



Challenges in Pre-Qualification Corrosion Testing of CRAs
based on ASTM G48

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ABSTRACT

The ASTM G48-A test (ferric chloride test) is widely used for pre-qualification of corrosion resistant alloys, welds and weld overlays for oil and gas industry. The test methods and their acceptance criteria vary between the different oil companies, and all the described methods leave some details to the test laboratory, details that may have decisive influence on the result. On this basis, coupons sometimes fail the test on a questionable basis, and materials must often be re-tested several times, or alternatively the involved companies may agree on a waiver. Such circumstances may lead to costly delays in the project schedule. The issues have been addressed earlier as part of a Nordtest project, where the ASTM G48 test was reviewed closely in order to establish and validate an improved method for testing weld coupons. The proposed test method has not yet been widely accepted or implemented, but the principles are sometimes applied when tests show questionable results. The paper presents and discusses experience gained from this project and recent testing of Corrosion Resistant Alloys (CRA), welds and weld overlays. Among the discussed items are coupon cutting and preparation, cut-face pitting, pit identification, selective corrosion and influence of test temperature.

Key words: Ferric chloride, pitting, duplex stainless steel, 6Mo, overlay welds.

INTRODUCTION

The ASTM⁽¹⁾ G48-A test¹ (ferric chloride test) is widely used for pre-qualification of corrosion resistant alloys, welds and weld overlays within the oil and gas industry. A wide range of test methods exist either as standards or company specific instructions. Their acceptance criteria vary, and some of the described methods leave some details to be decided by the test laboratory, details that may have decisive influence on the result. Other effects not described in the procedures have also been observed as part of the testing work. Such effects include selective corrosion of one of the phases in high alloy duplex stainless steels that occasionally occurs for no known reason, and this may not be relevant for an application in neutral chloride solutions.

Pre-qualification of corrosion resistant alloys usually includes several tests, such as chemical analysis, micro examination, ferrite measurement and mechanical testing. The purpose of the G48-A test is to assess whether intermetallic phases or defects, sensitive to corrosion, have been formed during final manufacturing. Generally, the risk of forming detrimental phases during welding or processing is increased with higher content of molybdenum and chromium. Consequently, the G48-A test is mainly required for qualification of duplex and highly alloyed CRAs whereas no additional corrosion testing is requested for standard grades like UNS S31603. For such grades, the intergranular corrosion test (e.g. ASTM A262²) in the certificate of the base material is usually the only documentation of corrosion resistance.

The experience of FORCE Technology⁽²⁾ is that coupons occasionally fail the test on a questionable basis. Consequently, welds often must be re-tested several times, or alternatively the involved companies may agree on a waiver or specific test conditions. Both circumstances may lead to costly delays.

Related problems have earlier been addressed by The Welding Institute⁽²⁾ (TWI)³ and others⁴⁻⁷, who have suggested minor changes to the procedures. However, key elements related to specimen cutting, coupon preparation and interpretation still remain unsolved. It is of common interest for oil companies, weld contractors and testing laboratories that improved test methods are established.

In 2004 a Nordtest⁽³⁾ project was carried out together with Det Norske Veritas⁽²⁾ (DNV) to define and validate an improved method for ASTM G48 testing of weld coupons. The improvements were mainly related to identification of pits and interpretation of pits on cut-faces^{8,9}. However, the proposed test method has not yet been widely accepted or implemented, but the principles are sometimes applied when tests show questionable results, as discussed in this paper.

Corrosion testing of overlay welds has also presented special challenges, especially related to specimen cutting. Such tests are part of the qualification work for submarine pipelines according to DNV-OS-F101¹⁰.

THE FeCl₃ TEST SOLUTION

The test solution used for ASTM G48 practice A is a 6 % (wt) solution of FeCl₃ prepared from FeCl₃•6H₂O. The combination of the acidic solution (pH 1-2), high chloride concentration (3.9 %) and strongly oxidizing nature makes this solution quite aggressive to stainless steel, and ideal for accelerated corrosion testing. The resistance against pitting of stainless steel and CRAs is usually determined by four parameters:

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² Trade name

³ Nordtest, Nordic Innovation, Stensberggt. 25, NO-0170 Oslo, Norway

pH,
Chloride concentration (or other aggressive anions),
Potential (or oxidizing force of solution) and
Temperature

The two first parameters are more or less fixed by the test solution. By using the minimum ratio solution volume to specimen area of 5 ml/cm² specified in ASTM G48, a fixed redox-potential can be expected in the solution that works as a chemical potentiostat. Typically, the obtained redox-potential is in the order of +700 mV SCE. The temperature is the variable parameter defining the corrosivity of the test.

At test temperatures above 45 °C, the ferric ions in the solution tend to precipitate as ferric hydroxide causing a lower pH of the ferric chloride solution⁷. This circumstance may also affect the redox-potential of the solution. Consequently, some procedures prescribe addition of ethylenediaminetetraacetic acid (EDTA) as a complexing agent in order to avoid precipitation³. Other procedures specify pH-adjustment to 1.3 of the FeCl₃ solution.

The ASTM G150¹¹ for determining the CPT of stainless steel at fixed potential (+700 mV SCE) in 1 M NaCl (3.5 % Cl⁻) is an electrochemical test having conditions somewhat comparable to those of ASTM G48-A. The pH-neutral solution used in ASTM G150 represents the main difference between the two test methods.

METHOD VARIANTS

Today testing is based on a wide range of practices. According to the TWI guideline³ the test solution should have an addition of EDTA to avoid precipitation of ferrous products, which is of particular relevance when testing at higher temperatures than approx. 50°C. Moreover, the required amount of test solution has been reduced to 5 ml/cm² in recent G48-editions.

Coupon geometry and preparation of butt welds and base metal involve minor differences between the existing methods¹²⁻¹⁴. Of greatest importance are the various polishing techniques of cut faces as well as pickling. From our point of view the final finish of the cut faces is less important as long as polishing is done wet (without heating). Possible pitting on cut faces is more dependent on a pickling treatment (as used in Norsok¹³), which can dissolve discovered slag particles that otherwise could initiate pitting. Cutting and preparation of weld overlay coupons involve a special case, as discussed separately below.

As for test temperature, this parameter should be defined by the grade of the tested material. The following test temperatures are usually applied:

22Cr duplex base metal	25 °C
22Cr duplex welds	22 °C
25Cr duplex and 6Mo welds	40 °C
25Cr duplex and 6Mo base metals and Ni-clad	50 °C

The exposure time ranges from 72 hours in the original G48 standard¹ to 24 hours for the weld pre-qualification tests^{3,10-14}. This parameter is not regarded as crucial for pitting test in contrast to crevice corrosion testing that involves a certain initiation time.

The most important issues in the Nordtest project^{8,9} have been evaluation and acceptance criteria. It appears that the allowable weight loss ranges from 1 g/m² (ASTM¹²) via 4 g/m² (Oil companies^{13,14}) to 8 g/m² (TWI³). The obtained weight loss strongly depends on coupon preparation (pickling). Therefore this parameter was evaluated closely in the project. Moreover, all the listed practices define that no

pitting must occur but lacks a precise definition or technique for detecting pits. Since location of pits on an irregular weld surface may be difficult, this issue was evaluated closely to propose a safe method.

TEST SPECIMENS OF WELD OVERLAYS

Testing of weld overlays poses particular challenges to the geometry of the test specimen and the ASTM G48 standard does not offer guidance. Industry-specific standards agree that for weld procedure qualification of weld overlays the surface of the test specimen shall be representative for the weld overlay at the minimum distance from the fusion line to be qualified¹⁰. However the importance of other aspects is often not taken sufficiently into account.

Due to impurities mixed into the overlay from the base material the distance from the fusion line between first pass and the base material is very important for the corrosion properties of weld overlays. Great care should be taken when determining the distance from the fusion line to the test specimen. In many cases it will be necessary to prepare the cut face of the sample perpendicular to the welding direction as a macro before the base material is removed by machining. As illustrated in Figure 1, the fusion may fluctuate several millimeters. Proper preparation before machining ensures that the distance from base material to the test specimen can be determined precisely and that the surface of the final specimen will correspond to the minimum distance from the fusion line to be qualified.

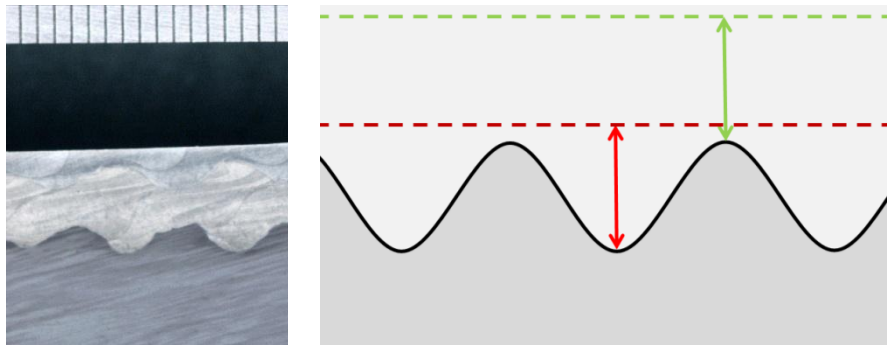


FIGURE 1: Fluctuation along fusing line between overlay weld and substrate.

When the minimum distance from the fusion line that is to be qualified has been determined, the test specimen should always be machined from the material above this distance ensuring that the specimen surface closest to the base material corresponds to the overlay thickness to be qualified. The corrosion resistance of weld overlay can vary significantly with thickness and if the test specimen is machined in a manner that includes material below the thickness to be qualified, one risks failing the overlay on a wrong basis.

A special problem often encountered in practice is that initial non-destructive tests required to qualify the weld are to be performed at the same distance as the ASTM G48 test. In practice this means that the workshop that is going to machine the test specimen for G48 testing receive a coupon where the material they need for machining test specimens for G48 test is no longer available.

Common applications for weld overlays are pipes and pipe fittings made from high strength carbon steel to be used in subsea applications. Because the thickness of the weld overlay that needs to be qualified often is relatively small, e.g. 2 or 3 mm, the curved surface of the pipe or fitting introduce special considerations to preparation of test specimens.

As illustrated in Figure 2a, the ideal shape of the test specimen is curved parallel to the surface of the pipe or fitting. This shape ensures that the entire surface closest to the base material correspond to the

surface to be qualified. However, subsequent preparation of the test specimen before testing e.g. polishing can be a very difficult task on a curved surface.

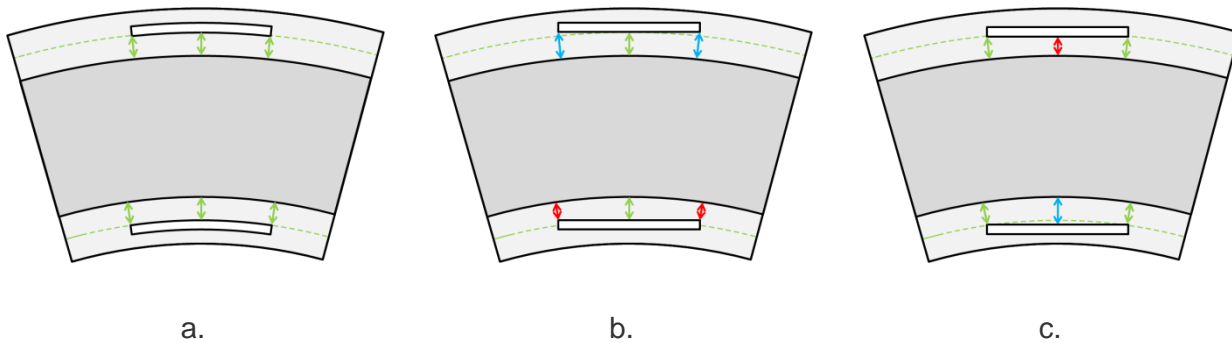


FIGURE 2: Cutting of overlay weld test specimens from curved surfaces.

Test specimens with parallel sides are normally accepted but the specimens will have a less area corresponding to the thickness of the overlay to be qualified, figure 2b top and 2c bottom. To minimize the introduced error the width of the test specimens can be reduced and the length (parallel to the pipe or fitting) increased to ensure sufficient area. The required specimen size is typically 25x25x2 mm¹⁰ but very often a longer and narrower specimen will give better resemblance to the surface on the final product.

Test specimens for corrosion test of weld overlays are to be machined very differently from test specimens for testing base materials or butt welds. Most industry standards are unclear on how to prepare specimens and in practice decisions are often left to the test laboratories. Potentially the decisions made can be the difference between a passed or failed test.

To ensure documentation it is very important that the test reports issued by the test laboratory include detailed information about the procedures applied for preparing test specimens. Preferably the industry standards should provide the laboratory with detailed procedures upfront.

When the above issues are resolved, G48 corrosion testing of overlay welds is straightforward. In most cases the specimen passes the test without any detectable weight-loss.

EXPERIMENTAL

A series of G48 tests including welds made from thin- to thick-walled pipe of super duplex stainless steel (UNS S32750) are discussed below to illustrate the issues encountered during pre-qualification work. This work led to the proposed Nordtest method⁸.

Coupon Preparation

The tested materials included six different welds (A – F) of UNS S32750. The materials were deliberately selected to include both high quality welds and discarded or questionable welds to cover a wide range of weld qualities. Identical weld coupons measuring approximately 25x50 mm were cut out of each weld. The cut-faces were wet ground to #120. Some of the coupons were pickled using either: a) paste pickling of cap face with paste containing 5-7 wt% HF and 20 wt% HNO₃ for 60 min at ambient temperature, or b) dip pickling in 5 wt% HF + 20 wt% HNO₃, 60°C, 5 min.

ASTM G48 Exposure

The coupons were exposed individually for 24 hours in the standard ferric chloride test solution containing 100 g FeCl₃·6H₂O in 900 g H₂O. The test temperature was either 35°C (welds A, B and F) or 40°C (welds C, D and E). The volume of the solution was at least 20 ml/cm² in respect to the coupon area.

Electrochemical Measurements

Electrical connection to the weld coupon was made using a spring-loaded titanium connector (wire) on the glass cradle. Tests were made in ferric chloride test solution and included three techniques: a) corrosion potential measurement (OCP) with saturated calomel electrode, b) cathodic sweeps at a scan rate of 6 mV/min and c) CPT determination in accordance with ASTM G150¹¹.

Evaluation

All exposed coupons were evaluated by weight loss determination and examination under a stereomicroscopy at 10-50x magnification. Additional evaluation for some coupons included dye penetrant (DP) examination or re-exposure (propagation test) for 24 hours.

RESULTS AND DISCUSSION

The first part involved a comparative study using different techniques for coupon preparation and evaluation. In addition, electrochemical measurements were made on selected coupons. Table 1 summarizes the results of these tests.

It was noticed that the welds with small wall thickness (groups D and E) pass the test with low weight losses well below the 1.0 g/m² criterion. Coupon preparation (i.e. pickling) does not affect the obtained weight loss significantly. Consequently, there was no need for additional examination of these coupons, i.e. dye penetrant and metallographic examination.

The thick-walled welds (A and B) showed higher weight losses. However, weld A did not show pitting for any of the six tested coupons, which was confirmed by dye penetrant and propagation tests. However, in three cases the weight loss exceeds 1.0 g/m², which in some practices would fail the coupon. Pickling with pickling paste (partly removing surface oxides) did not affect the weight loss significantly, whereas dip pickling (surface oxides removed) or propagation testing result in much lower weight loss. On this basis, the high weight loss observed for some of the coupons is ascribed to a pickling effect of surface oxides during exposure in the ferric chloride test solution.

Weld B fails in all tests when using the 1.0 g/m² weight loss criterion. Metallographic examination showed that the weld root suffered from severe corrosion, possibly due to precipitation of detrimental phases.

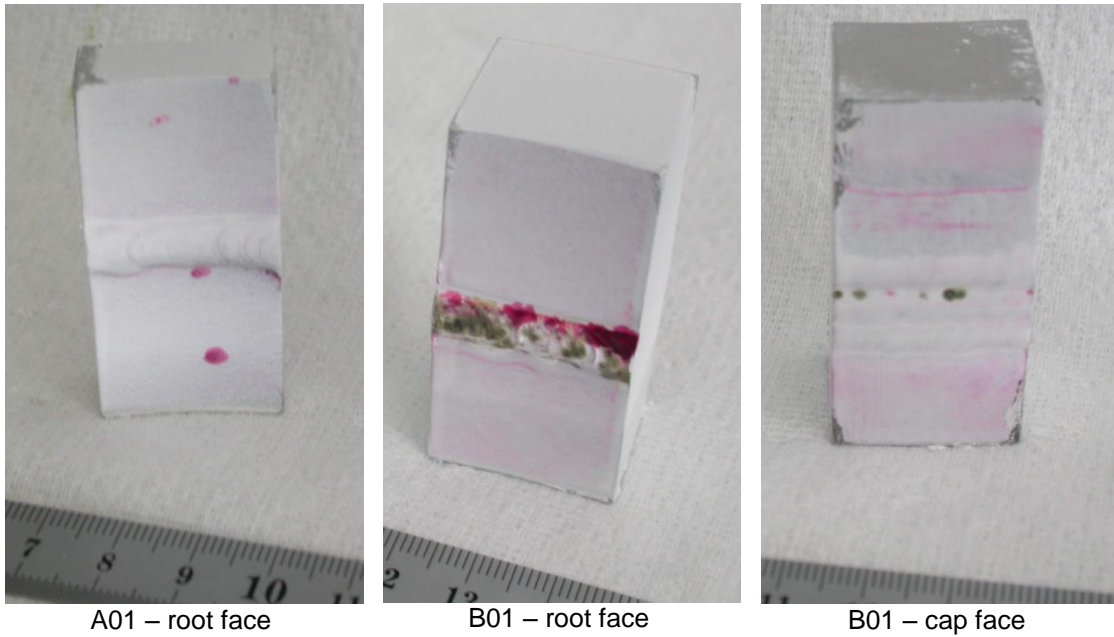
None of the tested coupons showed pitting on cut-edges despite that the prepared finish (120-grit) was coarser than some practices.

TABLE 1.
Result summary of exposure tests and electrochemical measurements.
All tests were made in 6% FeCl₃ at 35°C (A and B) or 40°C (D and E).

ID	Pickling	Thick-ness	Area	Weight loss			OCP Avg.	Pits		CPT ^d ASTM G150 ID: °C
				Pickling	Exposure	Propagat.		x20	DP	
		mm	cm ²	g/m ²	g/m ²	g/m ²	mV SCE			
A01	No	11.0	42.94		1.40	0.00	580 ^a	0	0	A07: 61
A02	No	11.2	43.25		0.53			0		
A03	Paste	11.0	43.66	-0.02	1.05	0.00		0	0	
A04	Paste	11.5	44.29	0.52	1.63	0.00		0	0	
A05	Dip	11.0	43.30	1.34	0.09			0		
A06	Dip	11.6	44.43	0.68	0.20			0		
B01	No	22.0	58.94		35.3		428	Many	Many ^c	B07: 33 B09: 43
B02	No	23.0	61.04		48.0			Many	Many	
B03	Paste	22.0	59.88	3.17	49.3			Many		
B04	Paste	23.0	62.05	3.22	43.5			Many	Many	
B05	Dip	22.0	60.82	3.78	3.29	0.66	598 ^b	0	Few ^c	
B06	Dip	22.3	63.81	3.60	24.8			Many	Many	
D01	No	2.5	27.44		0.11		695	0		
D05	No	2.7	26.21		0.04			0		
D02	Paste	2.5	27.44	0.33	0.22			0		
D06	Paste	2.8	26.98	0.33	0.07			0		
D03	Dip	2.5	27.70	0.36	0.14			0		
D07	Dip	2.9	28.66	0.45	0.07			0		
E01	No	6.0	30.92		0.45		685	0		
E07	No	6.3	29.51		0.10			0		
E02	Paste	6.0	30.92	1.07	0.06			0		
E08	Paste	6.4	28.75	0.66	0.24			0		
E03	Dip	6.0	30.92	1.46	0.10			0	E04: 66	
E09	Dip	6.4	30.93	0.78	0.13			0	E05: 62	

a) 687 mV in propagation test b) in propagation test c) additional corrosion was identified by dye penetrant (DP) examination d) CPT is read at 100 $\mu\text{A}/\text{cm}^2$.

Additional dye penetrant examination (DP) was applied on eight coupons when the weight loss indicated corrosion. This technique proved beneficial by revealing pits overlooked by microscopy in some cases. Figure 3 shows examples of DP-tested coupons. Similarly, propagation testing also proved useful by identifying pickling effects in some cases (A01, A03 and A04).



A01 – root face B01 – root face B01 – cap face
FIGURE 3: Exposed coupons after dye penetrant (DP) testing. Indications on A01 are not due to corrosion. DP on B01 revealed corrosion overlooked by microscopy.

The OCP measurements were performed for some of the coupons during exposure in the ferric chloride solution. It appears from Figure 4 that the passive coupons (D01 and E01) obtain a very stable potential of 685 to 695 mV SCE within five hours of exposure regardless of the test temperature being either 40 or 50°C. In comparison to this, the actively corroding coupon (B01) shows an unsteady corrosion potential in the range of 400-500 mV SCE. Coupon A01 falls in between, having a corrosion potential of approx. 600 mV SCE. This coupon showed no pitting but a considerable weight loss due to a pickling effect. The observed potential behavior shows that pickling effects depolarize the coupon, which may result in too optimistic test results.

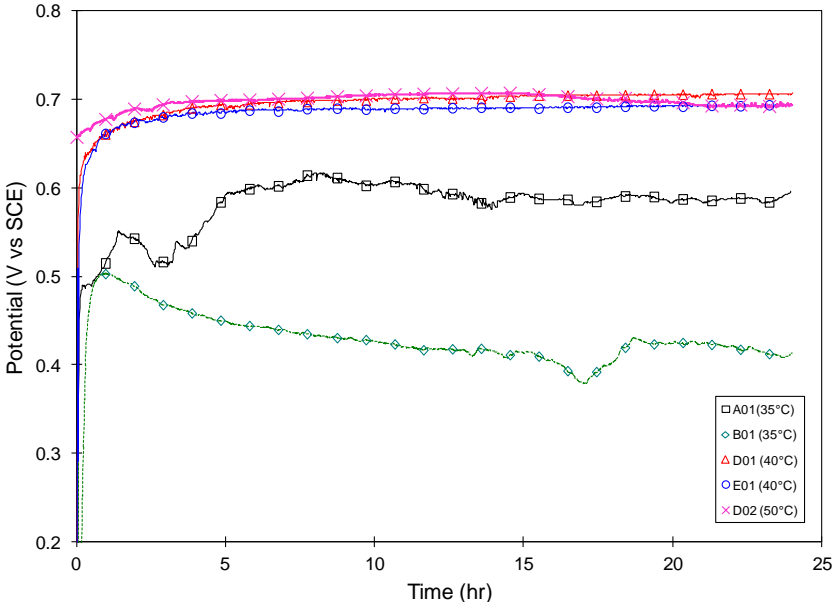


FIGURE 4: Development in corrosion potential during exposure in ferric chloride test solution. A01 and B01 show signs of active corrosion (i.e. depolarization) whereas D01, E01 and D02 show a stable passive behavior.

Coupon D02 was re-exposed at 50°C to see how the corrosion potential is affected by a higher test temperature in the range where precipitation of ferric hydroxide sometimes occur. Precipitation did not occur, and it appears that the potential level is similar to that recorded at 40°C, Figure 4.

One coupon was polarized cathodically to evaluate the cathode properties of stainless steel after 18 hours exposure in ferric chloride. Figure 5 compares this polarization curve with data points calculated from weight loss and average OCP during exposure tests in Table 1. Although only limited correlation is observed, the curves give an impression of the expected depolarization of coupons at different corrosion rates during exposure in ferric chloride.

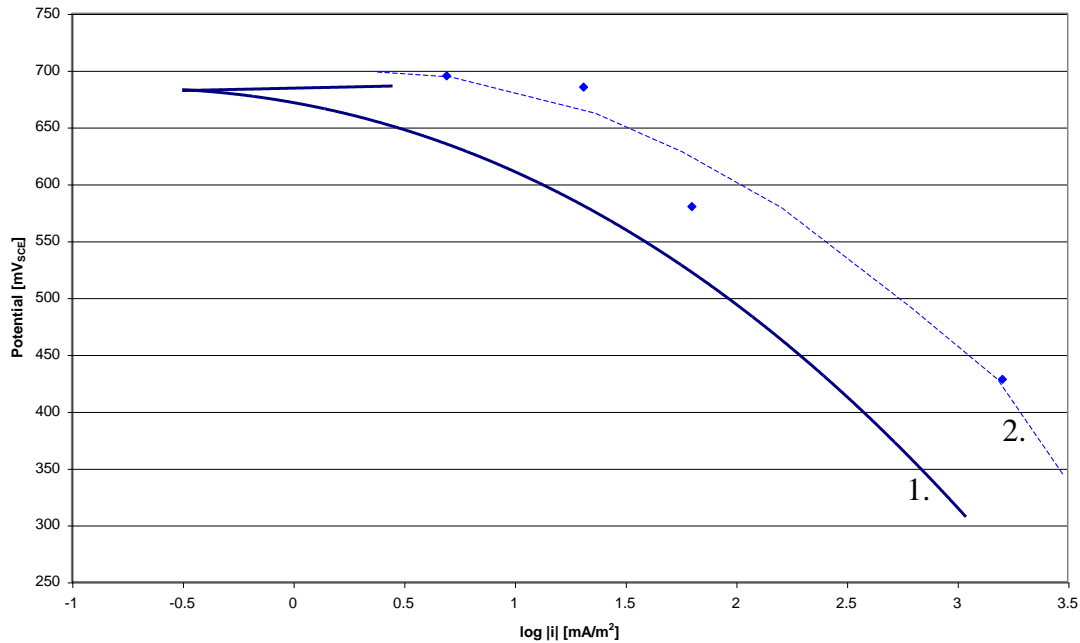


FIGURE 5: Recorded polarization curve (1) compared with cathodic curve (2) calculated from weight loss and OCP measurements of FeCl₃-exposure tests at 35 or 40°C.

The ASTM G150 test for determination of CPT was applied to evaluate whether the observed behavior in acidic ferric chloride correlated with that obtained in pH-neutral media. Figure 6 shows the CPT curves of welds A, B and E. CPT was read at 100 $\mu\text{A}/\text{cm}^2$.

It appears from Table 1 that there is good agreement between the measured CPT and the results of the exposure tests. In addition, the reproducibility of the CPT test was good. However, dip-pickling was necessary to obtain a distinct transition from passive condition to pitting. For comparison with the obtained values, Ames et al^{15,16} report CPT's between 50 and 60°C for welded UNS S32750 material, whereas CPT of the base metal is 85°C.

The CPT test provides a rapid and quantitative method for assessing the pitting resistance of welds. Thus, this technique may possibly be used as an alternative to G48-testing in doubt cases. However, more work needs to be done to establish suitable acceptance criteria for this technique.

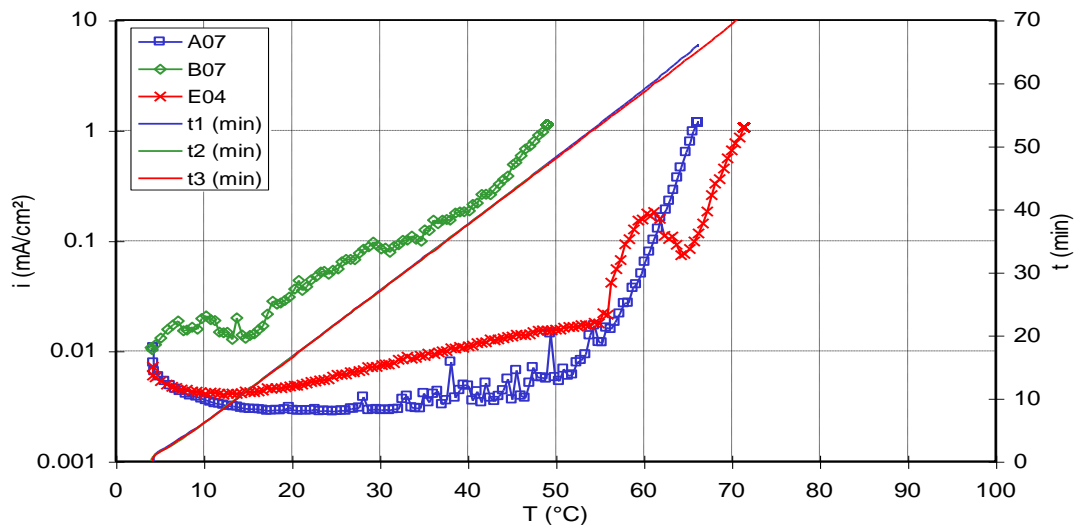


FIGURE 6: ASTM G150 CPT curves in 1M NaCl for welds A, B and E. Coupons were dip-pickled before test.

Based on the above results minor changes were introduced in the experimental procedure proposed for the Nordtest method⁸. Basically, these changes are related to two major recognitions:

- Pits on cut-faces and pickling effects depolarize the coupon, thereby compromising the test result
- Pits may be overlooked by microscopy

For these reasons it is recommended the coupon be dip-pickled before exposure. Alternatively, if pickling is undesirable the weight loss after exposure must still be less than 4.0 g/m² in order to approve the weld. At higher weight losses and no pitting, the non-pickled coupon shall be subjected to a propagation test for 24 hours.

Evaluation shall include dye penetrant (DP) examination in combination with microscopy. Unacceptable pits are defined as DP indications where subsurface attack is observed when probing with a needle.

The presence of pits on faces apart from the test face may be ignored if the possibility of depolarization during exposure can be excluded (this is a repeated sentence). For this purpose it shall be verified by measurement of depth and diameter that the total pit volume (V_{pits}) is less than V_{max} , given by:

$$V_{\text{max}} [\text{mm}^3] = \frac{\text{Area} \cdot W_{\text{max}}}{\text{Dens}} = \frac{\text{Area} [\text{cm}^2]}{78}$$

where:

Area = coupon area

W_{max} = weight-loss without depolarization (1.0 g/m²)

Dens = steel density (7.8 g/cm³)

This equation has been established from the OCP data and cathodic polarizations by defining a maximum depolarization of 100 mV.

Other indications that look like pits (e.g. dissolved slags or small weld pores etc.) may be ignored if:

- The presence of the imperfection was recorded before exposure, or
- The indication appears as “just visible” in the dye penetrant test

TABLE 2.
Criteria for evaluating of the exposed coupon in the Nordtest method⁸.

Dye penetrant indications		Weight loss	
		$\leq 4 \text{ g/m}^2$	$> 4 \text{ g/m}^2$
None		Pass	Propagation test
Indications but no pits		Pass	Propagation test
Pits on faces apart from test face	$V_{\text{pits}} \leq V_{\text{max}}$	Pass	New test
	$V_{\text{pits}} > V_{\text{max}}$	New test	
Pits on test face		Fail	

TABLE 3.
Result summary of second series of exposure tests performed in accordance with the Nordtest method⁸.

ID	Thick-ness	Area	Test temperature	Weight loss Exposure	No of pits DP	Test result
	mm	cm ²	°C	g/m ²		
C02	6.4	30.1	40	0.000	0	pass
C03	6.2	29.3	40	-0.034	0	pass
C04	6.3	31.0	40	0.000	0	pass
F01	34.8	98.3	35	6.202	3+	fail
F02	34.6	99.8	35	13.931	8+	fail
F03	34.8	102.1	35	17.048	5+	fail
F04	34.8	103.2	35	2.325	4	fail

The above approach is summarized in Table 2 and Figure 7. The applicability was tested with a second series of coupons presented in Table 3. Welds with small wall thickness (weld C) passed the test with low weight losses. The absence of pitting on these coupons was easily verified with the dye penetrant technique. No false indications were observed. The thick-walled welds (weld F) showed pitting in all cases and significant weight loss. By using the dye penetrant technique all pits were easily located. Although none of the tested welds were in the doubtful range, the experience with the new procedure from the second series of exposure tests was satisfactory.

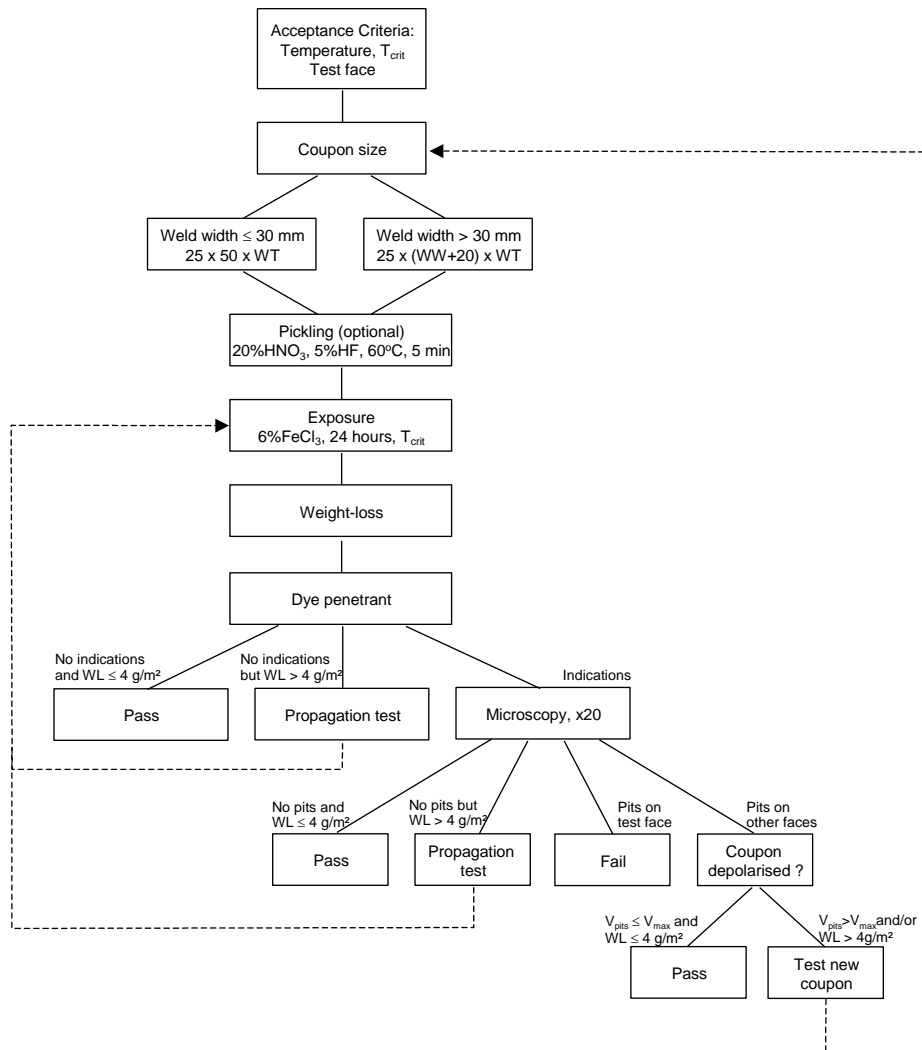


FIGURE 7. Decision diagram for ASTM G48 pre-qualification testing of welds according to the proposed Nordtest procedure⁸.

Even though the Nordtest procedure has been available for some time, it is not used or requested by the industry yet. However, the results gained in this work have occasionally provided a good basis for interpretation of tests in doubt cases.

Some may question whether pits on cut-faces never exposed to media can be permitted at all, even if they fulfill the limits given in Table 2. Presence of such pits may imply presence of brittle intermetallic phases inside the metal or weld, but usually the mechanical tests would detect this. Cut-face pits may also be caused by weld pores that are unacceptable too. However, by using the depolarization criterion for defining the allowable size of such pits, a reasonable safety margin can be expected.

CONCLUSIONS

The paper reviews potential problems associated with G48 testing for prequalification of base metals, weld and overlay welds of CRAs.

For overlay welds a detailed procedure for cutting the specimens must be established for each individual geometry of pipe or fitting to be tested.

Testing of butt welds or base materials has identified two potential problems:

- Pits on cut-faces and pickling effects depolarize the coupon and may thereby result in too optimistic test results,
- Pits may be overlooked by microscopy

To account for this, the Nordtest procedure was established and proposed together with DNV. The method includes two major additions in comparison to existing practices:

- Locating and identifying pits on the exposed coupon shall include dye penetrant examination in combination with microscopy.
- Pits on faces apart from the test face may be accepted if the possibility of depolarization can be excluded by volumetric assessments.

In addition, dip-pickling of the coupon prior to exposure is recommended, but still optional. The method includes a decision diagram as well as clear acceptance criteria to ensure a high degree of consistency in the test results.

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