Lab Report CS/ONH 27

G4 PHOENIX

- Diffusible Hydrogen Analysis in Weld Seams

Introduction

Hydrogen contributes to material failures in weld seams by so-called hydrogen induced cracking, also described as delayed, under-bead or cold cracking, and can take place hours or sometimes days after welding.

Hydrogen is the lightest element in the periodic table and has the smallest diameter, which makes it highly mobile. This mobility enables hydrogen to easily enter metal lattices and accumulate along grain boundaries and within the heat affected zone (HAZ) during welding. The solubility of hydrogen in the molten material is higher. During solidification of the weld seam, the solubility of hydrogen decreases, and hydrogen becomes trapped or supersaturated in the material, which can lead to failure of the material.

Three critical parameters need to be met to result in hydrogen induced cracking:
1. Presence of hydrogen (organic origin i.e., grease, oil, lubricants, hydrocarbons; or moisture i.e., humidity, electrode coating)
2. Tensile stress of the material
3. Microstructure of the material

To reduce the cracking susceptibility of completed welds, the amount of diffusible hydrogen should be reduced to a minimum. This can be achieved by controlling the welding environment (e.g., humidity), using only low hydrogen electrodes and by controlling pre-heating, interpass and post-heating temperature conditions.

The G4 PHOENIX is a dedicated instrument for the fast and easy determination of diffusible hydrogen. The tube diameter of 30 mm accepts even large samples according to ISO EN 3690 und AWS A4.3. Fast analyses times make this instrument the ideal tool for quality and process control, even in high sample throughput environments.

Measuring Principle

The G4 PHOENIX was developed for the determination of diffusible hydrogen in different sample matrices using the carrier gas hot extraction method. The analysis system comprises of a rapid heating and cooling infrared clamshell furnace and/or a wire-heated tube furnace, both equipped with a quartz tube. For the determination of diffusible hydrogen in weld seams and welding materials according to EN ISO 3690 and AWS A4.3 the infrared heated furnace with a tube diameter of 30 mm is used.

The evolved analyte H_2 is transported by the carrier gas, N_2, to the highly stable and sensitive thermal conductivity cell (TCD) with two measuring ranges. Removal of interfering gases, like CO, is performed by oxidizing CO to CO_2 with Schütze reagent and subsequent adsorption of CO_2 on a molecular sieve. A simple and reliable calibration is guaranteed by the integrated automatic gas dosing unit with 10 different volumes.

Sample Preparation

The base material for the deposition of the weld material is prepared according to the ISO standard and must be clean and free from grease. Degassing of the test pieces is carried out at a temperature of 650°C for a minimum of one hour. The prepared specimen should carry a well-defined identification number. The weight of the center test piece is determined (net weight) and stored in the weight buffer of the analyzer. It is important to guarantee a clear correlation of test piece and weight to the identification number of the respective specimen.

Subsequently, the welding procedure is carried out according to the ISO standard. The sample is cooled immediately and stored in liquid nitrogen or in acetone, saturated with dry ice. Both procedures, welding and cooling, have to be performed in one step and under controlled conditions. The sample is cleaned from impurities and residues by wire brushing under chilled conditions. The run-on and run-off pieces must be removed from the center piece before the sample is analyzed.

Figure 1: Examples of typical weld seam test samples.

Consumables and Accessories

- Schütze reagent
- Molecular sieve
- Glass wool

Method Parameters

The method parameters for analyzing diffusible hydrogen in weld seams should be programmed as follows:

Start delay	5 s	0°C
Max. of start comp.	100 s	
Analysis time	1800 s	400°C
End compensation	60 s	400°C
Max. of end comp.	120 s	

Calibration

The G4 PHOENIX can be calibrated using the integrated gas dosing unit. Possible calibration gases are 100% He, 100% H_2 (caution: calibration gas is exhausted at the outlet and must be lead into a suitable suction or burnt off by a controlled flame) or 5% H_2 in N_2 with a minimum purity of 99.995%.

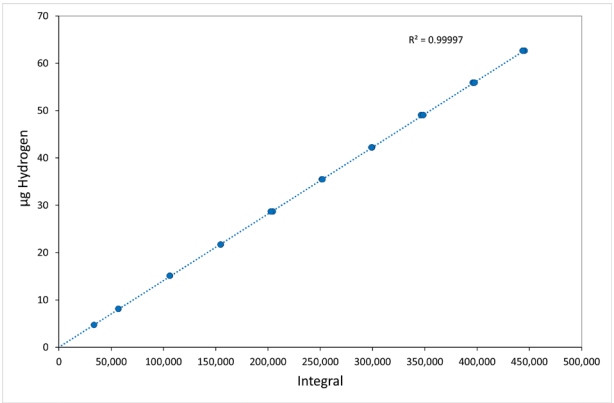


Figure 2: Plot of two cycles of ten calibration volumes with He calibration gas measured with the gas calibration unit, R^2=1.00.

Procedure

I. Determination of the blank value

To measure a blank, select 1 g as sample weight and "Blank" as sample code. Start the analysis cycle without inserting a sample into the quartz tube of the infrared furnace.

II. Sample measurement

The sample is removed from the repository filled with liquid nitrogen or dry ice, cleaned, and weighed (gross weight). The gross weight defines the weight of the carrier material now including the weld seam and is assigned to the according net weight of the empty carrier in the software. The true sample weight of the actual weld seam (gross weight - net weight) is automatically calculated by the software.

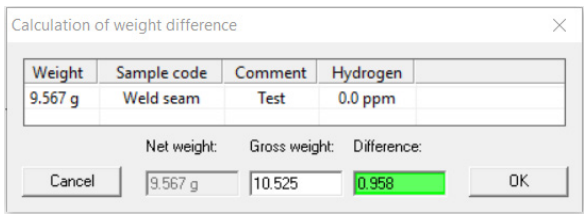


Figure 3: Calculation of the weight difference as function within the Bruker software.

After the weight was added to the weight buffer, the analysis can be started, and the sample inserted into the quartz tube of the infrared furnace. Once the analysis is finished, the sample can be removed.

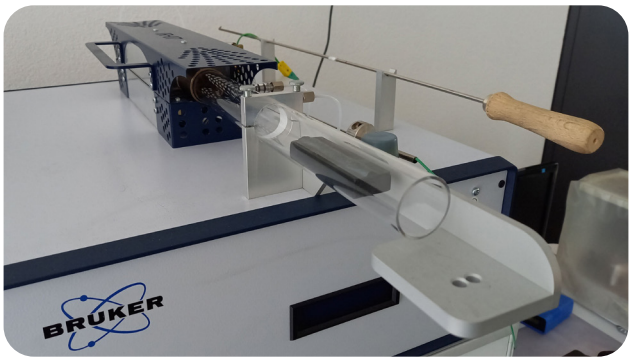


Figure 4: Sample resting at the front part of the quartz tube, ready to be inserted.

Typical Results

Sample 1	
Sample ID	H (ml/100g)
Welding seam 1	1.025
Welding seam 2	1.086
Welding seam 3	1.061
Mean[1)]	1.057
STD[1)]	0.031

[1)] Mean = arithmetic average; STD = absolute standard deviation (1σ)

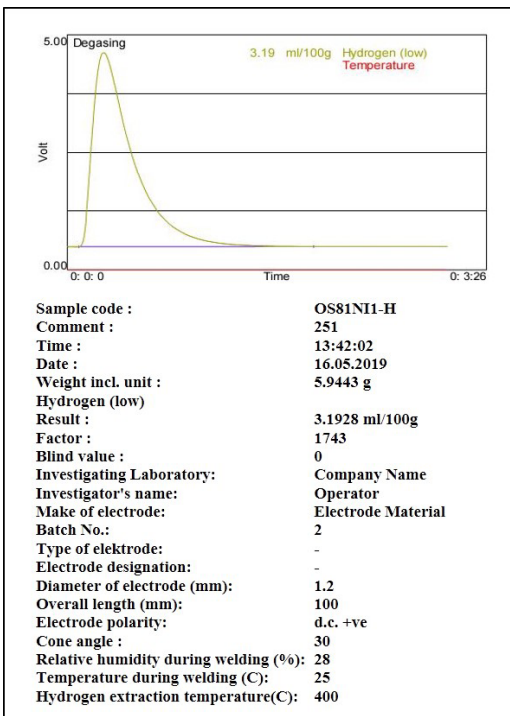
Sample 2	
Sample ID	H (ml/100g)
Welding seam 1	2.493
Welding seam 2	2.494
Welding seam 3	2.497
Mean[1)]	2.495
STD[1)]	0.002

Sample 3	
Sample ID	H (ml/100g)
Welding seam 1	4.356
Welding seam 2	4.324
Welding seam 3	4.338
Mean[1)]	4.339
STD[1)]	0.016

Sample 4	
Sample ID	H (ml/100g)
Welding seam 1	1.850
Welding seam 2	1.360
Welding seam 3	1.130
Mean[1)]	1.447
STD[1)]	0.368

Sample 5	
Sample ID	H (ml/100g)
Welding seam 1	12.600
Welding seam 2	12.090
Welding seam 3	10.680
Mean[1)]	11.790
STD[1)]	0.995

Sample 6	
Sample ID	H (ml/100g)
Welding seam 1	13.220
Welding seam 2	12.510
Welding seam 3	11.760
Mean[1)]	12.497
STD[1)]	0.730



Sample code :	OS81NI1-H
Comment :	251
Time :	13:42:02
Date :	16.05.2019
Weight incl. unit :	5.9443 g
Hydrogen (low)	
Result :	3.1928 ml/100g
Factor :	1743
Blind value :	0
Investigating Laboratory:	Company Name
Investigator's name:	Operator
Make of electrode:	Electrode Material
Batch No.:	2
Type of elektrode:	-
Electrode designation:	-
Diameter of electrode (mm):	1.2
Overall length (mm):	100
Electrode polarity:	d.c. +ve
Cone angle :	30
Relative humidity during welding (%):	28
Temperature during welding (C):	25
Hydrogen extraction temperature(C):	400

Figure 5: Example of an analysis report generated with the Bruker software.

Summary

The G4 PHOENIX provides high precision, easy operation and fast results. It therefore is an ideal choice for quality and process control in welding and other industries affected by hydrogen induced embrittlement.

The optional addition of a quadrupole mass spectrometer to the G4 PHOENIX allows to extend the measurement range to even lower concentration levels. As an alternative to the G4 PHOENIX, the G8 GALILEO with an external IR-furnace can also be used for diffusible hydrogen analysis, in addition to oxygen, nitrogen and total hydrogen determination by melt extraction.

References and further reading

The method parameters for analyzing diffusible hydrogen in weld seams should be programmed as follows:

- EN ISO 3690 (2018): Welding and allied processes – Procedure for Determining the Hydrogen Content in Arc Weld MetalMax. of start comp.
- Kannegiesser, T.; Tiersch, N. 2013: Comparative study between hot extraction methods and mercury method – a national round robin test, Welding in the World 54, R108-R114 (2010).
- Ström, C.; Elvander, J.: Calibration and verification of the hot extraction method including a comparison with the mercury method, IIW Doc II-1543-04, 2004.
- Stremming, H. 2011: Carrier Gas Hot Extraction Method with Thermal Conductivity Detection and Mass Spectrometer - Survey of Principles, Options and Applications. Bruker Elemental GmbH, Kalkar

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