.

- To prevent hydrogen desorption, keep fractured specimens or specimens not fractured after 100 h or more have elapsed in liquid nitrogen, or freezer or cooler box containing dry ice below -50 °C.
- Samples for the hydrogen content analysis are prepared as follows (see [Figure 7](#)). In case of broken specimen, cut out at a predetermined length from the flat part closest to the broken surface. For unbroken specimen, cut out at a predetermined length from the centre of the specimen.



a) Notched specimen (broken)



NOTE The shaded area represents the part taken for hydrogen content analysis

Figure 7 — Hydrogen content analysis

- c) For the Cd-coated specimens (pre-charged condition), removing the Cd layer is necessary before analysing diffusible hydrogen content. To remove the Cd layer, the cut specimen is immersed into Cd plating solution and the electrodes are connected in a reverse way of the Cd plating, i.e. the anode is the specimen and the cathode is the platinum (Pt). A constant current of 1,2 A is applied for 5 min to remove the Cd layer completely (removing the Zn layer is same as removing the Cd layer). This procedure shall not be applied for the continuous charging method. If a film is formed on the surface of the specimen after the Cd layer has been removed or after continuous charging, it is necessary to polish the specimen using emery paper until a metallic lustre is obtained, and clean it with ethanol in an ultrasonic cleaner for 30 s. After removing the Cd layer, the specimen shall be kept in liquid nitrogen again before performing hydrogen thermal desorption analysis.

10 Hydrogen thermal desorption

10.1 General

Diffusible hydrogen is generally calculated by integrating the first peak of the curve of thermal desorption analysis. In the case of tempered martensitic steel, when several peaks are observed at low temperature (i.e. below 400 °C), diffusible hydrogen may be calculated by integrating peaks below 400 °C of the curve of thermal desorption analysis. In the case of drawn pearlite steel, calculation is necessary by integrating the hydrogen desorption curve having a peak in the vicinity of 100 °C only. If peaks at 100 °C and 300 °C overlap, measure from the room temperature to the lowest point between the two peaks. In the range of the heating rate of up to 200 °C/h, the heating rate does not have a significant effect on the test results. The 100 °C/h heating rate is recommended. When the heating rate is over 200 °C/h, hydrogen desorption will not end with up to 400 °C. Then, make a calculation by integrating the range until the appropriate hydrogen desorption is terminated. The heating rate shall be reported.

10.2 Experimental apparatus (gas chromatograph)

The equipment for thermal desorption analysis consists of three parts: heating, gas sampling, and detecting. When the specimens are heated, hydrogen inside of the specimens diffuses out and flows into the gas chromatography column with carrier gas (high purity Ar or He gas). The heating rate was fixed at any value of up to 200 °C/h, and the specimen is heated up to 400 °C. The 100 °C/h heating rate is recommended. Gases can be separated by an adsorption column. For the accurate measurement of hydrogen content, measuring the background is recommended.