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Measurement of diffusible hydrogen in metallic materials - HELIOS 4 HOT PROBE method

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Ref. No.:CWA 17794:2021 E

Contents

Page

European Foreword

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Introduction

HELIOS 4 HOT PROBE test method was developed as a consequence of the use of advanced high strenght steels with increased mechanical properties that present awsome performance but can be susceptible to hydrogen emebrittlement phenomenon. Diffusible hydrogen may be absorbed in several steps of component production from steel making to part manufacturing, and it is fundamental to identify anomalous hydrogen content that may potentially lead to failure.

At the state of the art, at the best of the knowledge of the authors of this document, none non destructive method for industrial monitoring of hydrogen absorption and consequently assess the risk of hydrogen induced delayed fracture, has already been developed for bare and coated metals.

The present test method was developed within the framework of FormPlanet Horizon 2020 project to make the first step toward industrial online diffusible hydrogen monitoring and assess sheet metal part quality directly during manufacturing with a non-destructive methodology. We made a demonstrator on chassis for automotive industry, but also geometries as coils or any other components are allowed.

The research activity was focused on the optimization of equipment operation as well as the development and validation of test method.

In particular "Task Industrial on-line deiffusible H measurements" was carried out to improve the equipment functionality, instrument has been redesigned, both hardware and software, in order to reach the Industry 4.0 objectives, as well as sensor calibration.

Later in Task "Transport BiW or chassis part/AHSS" the device was used for industrial demonstrator of the HELIOS 4 HOT PROBE measurement method on high strength AlSi coated Press hardening Boron steel chassis reinforcement components. Test will be part of the final test bed service catalogue.

Test method falls within the patent EP2912452 B1 rights.

1 Scope

This CWA provides a set of guidelines for the measurement of diffusible hydrogen content in steel sheets, that can be susceptible to hydrogen embrittlement phenomenon. Hydrogen pick up can take place in several processes from steel production, through part manufacturing, till the service life of component.

Current risk assessment methods consists in complicated laboratory tests that involve time consuming sample preparation and long test duration. These procedure are not suitable for industrial monitoring where quick, easy and possibly non-intrusive metodologies are required.

In this framework, the HELIOS 4 HOT PROBE performs non-destructive diffusible hydrogen measuring method at industrial scale, in order to assess the safe operation of high strength steel parts. The innovative technique allows measurements directly on sheet metal coils and parts, to eventually immediately apply corrective actions in case of process out of control, increasing the safety of the final user.

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 15724:2001, *Metallic and other inorganic coatings — Electrochemical measurement of diffusible hydrogen in steels — Barnacle electrode method*

ASTM F519-18, *Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments*

ASTM F1624-12, *Standard Test Method for Measurement of Hydrogen Embrittlement Threshold in Steel by the Incremental Step Loading Technique*

ASTM G129-00, *Standard Practice for Slow Strain Rate Testing to Evaluate the Susceptibility of Metallic Materials to Environmentally Assisted Cracking*

ASTM STP 962:1988, *Hydrogen Embrittlement: Prevention and Control*

3 Terms, definitions and acronyms

3.1 Terms and definitions

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

ISO and IEC maintain terminological 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/

3.1.1
Advanced high strength steels (AHSS)
Complex, sophisticated materials, with carefully selected chemical compositions and multiphase microstructures resulting from precisely controlled heating and cooling processes. Various strengthening mechanisms, included phase transformation, are employed to achieve improved balance of strength, ductility, toughness, and fatigue properties (Advanced High-Strength Steel (AHSS) Definitions, s.d.).

3.1.2
Diffusible hydrogen content
Amount of hydrogen capable to move in the metal lattice or reversibly trapped at low temperature close to room temperature. Diffusible hydrogen can be quantified by hot extraction method with low temperature isothermal test (C. Georges, 2013).

3.1.3
Hydrogen Embrittlement
Degradation associated to corrosion and corrosion-control processes. It involves the presence of hydrogen into a component, an event that can seriously reduce the ductility and load-bearing capacity, causing cracking and catastrophic brittle failures at stresses below the yield stress of susceptible materials. Hydrogen embrittlement occurs in a number of ways, however, the common features are an applied tensile stress and hydrogen dissolved in the metal (Hydrogen Embrittlement, s.d.).

3.1.4
Hydrogen induced delayed fracture
Phenomenon occurring when material, environment and stress interact with each other, producing an embrittlement effect. Considering hydrogen needs time to diffuse through the metal lattice and accumulate in critical areas with consequent increase of local stresses that can exceed the threshold value of cohesion stress, failure event is unpredictable. Crack nucleation may take place with a certain delay after hydrogen absorption, during parts assembly or directly in service life (R. Valentini, 2019).

3.1.5
Hot Extraction Method
Process to determine dissolved or bound gases in metals. Sample is heated in a dedicated chamber and emitted gas is collected for measurement. The process is suitable for several gases like hydrogen, oxygen, nitrogen or methane that are principally of interest in technical applications (Hot extraction process, s.d.).

3.1.6
Non-destructive method
Appropriate methods and techniques used to detect and evaluate flaws in materials and objects without destroying the specimen at hand. Such tests include e.g. radiographic, ultrasonic, electromagnetic (eddy-current), X-ray, acoustic, and tomographic techniques. Detected flaws are evaluated against acceptance criteria to establish possible rejection or nonconformance. Above nondestructive standard testing are helpful to laboratories and to a wide variety of industrial plants to examine quality of materials and adequacy to their use (Nondestructive Testing Standards, s.d.).

3.2 Acronyms

AHSS	Advanced High Strength Steel
CH	Diffusible hydrogen content
HE	Hydrogen Embrittlement
PHS	Press Hardening Steel
TDA	Thermal Desorption Analysis

4 Equipment

4.1 General

HELIOS 4 HOT PROBE (see Figure 1) is an innovative instrument able to perform diffusible hydrogen measurements on sheet metal samples or components. It consists of a portable equipment that can be easily moved to be used in plant on coils or in the production line during part manufacturing, as well as in situ on components during service life.

The instrument permits maximum adaptability, it operates with no fixed geometry and a magnetic ring allows the probe to stay in place during measurments on ferromagnetic metals, in this way tests in every direction can be performed. Test is semi-automatic, there is no need of operator presence once the test is started.

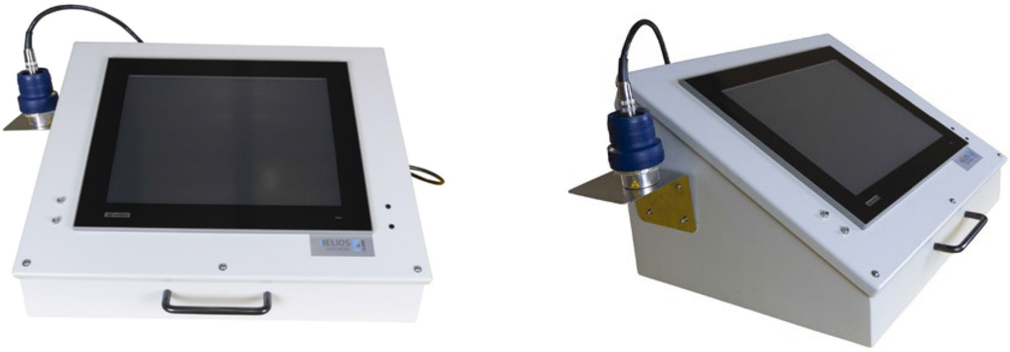


Figure 1 — HELIOS 4 HOT PROBE equipment

Electronic hardware components guarantee reliability and durability of the system. Moreover user interface and software application for instrument management offers maximum customization and optimal performances.

Until today the possibility of in-situ diffusible hydrogen measurements was restricted to the Barnacle Electrode Method (ISO 15724:2001) that was born for cadmium plated components and consists of an adaptation of hydrogen permeation.

It presents some relevant limitations, first of all any coating should be removed before testing. Then, an alkaline solution is needed to induce surface oxidation because hydrogen content is calculated by indirect method starting from the anodic current. Any sample or part result permanently damaged at the end of the test. Finally, considering the test is performed at room temperature, there is the risk of incomplete gas effusion with consequent underestimation of result.

The improvement of HELIOS 4 HOT PROBE in comparison with the Barnacle Electrode method is summarized in Table 1.

Table 1 — HELIOS 4 HOT PROBE features and comparison with old method

LIMITS of Barnacle Electrode method	HELIOS 4 HOT PROBE FEATURES
Time consuming procedure and test duration limit	QUICK RESULT – Analysis time significantly reduced by using innovative solid-state gas sensor and no need to wait surface passivation time
Strong surface condition influence (roughness, palladium, coating). No coating allowed on the exit surface	NO SAMPLE PREPARATION – No need to remove metal coating, measurement is totally non-destructive
Manual analysis and data elaboration. Specialized operator required due to complex assembly procedure	AUTOMATIC AND EASY USE – Measuring probe can be used in any direction by means of a magnetic fixing ring. Test is completely automated once located the probe on the component

Electrolytic test solution required. Barnacle cell has to be located in horizontal position to avoid solution leakage	SAFE ANALYSIS – Neither carrier gas nor electrolytic solution are required for analysis
Hydrogen content calculation by indirect method based on anodic current estimation	HOT EXTRACTION METHOD applied to finished components

The system needs to be trimmed for the single material to be tested: sheet thickness, coating thickness and coating specific weight are all parameters to take into account to define test temperature. Moreover a preliminary laboratory calibration is needed to define calibration parameters for each metal to be tested.

4.2 Technical specifications

HELIOS 4 HOT PROBE is equipped with dedicated control unit and operated by customized software application. This software was completely redesigned to manage the device, introducing the use of blockchain with cybersecurity features. Remote control possibility was also applied, especially to allow new material calibration upload and primary diagnostic.

Main features are schematically reported in Figure 2.

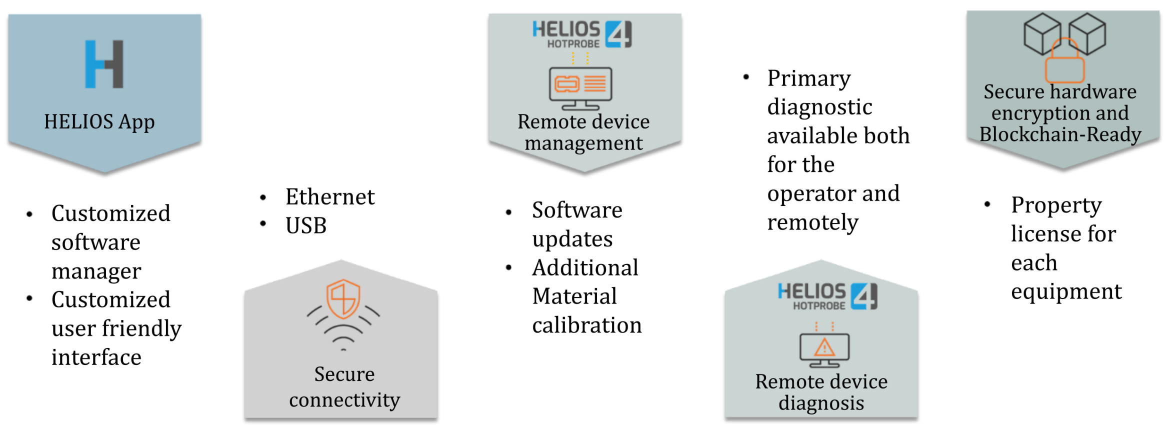


Figure 2 — HELIOS 4 HOT PROBE system main features

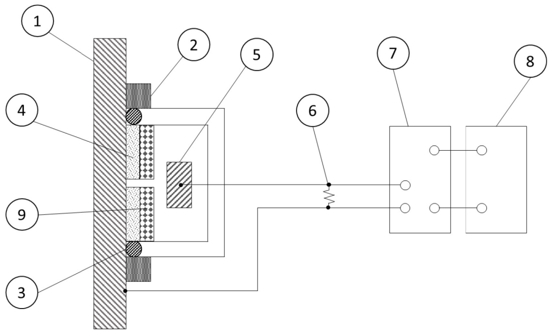
5 Principle

5.1 Measuring unit

5.1.1 General

The measuring unit of the instrument consists in a small probe with metallic case. The measuring process is based on a high sensitivity solid state hydrogen gas sensor. Good measurement repeatability and excellent stability are typical features.

A simplified scheme of the sensor probe is presented in Figure 3. The probe is connected to an electronic control box for data acquisition and elaboration.



a) Schema **b) Photo**

Key

1	test piece	6	known resistance
2	magnet	7	electrometer
3	o-ring	8	data recorder
4	brass disc	9	ceramic heater
5	solid-state sensor		

Figure 3 — HELIOS 4 HOT PROBE measuring unit simplified scheme

In detail the special HOT PROBE is designed to enhance hydrogen effusion out of the metal sample by means of a ceramic heater which locally heats the sample by conduction through the brass disc located on the very top of the probe. Local heating is useful to avoid any sample cut and coating removal, but test temperature remains always lower than 200 °C, in this way, measures are completely non-destructive.

Once acquired the electrical signal of the sensor, that depends on diffusible hydrogen flux exiting from the examined sample, is processed by mathematical codes (by means of a dedicated PC) to assess parameters related to hydrogen diffusion, flux and concentration.

The instrument returns the average diffusible hydrogen content coming out by the measuring area (correspondent to the o-ring located on the top of the probe). Thanks to the small dimensions, the measure is quite punctual but at the same time it can be influenced by some factors related to the test part:

- Lack of uniformity of coating’s thickness;
- Distribution of hydrogen inside the material;
- Presence of rust deposition, paint or oil on the surface;
- Local microstructure of the material.

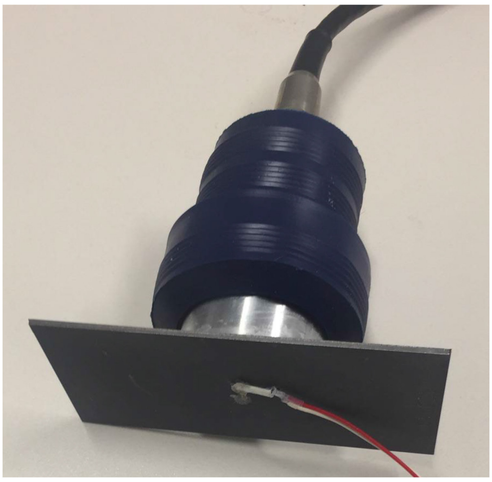
For larger components it can be useful the use of more than one probe or perform several measures in various areas, in order to get the material map in terms of diffusible hydrogen concentration and get statistical significance of results.

5.1.2 Sample temperature profile during measurement

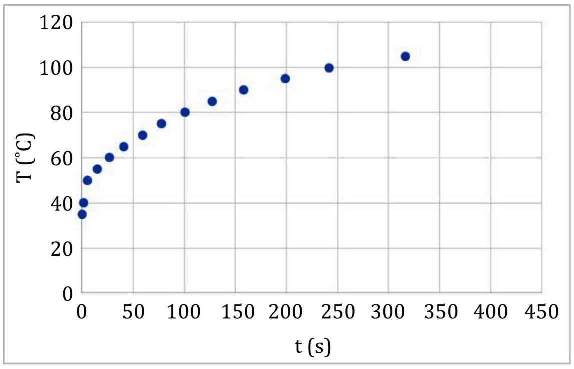
Test temperature is automatically set once the operator chooses material to be tested considering it is functon of material to be tested and is optimized during preliminary laboratory calibration process. Attaching the probe to the metallic part the temperature locally fastly increases thanks to the ceramic heater.

In Figure 4 b) an example of temperature profile as function of time is reported, T_{set} = 110 °C. For this evaluation a metallic sheet sample with thickness 3 mm and a PT100 thermistor were used.

HELIOS 4 HOT PROBE, after reaching test temperature, was attached on one surface of the sample (at room temperature) and the thermistor was applied to the opposite side with apposite high temperature adhesive, see Figure 4 a).



a) Test set up image



b) Time to reach test sample's temperature steady state

Figure 4 — Sample temperature profile during measurement

The difference of temperature, in the range 0-200 °C, for a test sample with thickness lower than 3 mm, is estimated to be less than 10 °C. Temperature profile was simulated and reported in Figure 5.

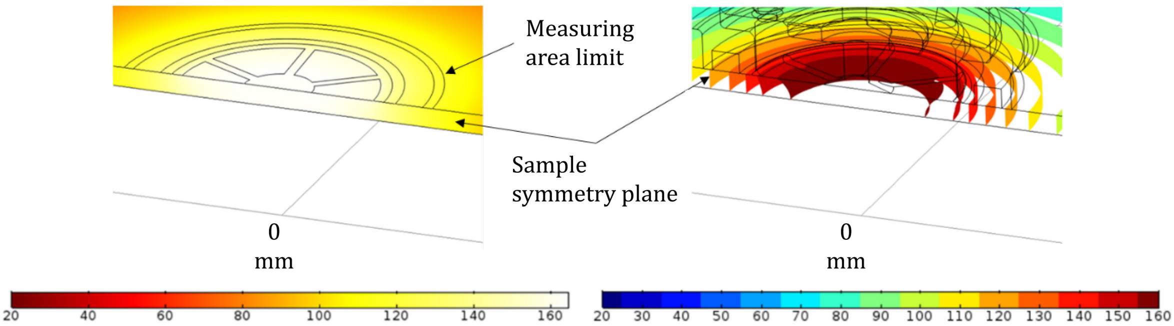


Figure 5 — Sample temperature profile obtained by simulation

5.2 Instrument and measurement validation

Finally, in order to assess equipment operation before of online measurement step, a series of repeatability measurements were performed on USIBOR® 2000. The instrument was preliminary calibrated on the specific material.

The procedure was the following:

1) Some sheet samples, with dimensions 200 × 200 mm, were cut by shearing.

2) Samples were subjected to furnace hydrogen charging with the same process parameters in order to reach the same diffusible hydrogen level inside the metal lattice.

3) Then samples were cut (again by shearing) to obtain both samples for TDA analisys and HELIOS 4 HOT PROBE. Measurements were performed on steel portion very close each other and located in the centre of the specimen.

4) All TDA samples were preliminary prepared with scotch brite grit, under the lubrication of ethyl alcohol, degreased with distilled water and dried in air.

5) TDA was performed by hot extraction method at 265 °C in order to produce the required desorption.

6) HELIOS 4 HOT PROBE test parameters were set according to calibration.

7) HELIOS 4 HOT PROBE and TDA results were compared.

8) Repeatability tests were carried out for two different hydrogen levels.

Test results were summarized in Table 2 and Figure 6. Error bars were plotted according to worst standard diffusivity of each instrument.

Table 2 — Repeatability diffusible hydrogen measurement results

H Level	**HELIOS 4 HOT PROBE (ppmw)**		**TDA (ppmw)**	
	Measurement	Average and standard deviation	Measurement	Average and standard deviation
Low	0,30	0,19 ± 0,07	0,2	0,25 ± 0,05
	0,20		0,3	
	0,13		0,3	
	0,13		0,2	
High	0,55	0,48 ± 0,09	0,5	0,48 ± 0,04
	0,33		0,5	
	0,49		0,4	
	0,56		0,5	

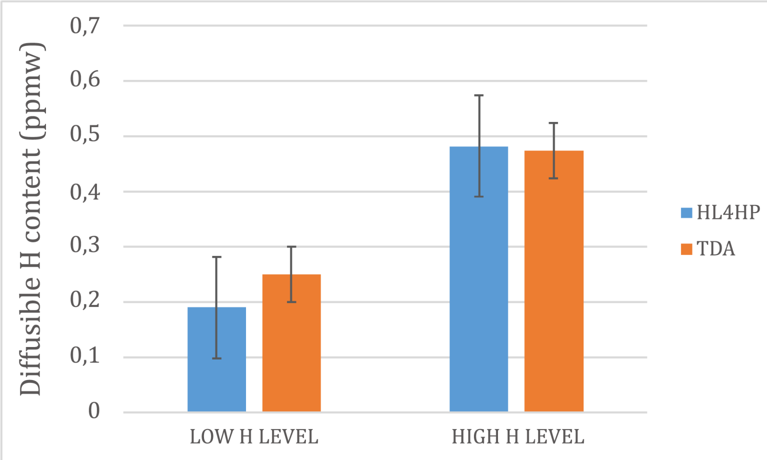


Figure 6 — Average value of diffusible H content measurements in validation samples

6 Sampling

6.1 Selection of measurement area

Measuring probe can be attached to metal, sample geometry can be complex as needed but measurement area should be a flat surface of at least 45 × 45 mm. Allowable sample thickness is lower than 2 mm, the maximum thickness already tested was 1.6 mm. Measurement repetitions should be performed at least two probe diameters far one from another.

For process monitoring, it is strictly recommended to pick at least two samples for each inspection and repeat twice hydrogen measurements on a large component. Avoid single measurement for statistical significance (see 8.2.3 for details).

6.2 Surface preparation and coating

Test method and device have been developed with the aim of simplifying sample preparation procedure. Considering this main objective, a ferromagnetic sheet metal sample or component can be directly tested applying the probe to the sheet metal surface without any coating removal. Coating layers that have already been tested are aluminum silicon layer and electrogalvanized zinc coating.

Care should be paid only to avoid the presence of oil or other liquid contaminants on the surface that may affect and invalidate test result. In case the surface is visibly oily, it can be cleaned by means of ethanol and rinsed with distilled water, then dryed with cold air flow. Pay attention to not use any acetonated and/or chlorinated solvents for sample cleaning.

7 Test procedure

7.1 Text execution

Test procedure consists in the following subsequent steps:

a) Preliminary laboratory calibration of device for the specific sheet metal to be tested (steel, coating, thickness, etc.) in order to be able to perform quantitative measurements.

b) A flat area of at least 45 × 45 mm should be identified on the sample/part for the measurement.

c) Surface preparation according to Clause 6.2.

d) Following the instruction on the display, a series of fields should be filled with test details. Then the material should be selected in the drop-down menu among calibrated materials.

e) Test temperature is automatically set as a function of material.

f) Test can start after reaching target temperature and the probe can be attached to the sample.

g) Signal acquisition starts and data are saved.

h) Test cruve is elaborated by the dedicated HELIOS App which returns the diffusible hydrogen content.

i) Several measurement are recommended for statistical significance.

The HELIOS 4 HOT PROBE method allows two types of evaluations. The first is the process assessment for a new steel grade, the second one is process monitoring, see Clause 8 for details.

8 Test results and report

8.1 New steel grade qualification

8.1.1 General

Qualification of a new steel grade, in terms of hydrogen embrittlement susceptibility, should be done by evaluating the degradationg of mechanical performace associated with hydrogen absorption.

In this specific case a series of mechanical test should be carried out. Both mechanical properties (UTS, elongation at break, etc.) and diffusible hydrogen should be checked for each test sample. A suitable regression curve should be used to determine the critical value that correspond to a reduction of the 30 % of a significant mechanical property (ASTM STP 962). Slow Strain Rate Test (ASTM F519-18, ASTM G129-00), Four Point Bending Tests (ASTM F1624-12) or any other relevant test can be performed to evaluate material performance.

Significant mechanical property and relevant mechanical tests are intended for the final application.

Diffusible hydrogen content can be measured by means of HELIOS 4 HOT PROBE method if samples are in agreement with requirements of Clause 6.1.

8.1.2 Test report

Reference standard procedure for mechanical tests should be indicated in the report.

Test results (mechanical property and diffusible hydrogen) should be summarized for all samples in a table. Regression curve (M. Beghini, 2001) and resulting critical diffusible hydrogen content should be identified with a dedicated plot, see Figure 7.

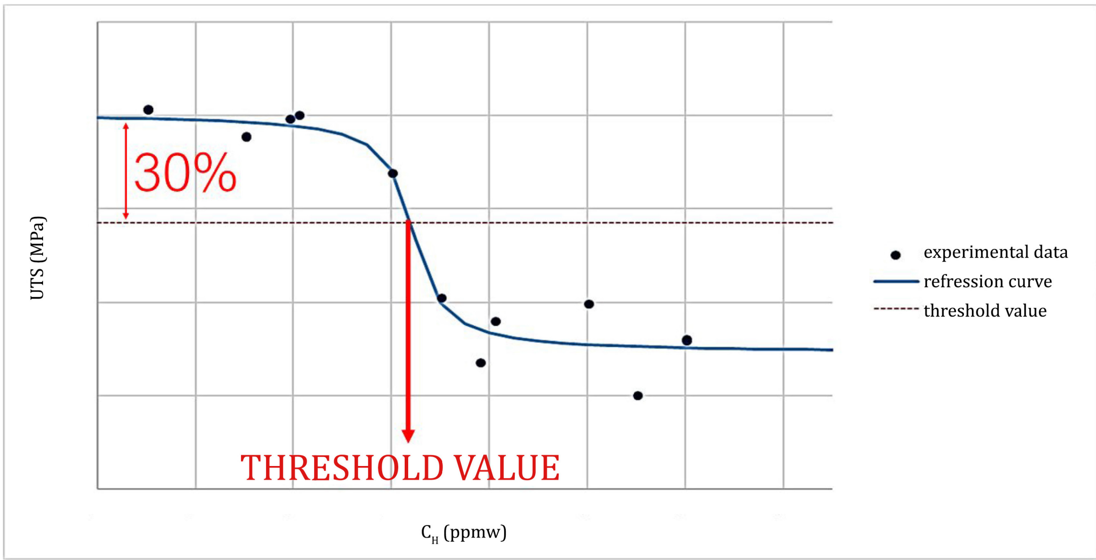


Figure 7 — HELIOS 4 HOT PROBE method applied to new steel grade qualification

8.2 Process monitoring

8.2.1 General

Process monitoring can be reached by performing a series of diffusible hydrogen mesurement along the production line, in particoular evaluating after processes where hydrogen absorption may take place.

In this framerwork it is possible to apply HELIOS 4 HOT PROBE method to evaluate diffusible hydrogen absorption in sheet metal components during the manufacturing of a first batch, or in general for periodic monitoring of process parameters to be under control.

The diffusible hydrogen content in this case will be compared to the threshold value previously determined for the specific material as in Clause 8.1. If any of results is higher than usual or too close to threshold value, corrective actions should be applied to restore a controlled process.

While process can be considered safe and under control, with reference to hydrogen embrittlement risk, if all measured values are quite lower than critical value.

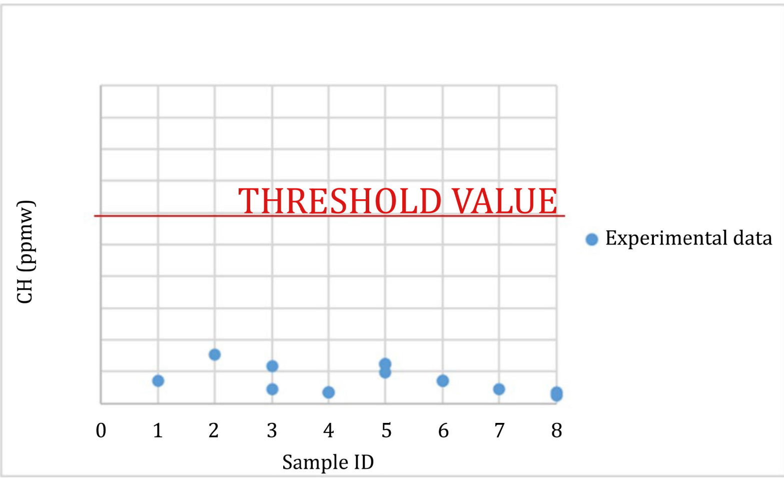


Figure 8 — HELIOS 4 HOT PROBE method applied to process monitoring

8.2.2 Test report

Test results, related to a certain test campaign, should be summarized in a table. Test positions should be uniquely identified and noted (a picture can be attached, see Annex A).

8.2.3 Statistical evalutation

Results can be analyzed in statistical terms to check for average value and standard deviation. Single curves related to the same material, component and phase of production can be graphically compared to understand if there is any anomalies.

Minimum and maximum values should also be checked to understand extreme conditions that can be reached.

8.3 Results to be discarded

There are a few cases where results should be rejected:

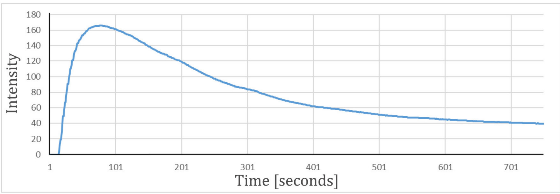
1) in presence of any contaminant on the surface (i.e. oil or rust traces);

2) in presence of contaminants in the air (i.e. acetonated and/or chlorinated solvents spray);

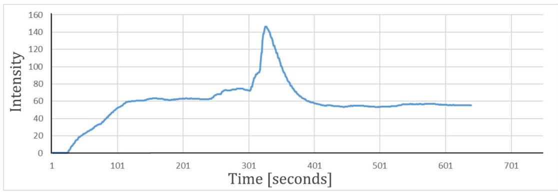
3) in case the instrument registers a signal curve with spikes;

4) considering more than one measurement are strictly recommended, if one of the signal curves results visibly anomalous in comparison with the rests of test series.

Figure 9 a) represents a regular signal curve, on the other hand Figure 9 b) presents a spike and the test should be discarded.



a) Regular signal curve



b) Spike

Figure 9 — Comparison among acceptable and discarded curves

Therefore, it is useful to perform several measurements. Refer to Clause 8.2.3 for statistical significance.

Annex A
(informative)

Application to FormPlanet project case study: Transport BiW or chassis part/AHSS at Cassino FCA plant

A.1 General

LETOMEC has studied together with CRF the implementation, in real production plant, of HELIOS 4 HOT PROBE for raw material and stamped components hydrogen content monitoring to carry out safety parts.

In ideal assembly factory all the components could be assembled without making any force or straining of components, but considering the tolerance that each component has, it is easy to find some millimetres of distance, in worst cases, between the components. When the robot makes the welding, it forces the two components to be near, giving rise to a stress on the component that can potentially create a fracture due hydrogen. The press hardened components are safety parts, so it's fundamental their integrity and the absence of any cracks or defect that could affect the component behaviour during the vehicle crash.

CRF has received from ArcelorMittal 100 USIBOR® 2000 blanks laser cut to stamp the A pillar body in white reinforcement as in the Figure A.1 a).

a) USIBOR® 2000 blanks received by ArcelorMittal

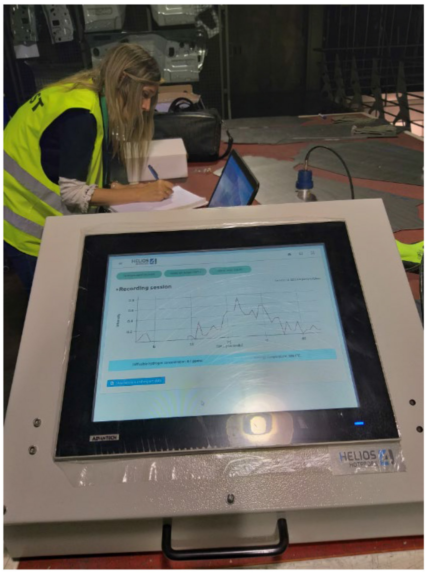
b) FCA Cassino plant hot press forming oven

Figure A.1 — Raw material and plant

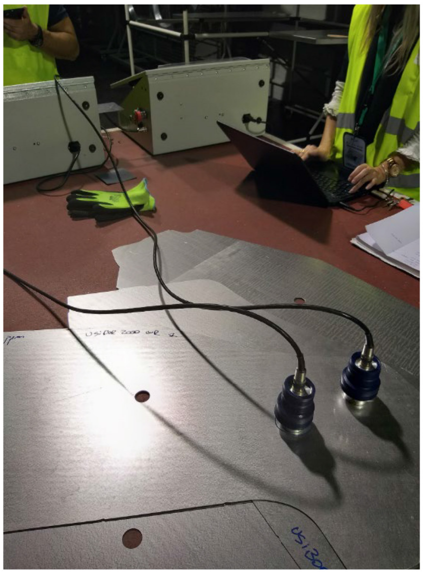
The blanks were stamped in real production line in Cassino FCA plant, the line is equipped with 40 meters oven able to control and measure the air dew point (Figure A.1 b).

A.2 Test procedure

The hydrogen content was measured on the raw material blanks prior to hot stamping process to evaluate this value before and after the process. The HELIOS 4 HOT PROBE was used to perform these measurements in order to conduct the tests without removing the aluminum-silicon coating that cover the blanks. Test procedure was according to Clause 8.2, some photos in Figure A.2.



a) HELIOS 4 HOT PROBE equipment



b) Probes on blanks

Figure A.2 — Hydrogen measurements on the raw material before the hot press forming

Hydrogen content in raw material condition was about 0,1 ppmw or less, that means the material is very clean in terms of hydrogen and this is the base to have high quality components. The stamping process was done in different conditions, they were summarized in Table A.1.

Table A.1 — Hot forming parameters used in the tryout

SAMPLE #	DEW POINT CONTROL	DUELL TIME
1 – 4 – 8	-7 °C	300 s
2	OFF	600 s
3 – 6 – 7	-7 °C	600 s
5	OFF	600 s

The sample number 5 was made in the worst-case condition, without dew point control in the oven, leaving the samples in the oven the maximum time of process window and in addition spraying, before the oven entrance, a water film on the blank to reproduce the worst condition that simulate a possible humidity due to the transportation of the coil.

After the hot forming process, the hydrogen content was measured in different areas of each component to monitor the possible differences in terms of concentration area by areas. 3 areas were identified as per Figure A.3.

a) Measurement areas **b) Probe on component**

Figure A.3 — Measurements on the stamped components

Also in this case the measurements were carried our without removing the coating thanks to the hot probe that heat up the component and is able to check the hydrogen effusion from the material.

A.3 Test report

In Table A.2 the results were reported according to instrument calibration determined in Task 3.3 of FormPlanet project.

Table A.2 —Hydrogen content in the component after hot press forming process in different conditions

MATERIAL	THICKNESS [mm]	COATING	SIGNAL [mV]	H CONTENT [ppmw]	SAMPLE ID	POSITION
USIBOR 2000	1,2	AS150	8	0,07	1	B
USIBOR 2000	1,2	AS150	17	0,15	2	B
USIBOR 2000	1,2	AS150	13	0,12	3	A
USIBOR 2000	1,2	AS150	5	0,05		B
USIBOR 2000	1,2	AS150	4	0,04	4	A
USIBOR 2000	1,2	AS150	4	0,04		B
USIBOR 2000	1,2	AS150	14	0,13	5	A
USIBOR 2000	1,2	AS150	14	0,13		B
USIBOR 2000	1,2	AS150	11	0,10		C
USIBOR 2000	1,2	AS150	8	0,07	6	A
USIBOR 2000	1,2	AS150	8	0,07		B
USIBOR 2000	1,2	AS150	5	0,05	7	B
USIBOR 2000	1,2	AS150	4	0,04	8	A
USIBOR 2000	1,2	AS150	3	0,03		B

Considering critical diffusible hydrogen content prior estimated is around 0,5 ppmw for USIBOR® 2000, all stamped components are safe after press hardening process. Statistical analysis was reported in Table A.3.

Table A.3 — Statistical analysis

Statistic sample count	14
Average value	0,078
Standard deviation	0,04
Minimum value	0,03
Maximum value	0,15

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