MEASUREMENTS OF DIFFUSIBLE HYDROGEN CONTENTS AT ELEVATED TEMPERATURES USING DIFFERENT HOT EXTRACTION TECHNIQUES – AN INTERNATIONAL ROUND ROBIN TEST

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ABSTRACT

This international round robin test served to scrutinize the procedures specified in ISO/DIS 3690:2009 for determining the diffusible hydrogen content in weld metals with bcc-lattice structure. It was specifically intended to check in what respect the specifications defined in the indicated standards for specimen preparation, storage and hydrogen analysis provide comparable measurement results. The round robin test is presented comprising comparative measurements at various degassing temperatures using hot extraction techniques and a thermal conductivity detector (TCD). A major focus of this investigation was the examination of the maximum degassing temperature for analysing the diffusible hydrogen in materials with bcc-lattice structure. The analyses were performed using two different stick electrodes and three different filler wires. As a significant result it was found that no deviations or increases, were detected in the measured contents of diffusible hydrogen for the investigated degassing temperatures ranging between 45 °C and 400 °C. Hydrogen analyses for contents below HD = 1.5 ml/100 g with the hot extraction techniques in conjunction with TCD applied in this study led to considerable relative standard deviations.

IIW-Thesaurus keywords: *Hydrogen; Measurement.*

1 INTRODUCTION

In order to avoid hydrogen assisted cold cracking of materials with bcc-lattice structure, rigorous determination of limiting values for the diffusible hydrogen is vitally important. This is the reason why, managed by IIW Sub-Commission II-A, the specimen preparation and hot extraction procedures specified in ISO/DIS 3690:2009 [1] for determining the diffusible hydrogen in weld metal with bcc-lattice structure were examined. It was in particular intended in this international round robin test to check whether hot extraction techniques with thermal conductivity detector (TCD) can be used unrestrictedly as reference methods and whether the maximum degassing temperatures and times given in the international standards yield comparable results or not.

This aspect has already been investigated in a previous national round robin test [2] conducted in Germany in which six different laboratories have carried out comparative measurements using both the hot extraction technique at various degassing temperatures and the mercury method. One focus of attention in this investigation was on the examination of the maximum degassing temperature for analysing the diffusible hydrogen in materials with bcc-lattice structure. The results showed that at the applied degassing temperatures of 150 °C and 400 °C there was no increase in the

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measured contents of diffusible hydrogen compared to the mercury method at room temperature. From this, it could be concluded that the mercury method and hot extraction methods with TCD can be regarded as equivalent reference methods.

Based on these studies, the present document summarizes the results of an international round robin test with worldwide ten laboratories of IIW Commission II. Five different filler materials were applied with the object of investigating a widest possible range of different hydrogen contents for H_{Dref} around 1 ml/100 g to 8 ml/100 g deposited weld metal. In this round robin test, 16 test series were performed in different hot gas extraction installations at various degassing temperatures ranging from 45 °C to 400 °C.

2 WELDING PROCEDURE AND APPLIED FILLER MATERIALS

According to the international specifications in ISO/DIS 3690 [1], ANSI/AWS A4.3-93 [3] and JIS Z 3118 [4], respectively, the participants used as base material a carbon non-rimming steel with a carbon content of not more than 0.18 % and a sulfur content of not more than 0.02 % (grade ASTM A36 or SAE 1020). The test piece assembly is degassed at 400 °C ± 10 °C to 650 °C ± 10 °C for one hour and cooled in a dry inert gas atmosphere or a vacuum to remove any hydrogen present in the material. The test piece assembly may be degassed in air if the surface oxide layer is removed prior to testing. Degassed test piece assemblies are stored in a desiccator or under other suitable conditions to prevent oxidation of the test pieces.

In this international round robin test, analyses were performed for determining the diffusible hydrogen content in the weld metals of two different stick electrodes (expected diffusible hydrogen content of around H_{Dref} = 3 ml/100 g and H_{Dref} = 8 ml/100 g, respectively) and three different filler wires (expected diffusible hydrogen content of around H_{Dref} = 1 ml/100 g, H_{Dref} = 2 ml/100 g and H_{Dref} = 6 ml/100 g, respectively, see Section 3.1). The applied filler materials are listed in Table 1.

Table 2 shows the determined welding parameters for the individual test series of the laboratories/participants 1 to 10. The subcategories (5.1 to 7.3) listed in Table 2 were introduced when different test parameters (e.g. degassing temperature) were selected in the same laboratory or by the same participants, respectively (see Table 3). The individual test series served the purpose of varying the degassing temperatures in the hydrogen analysis. The specimen sizes standardized at international level for the hydrogen measurement are depicted in Figure 1.

The hydrogen analysis was performed according to the revised ISO/DIS 3690 [1], using ISO, AWS and JIS type welding fixture, respectively. Static or dynamic hot extraction installations with thermal conductivity detector (TCD) were used (Table 3). Detailed

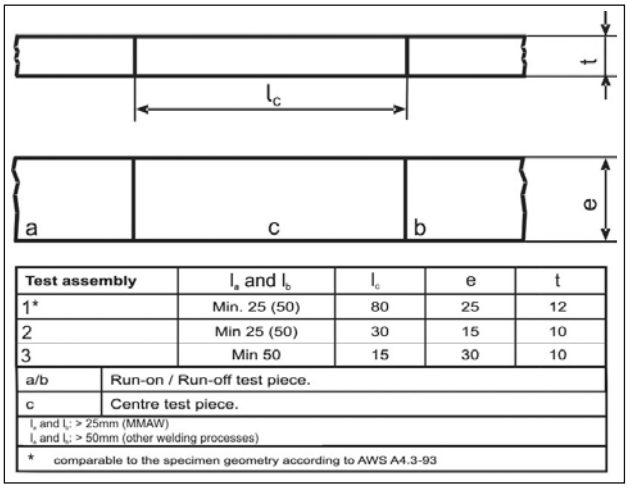
Table 1 – Summary of filler materials and of processing specifications of producer

	consumable				
	stick electrode		wire		
	A	B	C	D	E
type	basic coated electrode with high resistance to weld metal cracking	coated electrode	cored wire, low alloyed, high temperature resistant	seamless copper coated basic flux cored wire for welding of high-strength fine grain structural steels	wire electrode
name	Kestra Kb AC		DCMS Ti-FD	Fluxofil 42	OK Autorod 12.51
producer	Böhler	Lincoln Electric Europe	Böhler	Air Liquide	Esab
norm	DIN EN ISO 2560-A: E42 4 B 32 H5; AWS A5.1-04: E7018-1		EN ISO 17634-A:2006: (T CrMo1 P M 1); AWS A5.29-05: E81T1-B2M	EN 12535: T 69 6 Mn2NiCrMo B C 3 H5; AWS A5.29: E110T5-K4 H4	EN 440-G3Si1; AWS A5.18:ER70S-6
diameter in mm	3,2	4	1,2	1,2	1,2
current in A	125	185	230	290	230
polarity	=+	~	=+	=+	=+
voltage in V	25	25	27	27	27
length in mm	350	450			
welding speed in cm/min			50	50	50
wire feed in m/min			8	8	8
shielding gas			EN 439: M21	EN 439: M21	EN 439: M21
flow rate in l/min			19	20	19
stick out in mm			15 - 20	15 - 20	15 - 25
notes	redrying conditions: 1 hour / 350 °C	redrying is not required; electrodes are in vacuum seales packages			

Table 2 – Welding parameters determined in individual test series of laboratories 1 to 10

			laboratory														
			1	2	3	4	5.1	5.2	5.3	5.4	6.1	6.2	7.1	7.2	7.3	8	9	10
consumable	A	voltage [V]	23.0	22.0	25.0	22.0	25.0	25.0	25.0	25.0	25.0	25.0	27.0	27.0	27.0	25.0	25.0	21.9
		current [A]	129.0	126.0	125.0	125.0	125.0	125.0	125.0	125.0	125.0	125.0	125.0	125.0	125.0	125.0	125.0	126.0
		current canufacturer [A]	125.0															
		weight of deposit [g]	2.58	7.15	2.79	2.67					7.73	8.30	2.73	2.53	2.43		3.33	4.18
		welding speed [cm/min]	18.0								16.5	16.5						
		wire feed speed [m/min]																
		heat input [kJ/mm]																
	B	voltage [V]	24.0	20.0	32.0	25.0	25.0	25.0	25.0	25.0			24.0	24.0	24.0	25.0	25.0	26.5
		current [A]	185.0	205.0	185.0	185.0	185.0	185.0	185.0	185.0	185.0	185.0	205.0	205.0	205.0	185.0	185.0	1850
		current manufacturer [A]	185.0															
		weight of deposit [g]	2.58	12.99	4.52	3.80					9.38	9.93	3.30	2.97	2.77		3.76	4.34
		welding speed [cm/min]	18.0								21.6	21.6						
		wire feed speed [m/min]																
		heat input [kJ/mm]																
	C	voltage [V]	25.0	28.0	27.0	29.0	28.0	28.0	28.0	28.0	27.0	27.0	25.0	25.0	25.0	25.0	30.0	30.4
		current [A]	202.0	230.0	230.0	230.0	230.0	230.0	230.0	230.0	200.0	200.0	230.0	230.0	230.0	180.0	239.0	230.0
		current manufacturer [A]	230.0															
		weight of deposit [g]	2.55	14.54	2.99	3.56					6.83	6.95	3.53	3.73	3.50		3.56	3.80
		welding speed [cm/min]	48.0	30.0	50.0	22.5	50.0	50.0	50.0	50.0	50.8	50.8	50.0	50.0	50.0	50.0	50.0	48.0
		stickout [mm]		18.0		15.0					20.0	20.0	20.0	20.0	20.0			18.0
		wire feed speed [m/min]	8.0	10.0	8.0		8.0	8.0	8.0	8.0	8.0	8.0					8.0	10.0
		heat input [kJ/mm]	0.6	1.3	0.7	1.8	0.8	0.8	0.8	0.8	0.6	0.6	0.7	0.7	0.7	0.5	0.9	0.9
	D	voltage [V]	27.0	32.0	27.0	27.0	27.0	27.0	27.0	27.0	27.0	27.0	27.0	27.0	27.0	27.0	29.5	27.0
		current [A]	294.0	265.0	290.0	290.0	290.0	290.0	290.0	290.0	220.0	220.0	290.0	290.0	290.0	230.0	271.0	288.0
		current manufacturer [A]	290.0															
		weight of deposit [g]	7.98	18.25	3.54	4.07					8.38	8.18	4.87	4.80	4.73		4.15	4.94
		welding speed [cm/min]	36.0	28.0	50.0	28.0	50.0	50.0	50.0	50.0	50.8	50.8	50.0	50.0	50.0	50.0	50.0	53.0
		stickout [mm]		18.0		15.0					20.0	20.0	20.0	20.0	20.0			18.0
		wire feed speed [m/min]	11.0	10.0	8.0		8.0	8.0	8.0	8.0	8.0	8.0					8.0	11.0
		heat Input [kJ/mm]	1.3	1.8	0.9	1.7	0.9	0.9	0.9	0.9	0.7	0.7	0.9	0.9	0.9	0.7	1.0	0.9
	E	voltage [V]	24.0	34.0	27.0	21.0	27.0	27.0	27.0	27.0	27.0	27.0	25.0	25.0	25.0	29.0	30.3	27.7
		current [A]	224.0	260.0	230.0	230.0	230.0	230.0	230.0	230.0	220.0	220.0	230.0	230.0	230.0	270.0	251.0	231.0
		current manufacturer [A]	230.0															
		weight of deposit [g]	3.80	16.44	4.13	3.59					10.03	9.90	3.67	3.77	3.73		4.03	4.06
		welding speed [cm/min]	48.0	40.0	50.0	23.5	50.0	50.0	50.0	50.0	50.8	50.8	50.0	50.0	50.0	50.0	50.0	48.0
		stickout [mm]		15.0		15.0					20.0	20.0	20.0	20.0	20.0			22.0
		wire feed speed [m/min]	8.0	8.0	8.0		8.0	8.0	8.0	8.0	8.0	8.0					8.0	8.0
		heat input [kJ/mm]	0.7	1.3	0.7	1.2	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.9	0.9	0.8

description of the individual measurement techniques is described elsewhere [5]. In a static measurement system, hydrogen is first, at a specific degassing temperature, collected in a separate closed system and is subsequently admitted to the TCD, whereas in a dynamic system it is measured continually while the specimen is heated.



Test assembly	l_a and l_b	l_c	e	t
1*	Min. 25 (50)	80	25	12
2	Min 25 (50)	30	15	10
3	Min 50	15	30	10

a/b Run-on / Run-off test piece.
c Centre test piece.
l_a and l_b: > 25mm (MMAW)
l_a and l_b: > 50mm (other welding processes)
* comparable to the specimen geometry according to AWS A4.3-93

Figure 1 – Possible specimen geometries according to ISO/DIS 3690 [1], ANSI/AWS A4.3-93 [3], respectively

3 RESULTS AND DISCUSSION

3.1 Measured values of the individual laboratories and statistical calculations

The statistical evaluation of the measurement results of all participating laboratories was carried out according to ISO 5725-2 [6]. Table 4 lists the average values H_D and standard deviations of the test series performed by laboratories 1 to 10.

The values for the total average value m_j, the repetition variance s_{rj} and the comparison variance s_{Rj} for the individual parameters are represented in Table 5. The hydrogen content H_{Dref} to be expected according to the producer's data is also indicated.

$$\hat{m}_j = \bar{\bar{y}}_j = \frac{\sum_{i=1}^{p} n_{ij}\,\bar{\bar{y}}_{ij}}{\sum_{i=1}^{p} n_{ij}} = \text{Total average value} \qquad (1)$$

$$s_{rj}^2 = \frac{\sum_{i=1}^{p} (n_{ij}-1)s_{ij}^2}{\sum_{i=1}^{p} (n_{ij}-1)} = \text{Repetition variance} \qquad (2)$$

Table 3 – Survey of standards and TCD techniques applied in laboratories

Lab. Test series	Standard	TCD techniques	Temp. °C	Measuring time min
1	ISO/DIS 3690	Static (Yanaco)	150	360
2	AWS A 4.3-93	Dynamic (H-mat 2500)	400	20
3	ISO/DIS 3690	Static (Yanaco)	150	360
4	ISO/DIS 3690	Dynamic (H-mat 2500)	400	25
5.1	ISO/DIS 3690	Static (Yanaco)	45	4 320
5.2	ISO/DIS 3690	Static (Yanaco)	150	360
5.3.	ISO/DIS 3690	Dynamic (H-mat 221)	400	20
5.4	ISO/DIS 3690	Dynamic (H-mat 286)	400	20
6.1	AWS A 4.3-93	Static (Chromatograph 3350)	150	360
6.2	AWS A 4.3-93	Static (Chromatograph 3350)	100	1 440
7.1	JIS Z 3118:2007[a]	Static (Yanaco)	150	360
7.2	JIS Z 3118:2007 [a]	Static (Yanaco)	100	1 080
7.3	JIS Z 3118:2007 [a]	Static (Yanaco)	45	4 320
8	ISO/DIS 3690	Dynamic (H-mat 221)	400	20
9	ISO/DIS 3690	Dynamic (H-mat 221)	400	20
10	ISO/DIS 3690	Static (Yanaco)	150	360

[a] Welding fixture was JIS type but preparation, the size of specimens and welding procedures were the same as those described in ISO/DIS 3690 [1].

Table 4 – Overview of H_D average values and standard deviations of test series of laboratories 1 to 10

		Lab-no.	Consumable (H_D in ml/100g)				
			A	B	C	D	E
temperature	45 °C	5.1	Ø 2.117 ± 0.127	Ø 5.027 ± 0.116	Ø 2.040 ± 0.110	Ø 0.147 ± 0.005	Ø 1.323 ± 0.374
		7.3	Ø 4.133 ± 0.471	Ø 7.900 ± 0.497	Ø 6.367 ± 0.858	Ø 0.070 ± 0.094	Ø 1.367 ± 0.205
	100 °C	6.2	Ø 2.867 ± 0.125	Ø 7.850 ± 0.206	Ø 5.025 ± 0.192	Ø 0.225 ± 0.043	Ø 2.050 ± 0.287
		7.2	Ø 6.067 ± 0.330	Ø 10.333 ± 0.249	Ø 7.967 ± 0.170	Ø 0.800 ± 0.082	Ø 3.200 ± 0.082
	150 °C	1	Ø 3.280 ± 0.435	Ø 8.570 ± 0.758	Ø 11.460 ± 0.417	Ø 0.527 ± 0.039	Ø 2.877 ± 0.034
		2	Ø 5.693 ± 0.432	Ø 11.060 ± 0.495	Ø 7.800 ± 0.647	Ø 0.713 ± 0.054	Ø 4.340 ± 0.179
		3	Ø 5.020 ± 0.158	Ø 9.450 ± 0.260	Ø 5.570 ± 0.477	Ø 0.660 ± 0.087	Ø 3.320 ± 0.136
		5.2	Ø 2.837 ± 0.079	Ø 7.037 ± 0.405	Ø 3.260 ± 0.177	Ø 0.200 ± 0.064	Ø 2.160 ± 0.174
		6.1	Ø 3.000 ± 0.082	Ø 8.875 ± 0.618	Ø 5.725 ± 0.228	Ø 0.400 ± 0.071	Ø 2.075 ± 0.311
		7.1	Ø 5.633 ± 0.125	Ø 9.300 ± 0.920	Ø 7.800 ± 0.572	Ø 0.667 ± 0.094	Ø 2.733 ± 0.205
		10	Ø 5.058 ± 0.133	Ø 10.980 ± 0.476	Ø 9.226 ± 0.407	Ø 0.862 ± 0.088	Ø 3.204 ± 0.251
	400 °C	4	Ø 3.677 ± 0.208	Ø 7.270 ± 0.376	Ø 5.628 ± 0.206	Ø 0.608 ± 0.191	Ø 1.615 ± 0.119
		5.3	Ø 2.397 ± 0.151	Ø 6.907 ± 0.428	Ø 2.587 ± 0.393	Ø 0.207 ± 0.037	Ø 2.157 ± 0.170
		5.4	Ø 2.073 ± 0.362	Ø 5.960 ± 0.593	Ø 2.540 ± 0.116	Ø 0.207 ± 0.037	Ø 1.467 ± 0.180
		8	Ø 2.627 ± 0.352	Ø 6.043 ± 0.326	Ø 4.917 ± 0.253	Ø 0.213 ± 0.049	Ø 1.350 ± 0.094
		9	Ø 2.478 ± 0.157	Ø 7.172 ± 0.651	Ø 5.760 ± 0.634	Ø 0.502 ± 0.223	Ø 1.440 ± 0.178

Table 5 – Final values for parameter sets

Consumable	p_j	H_{Dref} ml/100 g	m_j ml/100 g	s_{rj} ml/100 g	s_{Rj} ml/100 g
A	16	3	3.657	0.267	0.433
B	16	8	8.229	0.507	0.674
C	16	6	6.009	0.446	0.765
D	16	1	0.455	0.109	0.127
E	16	2	2.282	0.201	0.300

p_j Number of laboratories. s_{rj} Repetition variance.
m_j Total average value. s_{Rj} Comparison variance.

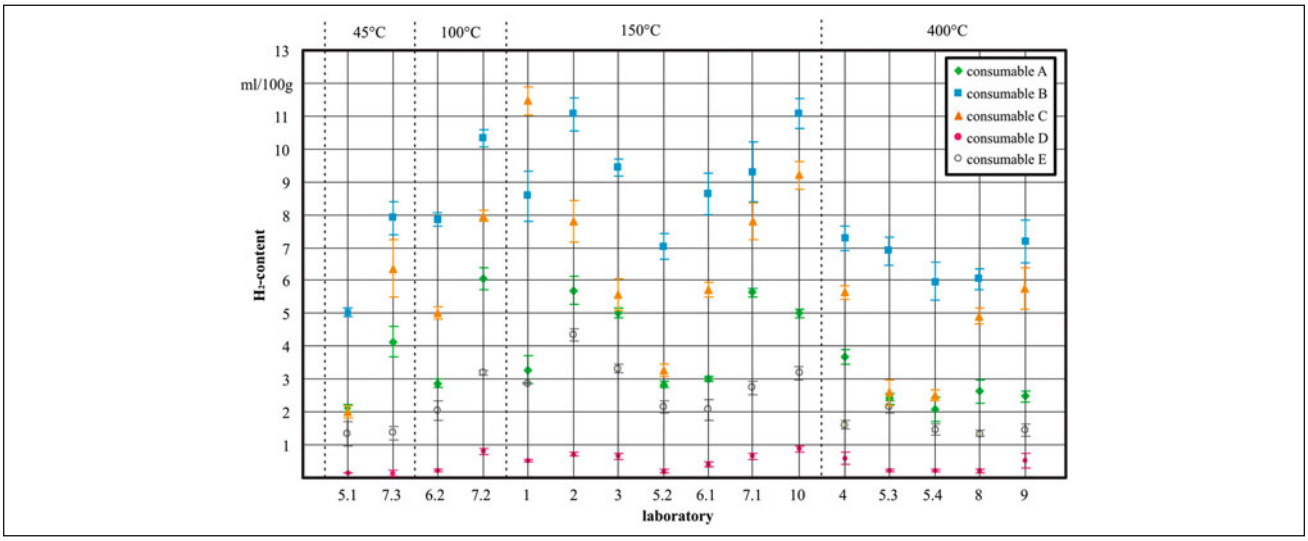


Figure 2 – Average values and standard deviations in individual test series of laboratories 1 to 10 for consumables A to E

$$s_{Lj}^2 = \frac{s_{dj}^2 - s_{rj}^2}{\bar{n}_j} = \text{Variance between laboratories} \quad (3)$$

$$s_{Rj}^2 = s_{rj}^2 + s_{Lj}^2 = \text{Comparison variance} \quad (4)$$

where

n_{ij} is the number of test results in the cell for laboratory i at level j (ISO 5725-2) [9]

y_{ij} is any one of the test results (ISO 5725-2) [9].

3.2 Influence of degassing temperature

Figure 2 represents the average values and standard deviations for the applied filler materials in the individual test series of laboratories 1 to 10.

Degassing temperatures of up to a maximum of 400 °C are found to provide no increased hydrogen contents due to activation of trapped hydrogen. These results demonstrate that up to applied degassing temperatures of 400 °C, hot gas extraction is suitable for determining the diffusible hydrogen content in the weld metals with bcc-lattice structure examined within the scope of this study.

In Figures 3 and 4, the relative standard deviations are compared depending on the average value. As expected, the absolute standard deviations (Figure 3) are seen to increase with increasing hydrogen content. In addition, Figure 4 shows that below a diffusible hydrogen H_D content of approximately 1.5 ml/100 g, high relative standard deviations occur with the TCD analysis techniques used within the scope of this study. For highly cold crack sensitive steels which may experience substantial losses of ductility already at such low hydrogen contents (e.g. high-strength materials with yield strengths exceeding 1 000 MPa), it is therefore recommended to use an alternative measurement technique (e.g. vacuum extraction) for validation.

3.3 Evaluation of influence of welding parameter and hydrogen analysis procedure

The welding parameters used during the weld metal specimen preparation according to ISO/DIS 3690 [1]

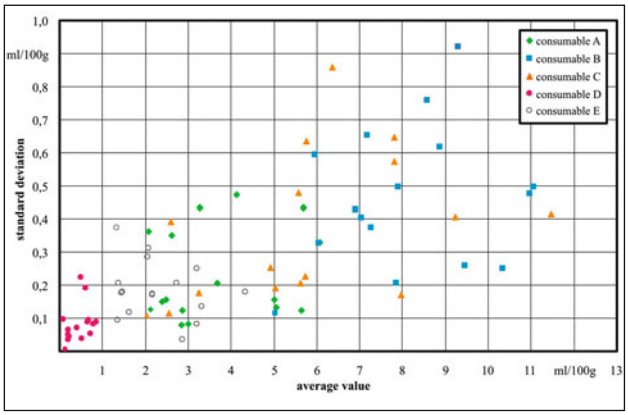


Figure 3 – Absolute standard deviation versus average value of hydrogen content

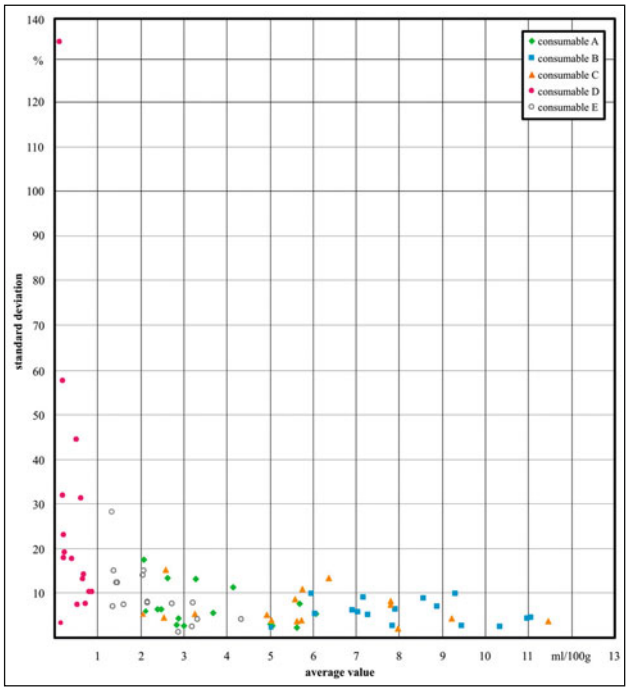


Figure 4 – Relative standard deviation versus average value of hydrogen content

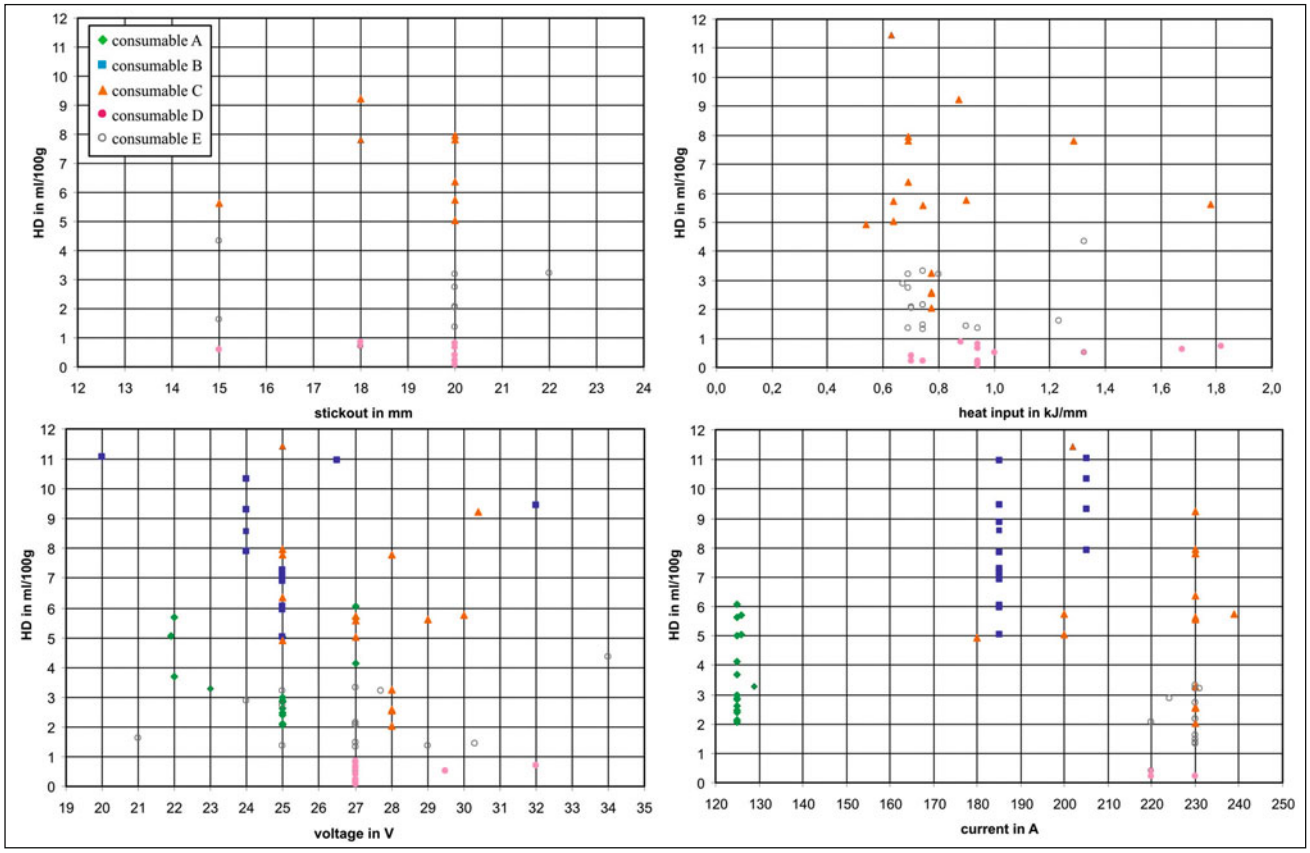


Figure 5 – Diffusible hydrogen content depending on welding parameter

may exert a substantial influence on the level of dissociated hydrogen. The stickout, i.e. the length of unmelted electrode extending beyond the end of the gas nozzle, as well as the heat input (introduced energy per unit length, voltage, current) were evaluated. As can be seen from Figure 5, these welding parameter variations do not affect the measured hydrogen contents.

Furthermore, both the absolute and relative laboratory-specific errors in the total average value of the individual parameter sets were determined according to Equations (6) and (7) in order to evaluate the influence of the hydrogen analysis procedure standardized in ISO/DIS 3690 [1].

Absolute laboratory-specific error: $\bar{y}_{ij} - \hat{m}_j$ (5)

Relative laboratory-specific error: $f = \pm\left(\frac{\bar{y}_{ij} - m_j}{m_j}\right) \cdot 100\%$ (6)

Figures 6 and 7 represent the absolute and relative laboratory-specific errors in the individual test series. Figure 8 shows additionally the determined relative laboratory-specific errors in the national round robin test [2], in which, as opposed to this international round robin test, all weld metal specimens for the diffusible hydrogen analysis were prepared by the same laboratory and transported in liquid nitrogen to the participants within the maximum storage times [1]. Based on these data, a comparison between the international round robin test results (Figure 7) and the national round robin test results (Figure 8) shall provide information about the influence of the test parameters specified in the codes for specimen preparation and welding experiments on the analysis result. A salient feature is the smaller errors, determined relative laboratory-specific errors in the national round robin test compared to international round robin test, since apparently not all influencing parameters are adequately considered in the standards and the hitherto standardized test conditions for the specimen preparation and welding experiments are not sufficiently defined, respectively.

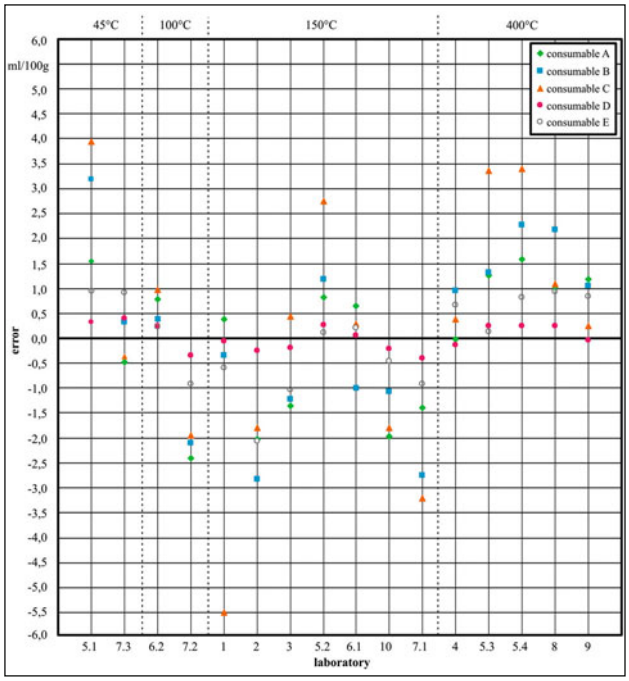


Figure 6 – Absolute laboratory-specific error of total average

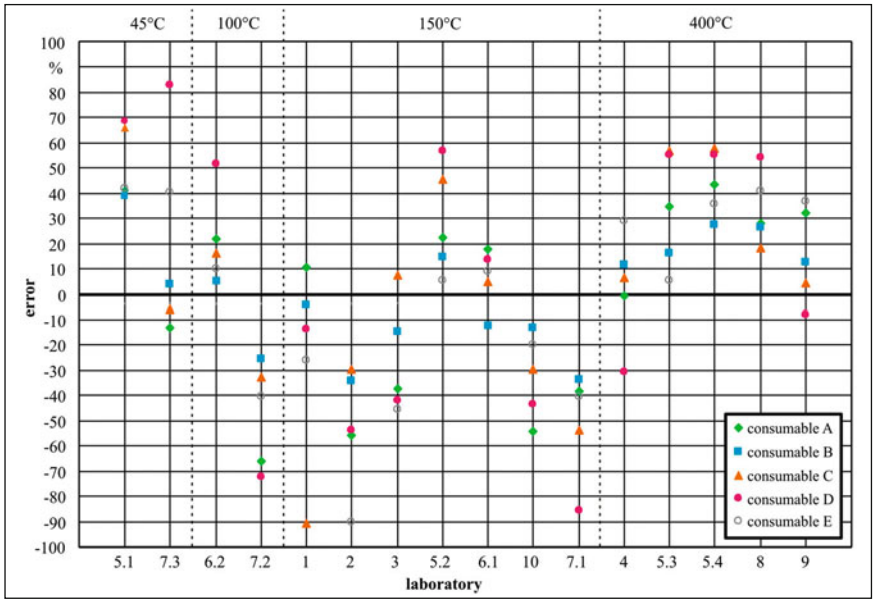


Figure 7 – Relative laboratory-specific error of total average

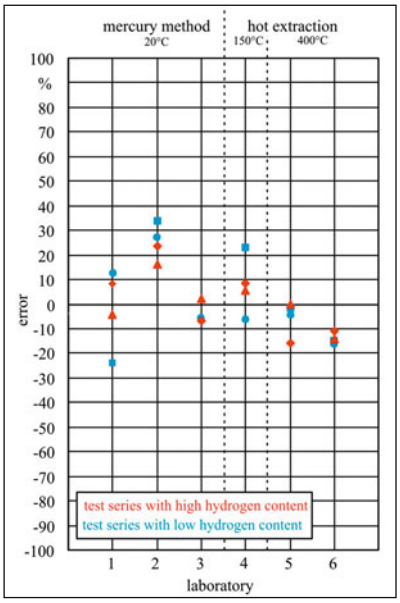


Figure 8 – Relative laboratory-specific error of total average of national round robin test [2]

3.4 Evaluation of influence of hydrogen analysis techniques

Finally, the influence of the applied hot extraction procedure was evaluated. Differentiation shall however be made between static or dynamic measurement systems (see Table 3). The two systems are compared in Figure 9. For the dynamic measurement technique, the chosen degassing temperature was always 400 °C, while for the static measurement technique it was 45 °C, 100 °C and 150 °C, respectively. The comparison shows relatively good agreement of the static with the dynamic method. At the degassing temperature of 45 °C the dynamic method gives slightly higher measured values, while at higher degassing temperatures of 100 °C and 150 °C the static method gives higher measured values.

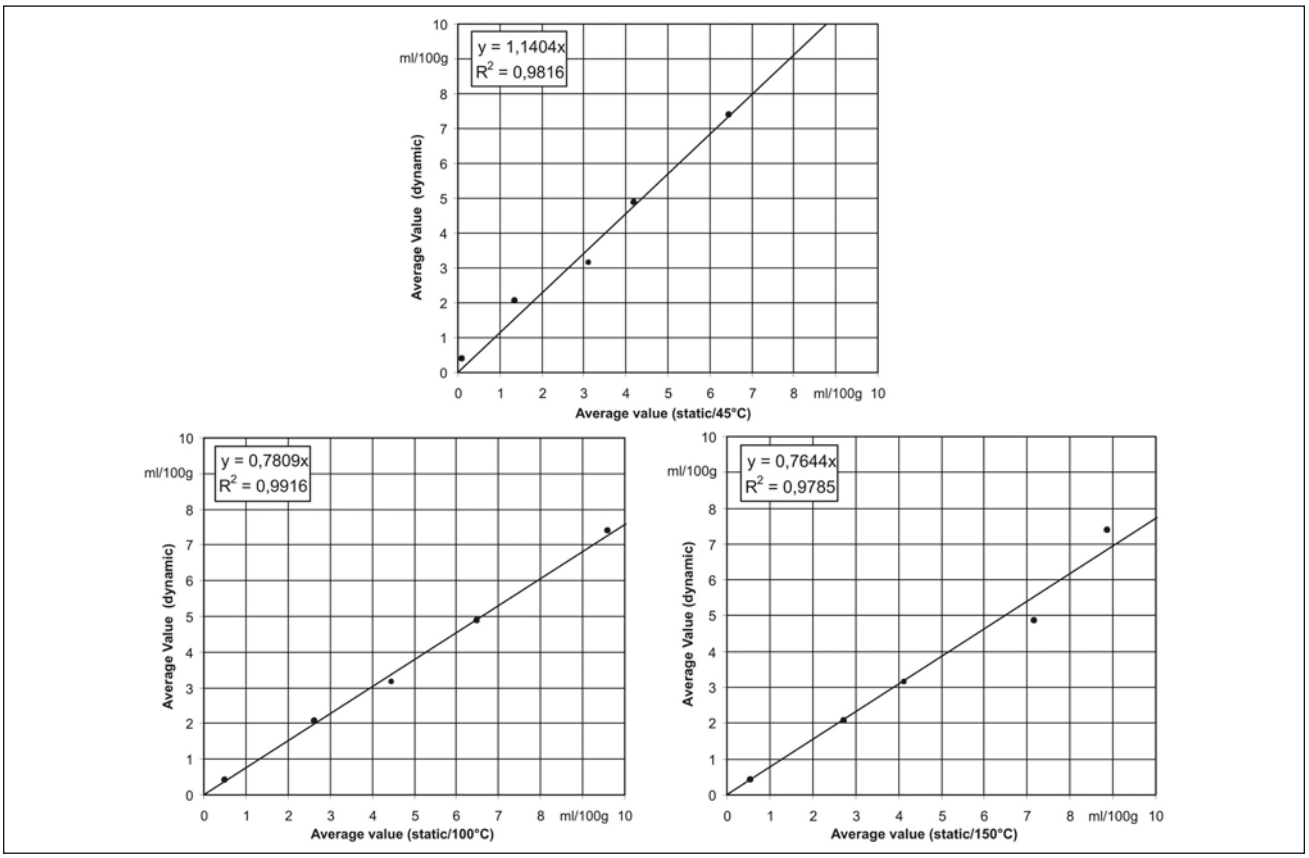


Figure 9 – Comparison between static and dynamic measurement techniques for various degassing temperature

4 CONCLUSIONS

The reported international round robin test served to scrutinize the procedures specified in ISO/DIS 3690:2009 [1] for determining the diffusible hydrogen content in weld metals with bcc-lattice structure. It was specifically intended to check in what respect the specifications defined in the indicated standards for specimen preparation, storage and hydrogen analysis provide comparable measurement results. Five different weld metals with various hydrogen contents were distributed to the ten participants in this round robin test. Various hot extraction installations with thermal conductivity detector (TCD) were applied for the hydrogen analysis. The degassing temperatures were varied between 45 °C and 400 °C and matched to the degassing times. The following results were registered:

– Degassing temperatures ranging between 45 °C and 400 °C do not lead to an increase in the measured contents of diffusible hydrogen in the weld metals with bcc-lattice structure investigated within the scope of this study.

– The applied filler materials were in compliance with the producer specifications. The selected welding parameters such as stickout and heat input did not reveal any influence on the measured diffusible hydrogen content.

– Static and dynamic measurement techniques may yield different results.

– Hydrogen analyses using hydrogen contents below H_D = 1.5 ml/100 g lead to considerable relative standard deviations. For extremely cold crack sensitive steels which experience substantial losses of ductility already with such low hydrogen contents (e.g. high-strength materials with yield strengths exceeding 1 000 MPa); validation therefore recommends an investigation of the complete process (sample generation, preparation, storage and handling as well as analysis procedure).

– Furthermore, the absolute and relative laboratory-specific errors in the total average value of the individual test series were determined in order to evaluate the standardized conditions and influencing parameters for specimen preparation and storage. Compared to the national round robin test [2], a greater measuring error is found, since apparently not all influencing parameters are adequately considered in the standards and the hitherto standardized test conditions for specimen preparation and welding experiments are not sufficiently defined.

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