# CORROSION TESTING OF WELDS, A REVIEW OF METHODS



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# FOREWORD

This document has been produced by experts in the field of corrosion testing and corrosion resistant alloys under the auspices of sub-commission IX-H of the International Institute of Welding (IIW). It is intended to be a useful reference guide to standardised corrosion tests, listed in numerous national and international standards. Most corrosion tests were originally developed to assess various corrosion properties of parent metals and alloys. Many of these tests have been modified or adapted for use with weld metals and welded joints and this document is designed to act as a summary of the features of the various tests in the context of welds and welded joints. It is not intended to be a text book on corrosion testing but it is hoped that it will prove to be a useful aid to welding engineers and materials engineers who are not corrosion specialists in their own right. It is important to recognise that this document is a summary of published standards and should not be used as a substitute for such standards. The user is strongly advised to refer to the original standards to confirm precise and specific details.

*IIW-Thesaurus keywords:* Austenitic stainless steels; Carbon steels; Corrosion; Corrosion tests; Crevice corrosion; Duplex stainless steels; Fatigue strength; Galvanic corrosion; Intergranular corrosion; Low alloy steels; Mechanical properties; Nickel alloys; Pitting corrosion; Stress corrosion; Stainless steels; Steels; Unalloyed steels; Welded joints; Weld metal.

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# **2 INTRODUCTION**

A number of tests are used to establish the corrosion resistance of stainless steels and nickel alloys. Several methods are found in national and international standards but procedures are also employed that are specific for individual laboratories and for manufacturers of corrosion resistant alloys.

This document is a statement of different common standards and methods used for corrosion testing of stainless steels and nickel alloys, with particular emphasis on weldments and weld metal. Since welding is a major joining method for fabrication of equipment, weldments should be included as much as possible in test programmes. It concentrates on wet corrosion environments and does not cover high temperature corrosion in gaseous environments. It is also important to bear in mind that accelerated corrosion tests give indicative results only.

The expression "as welded" in this document, refers to a non post weld heat treated weldment for which slag and oxides have been removed. For the respective test method, "key headlines" will be followed in order to obtain rapid information about the intention of a certain test method and its applicability for weldments.

# The "key headlines" are *Materials, Test solution, Procedure and Weldments*.

The forms of corrosion considered are:

- General corrosion
- Pitting corrosion
- Intergranular corrosion
- Crevice corrosion

- Stress corrosion
- Corrosion fatigue
- Galvanic corrosion
- Field testing

# **3 LIST OF STANDARDS**

Page	Nr	Standard	Materials	Solution	Comments
4.1 G	eneral	corrosion			
8	4.1.1	ASTM G 1: Standard Practice for Preparing, Cleaning and Evaluating Corrosion Test Specimens	Bare solid metal specimens	Not applicable	
8	4.1.1	ASTM G 31: Standard Practice for Laboratory Immersion Corrosion Testing of Metals	Bare solid metal specimens	To be selected	Suitable for weldments
8	4.1.2	JIS G 0591: Method of sulphuric acid test for stainless steels	Stainless steels	H <sub>2</sub> SO <sub>4</sub> , 5 – 50 %	Suitable for weldments
8	4.1.3	ASTM G 157: Standard Guide for Evaluating the Corrosion Properties of Wrought Iron- and Nickel- Based Corrosion Resistant Alloys for the Chemical Process Industries	Wrought iron- and nickel alloys	Fourteen environments that are described in the standard	Suitable for weldments in as-welded condition. Can also be used with other environments than those given in the standard. Most used standard for testing general corrosion
8	4.1.4	MTI-1 Test Method: Laboratory Testing of Iron- and Nickel-Based Alloys for Corrosion Resistance in Selected Media	Wrought iron- and nickel-based alloys	Fourteen corrosive environments consisting of four inorganic acids, two organic acids, sodium hydroxide and two acid mixtures	Intended for as- manufactured and as- welded specimens in corrosive systems
4.2 Pit	tting co	rrosion	·		
9	4.2.1	ASTM G 48: Standard Test Methods for Pitting and Crevice Corrosion Resistance of Stainless Steels and Related Alloys by use of Ferric Chloride Solution	Bare solid metal specimens	6 % FeCl <sub>3</sub> solution and 6 % FeCl <sub>3</sub> + 1 % HCl solution	Can be used for all-weld- metal and weldments As- welded or post weld treated
9	4.2.2	JIS G 0578: Method of ferric chloride test for stainless steels	Stainless steels	6 % FeCl <sub>3</sub> solution	Same as for 4.2.1
10	4.2.3e	ASTM G 61: Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion Susceptibility of Iron-, Nickel-, or Cobalt-Based Alloys	Iron-, nickel-, or cobalt-alloys	Chloride environment	Can be used for weld metal and weldments. Not as suitable as 4.2.4

Page	Nr	Standard	Materials	Solution	Comments
10	4.2.4e	ASTM G 150: Standard Method for Electrochemical Critical Pitting Temperature Testing of Stainless Steels	/ I	1M NaCl	Suitable for weldments. Can be used on as-welded surfaces
10	4.2.5	ISO 11463: Corrosion of metals and alloys – Evaluation of pitting corrosion	General metals and alloys	Not applicable	Guidance in examination and evaluation of pits
10	4.2.6e	ISO 17864: Determination of the critical pitting temperature under potentiostatic control	Austenitic stainless steels, Duplex (Austenitic-Ferritic) stainless steels and other related alloys	1M NaCl	Suitable for weldments. Can be used on as welded surfaces. ASTM G150 is more referred to
10	4.2.7	ASTM A 923-C: Ferric chloride corrosion test for classification of structures of duplex stainless steels	Duplex (Austenitic- Ferritic) stainless steels	6 % FeCl <sub>3</sub> solution	Not to be confused with ASTM G 48. This method does not determine the critical pitting temperature. It is designed solely for detection of detrimental intermetallic phases in duplex stainless steels
4.3 In	tergran	ular corrosion			
10	4.3.1	ASTM A 262 Practice B: Ferric Sulphate-Sulphuric Acid Test for detecting susceptibility to Intergranular Attack in Austenitic Stainless Steels	Austenitic stainless steels	Approximately 3 % $Fe_2(SO_4)_3$ and 50 % $H_2SO_4$	Suitable method for weld metal and weldments
11	4.3.2	ASTM A 262 Practice C: Nitric Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels	Austenitic stainless steels	HNO <sub>3</sub> 65 %	Suitable method for weld metal and weldments for use in strongly oxidizing environments
11	4.3.3	ASTM A 262 Practice E: Copper – Copper Sulphate – 16 % Sulphuric Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels	Austenitic stainless steels	6 % CuSO <sub>4</sub> + 16 % H <sub>2</sub> SO <sub>4</sub>	Suitable method for weld metal and weldments
11	4.3.4	ASTM A 262 Practice F: Copper – Copper Sulphate – 50 % Sulphuric Acid Test for Determining Susceptibility to Intergranular Attack in Austenitic Stainless Steels	Austenitic stainless steels	10 % CuSO <sub>4</sub> + 50 % H <sub>2</sub> SO <sub>4</sub>	Suitable method for weld metal and weldments
12	4.3.5	ISO 3651-1: Determination of resistance to intergranular corrosion of stainless steels – Part 1: Austenitic and ferritic-austenitic (duplex) stainless steels – Corrosion test in nitric acid medium by measurement of loss in mass	stainless steels	HNO <sub>3</sub> 65 %	Corresponds to 4.3.2. Suitable method for weld metal and elements for use in strongly oxidizing environments

Page	Nr	Standard	Materials	Solution	Comments
12	4.3.6	ISO 3651-2: Determination of resist. to intergran. cor- rosion of stainless steels – Part 2: Ferritic, austenitic and ferritic-austenitic (duplex) stainless steels – Corrosion test in media containing sulphuric acid	Ferritic, austenitic and austenitic- ferritic (duplex) stainless steels	Method A: 16 % $H_2SO_4$ + CuSO <sub>4</sub> Method B: 35 % $H_2SO_4$ + CuSO <sub>4</sub> Method C: 40 % $H_2SO_4$ + CuSO <sub>4</sub>	Suitable method for weld metal and weldments
12	4.3.7	JIS G 0572: Method of ferric sulphate-sulphuric acid test for stainless steels	Austenitic stainless steels	50 % H <sub>2</sub> SO <sub>4</sub> + 4 % Fe <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>	Suitable method for weld metal and weldments
12	4.3.8	JIS G 0573: Method of 65 % $HNO_3$ acid test for stainless steels	Austenitic stainless steels	65 % HNO <sub>3</sub>	Conform to 4.3.2. Suitable method for weld metal and elements for use in strongly oxidizing environments
13	4.3.9	JIS G 0574: Method of nitric-hydrofluoric acid test for stainless steels	Molybdenum- bearing austenitic stainless steels	10 % HNO <sub>3</sub> + 3 % HF	May be used for weldments and weld metal
13	4.3.10	JIS G 0575: Method of copper sulphate-sulphuric acid test for stainless steels	Ferritic, austenitic and austenitic- ferritic(duplex) stainless steels	Method A: 16 % $H_2SO_4$ + CuSO <sub>4</sub> Method B: 35 % $H_2SO_4$ + CuSO <sub>4</sub> Method C: 40 % $H_2SO_4$ + CuSO <sub>4</sub>	Conform to 4.3.6. Suitable method for weld metal and weldments
13	4.3.11	GOST 6032: Methods for determination of intercrystalline corrosion resistance	Austenitic, austenitic- martensitic, austenitic-ferritic, and ferritic stainless steels		Similar to 4.3.5 and 4.3.6. Suitable method for weld metal and weldments
13	4.3.12	ASTM A 763: Standard Practices for Detecting Susceptibility to Intergranular Attack in Ferritic Stainless Steels	Ferritic stainless steels	Method W: Oxalic acid Method X: $Fe_2(SO_4)_3 + H_2SO_4$ Method Y: $H_2SO_4 50 \% + CuSO_4$ Method Z: $H_2SO_4 16 \% + CuSO_4$	Suitable method for weld metal and weldments
13	4.3.13	ISO 9400: Nickel-based alloys – Determination of resistance to intergranular corrosion	Nickel alloys	Method A: $H_2SO_4 + Fe_2(SO_4)_3$ Method B: $H_2SO_4 + CuSO_4$ Method C: HCl Method D: HNO <sub>3</sub>	Suitable method for weld metal and weldments
14	4.3.14	ASTM G 28: Standard Test Methods of Detecting Susceptibility to Intergranular Corrosion in Wrought, Nickel-Rich, Chromium-Bearing Alloys	Nickel alloys	Fe <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> + 50 % H <sub>2</sub> SO <sub>4</sub>	Can be used on weldments and weld metal
14	4.3.15e	GOST 9.914: Electrochemical methods for determination of intercrystalline corrosion resistance	Austenitic stainless steels		Can be used for weld metal and weldments

Page	Nr	Standard	Materials	Solution	Comments
4.4 Cr	evice c	orrosion			
14	4.4.1	ASTM G 48: Standard Test Methods for Pitting and Crevice Corrosion Resistance of Stainless Steels and Related Alloys by use of Ferric Chloride Solution	Stainless steels and nickel-based chromium-bearing alloys	6 % FeCl <sub>3</sub> solution and 6 % FeCl <sub>3</sub> + 1 % HCl solution	Can be used for welds when reinforcements are removed. Flat specimens needed to get a well defined crevice
15	4.4.2	ASTM G 78: Standard Guide for Crevice Corrosion Testing of Iron- Base and Nickel-base Stainless Alloys in Seawater and other Chloride Containing Aqueous Environments	Iron and nickel alloys	Chloride-containing aqueous solutions including seawater	Can be used for welds when reinforcements are removed. Flat specimens needed to get a well defined crevice
15	4.4.3	MTI-2 Test Method: Laboratory Testing of Iron- and Nickel-Based Alloys for Relative Resistance to Crevice Corrosion in a 6 % Ferric Chloride (FeCl <sub>3</sub> ) Solution	Wrought iron- and nickel -based alloys	6 % FeCl <sub>3</sub> solution	Can be used for welds when reinforcements are removed. Flat specimens needed to get a well defined crevice
1.5 St	ress co	rrosion			
15	4.5.1	ASTM G 36: Standard Practice for Evaluating Stress-Corrosion-Cracking Resistance of Metals and Alloys in a Boiling Magnesium Chloride Solution	Stainless steels	45 % MgCl <sub>2</sub>	Can be used on weldments. Not suitable for certain alloys. 4.5.8 is then a better choice
15	4.5.2	ISO 7539-1: General guidance on testing procedures			
16	4.5.2	ISO 7539-2: Preparation and use of bent beam specimens	Metals and alloys	To be selected	
16	4.5.2	ISO 7539-3: Preparation and use of U-bend specimens	Metals and alloys	To be selected	
16	4.5.2	ISO 7539-4: Preparation and use of uniaxially loaded tension specimens	Metals and alloys	To be selected	
16	4.5.2	ISO 7539-5: Preparation and use of C-ring specimens	Metals and alloys	To be selected	
17	4.5.2	ISO 7539-6: Preparation and use of pre-cracked specimens for tests under constant load or constant displacement	Metals and alloys	To be selected	
17	4.5.2	ISO 7539-7: Slow strain rate testing	Metals and alloys	To be selected	
17	4.5.2	ISO 7539-8: Preparation and use of specimens to evaluate weldments	Metals and alloys	To be selected	Developed for testing of weldments

evaluate weldments

Page	Nr	Standard	Materials	Solution	Comments
17	4.5.2	ISO 7539-9: Preparation and use of pre-cracked specimens for tests under rising load or displacement			
17	4.5.3	ASTM G 38: Standard Practice for Making and Using C-ring Stress - Corrosion Test Specimens	Metals and alloys	To be selected	Suitable for testing of weldments
17	4.5.4	ASTM G 39: Standard Practice for Preparation and Use of Bent-Beam Stress-Corrosion Test Specimens	Metals and alloys	To be selected	Suitable for testing of weldments
18	4.5.5	ASTM G 30: Standard Practice for Making and Using U-bend Stress- Corrosion Test Specimens	Metals and alloys	To be selected	Suitable for testing of weldments
18	4.5.6	ASTM G 49: Standard Practice for Preparation and Use of Direct Tension Stress-Corrosion Test Specimens	Metals and alloys	To be selected	Suitable for testing of weldments
18	4.5.7	ASTM G 58: Standard Practice for Preparation of Stress-Corrosion Test Specimens for Weldments	Weldable metals and alloys	To be selected	
18	4.5.8	ASTM G 123: Standard Test Method for Evaluating Stress- Corrosion Cracking in alloys with Different Nickel Contents in Boiling Acidified Sodium Chloride Solution	Stainless steels including austenitic- ferritic (duplex) steels	25 % NaCl + H <sub>3</sub> PO <sub>4</sub> for pH adjustment	Suitable for testing of weldments. To be used when 4.5.1 does not give a relevant environment
19	4.5.9	NACE TM0177: Laboratory Testing of Metals for Resistance to Specific Forms of Environmental Cracking in H <sub>2</sub> S Environments	Metals and alloys	Acidified aqueous environment containing H <sub>2</sub> S	Can be used for testing of weldments
19	4.5.10	ASTM G 129: Standard Practice for Slow Strain Rate Testing to Evaluate the Susceptibility of Metallic Materials to Environmentally Assisted Cracking	Corrosion resistant alloys and steels	Acidified aqueous environment containing H <sub>2</sub> S	Suitable for testing of weldments
19	4.5.10	NACE Standard TM 0198- 98: Slow Strain Rate Test Method for Screening Corrosion Resistant Alloys (CRAs) for Stress Corrosion Cracking in Sour Oilfield Service	Corrosion resistant alloys and steels	Acidified aqueous environment containing H <sub>2</sub> S	Suitable for testing of weldments

Page	Nr	Standard	Materials	Solution	Comments	
20	4.5.11	NACE TM 0284-96: Evaluation of Pipeline and Pressure Vessel Steels for Resistance to Hydrogen- Induced Cracking	Pipeline steels	Solution A: 5 %NaCl + 0,5 % Acetic acid Solution B: Synthetic seawater saturated with $H_2S$	Suitable for testing of weldments	
20	4.5.12	NACE MR 0175 ISO 15156: Petroleum and natural gas industries – Materials for use in $H_2S$ containing environments in oil and gas production	Carbon steels, low- alloy steels and corrosion-resistant alloys	To be selected	Suitable for testing of weldments	
21	4.5.13	MTI-3 Test Method: Laboratory Testing of Iron- and Nickel-Based Alloys for Relative Resistance to Stress Corrosion Cracking in a Boiling Magnesium (MgCl <sub>2</sub> ) Solution	Wrought Iron- and Nickel-Based Alloys	A solution of MgCl <sub>2</sub> that boils at 155 ± 1,0 °C	Can be used for weldments	
21	4.5.14	MTI-5 Test Method: Laboratory Testing of Iron- and Nickel-Based Alloys for Relative Resistance to Stress Corrosion Cracking in a Sodium Chloride (NaCl) Drop Evaporation System	Wrought Iron- and Nickel-Based Alloys	A 0,6 % solution of NaCl	Can be used for weldments	
4.6 Co	orrosion	fatigue				
21	4.6.1	ISO 11782-1: Cycles to failure testing	Metals and alloys	To be selected	Suitable for testing of weldments	
21	4.6.2	ISO 11782-2: Crack propagation testing using pre-cracked specimens	Metals and alloys	To be selected	Suitable for testing of weldments	
4.7 Ga	alvanic	corrosion				
21	4.7.1	ASTM G 71: Standard guide for Conducting and Evaluating Galvanic Corrosion Tests in Electrolytes	Metals and alloys	To be selected	Suitable for testing of weldments	
4.8 Fie	4.8 Field testing					
22	4.8.1	ASTM G 4: Standard Guide for Conducting Corrosion Coupon Tests in Field Applications	Metals and alloys	Process environment	Very suitable for testing of weldments	
22	Appendix I: Environmentally assisted cracking					
23	Appendix II: Full scale testing					
25	Appendix III: Applications of Stress Corrosion Standards					
	NOTE e = electrochemical method					

# **4 CORROSION TEST METHODS**

**ISO 11845** standard: "**Corrosion of metals and alloys** – **General principles for corrosion testing**" serves as a useful introductory document.

## 4.1 General corrosion

The susceptibility to general corrosion of a material is studied by immersion of a test coupon in a specified corrosive environment for a certain period of time. The corrosion rate is determined by measuring the mass loss. Pre cleaning is important to prevent oxides and slag residues contributing to the weight loss after immersing. Post cleaning, to remove corrosion products, must be done with care in order not to remove uncorroded metal.

This can be a useful test for weldments, particularly where there is a risk of galvanic corrosion with certain weld metal/base metal combinations, which the test will reveal.

Corrosion of a welded coupon is best reported by description and thickness measurements, rather than a millimetre per year rate, because the attack is often localised and not representative for the whole surface.

#### 4.1.1 ASTM G 1: "Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens"

#### ASTM G 31: "Standard Practice for Laboratory Immersion Corrosion Testing of Metals"

Materials: Bare solid metal specimens.

*Test solution:* To be selected based on material and application.

*Procedure:* The test specimen is fully immersed in the test solution and corrosion rate is determined by weight loss. The temperature of the test solution is to be selected but should be controlled within  $\pm 1$  °C.

*Weldments:* Feasible test for weldments. However, result should not be reported as weight loss for the reasons mentioned above.

# 4.1.2 JIS G 0591: "Method of sulphuric acid test for stainless steels"

The scope of this Japanese Industrial Standard is to specify a method to evaluate the susceptibility of stainless steels to general corrosion by measuring the weight loss of a sample, when immersed in boiling sulphuric acid.

Materials: Stainless Steels.

Test Solution: Sulphuric acid solution, 5 - 50 wt-percent.

*Procedure:* Test specimen to be prepared having  $10-30 \text{ cm}^2$  of total surface area. Sample to be machined, ground and polished to 240 paper followed by wet polishing up to 600 grit paper. Degreasing followed by drying.

Specimen to be weighed to nearest 1 mg before immersing in the boiling test solution for 6 hours. After test, remove eventual corrosion products by cleaning with 30 % nitric acid followed by brushing with a soft brush in running water. After drying, weigh to obtain the loss of mass. Mass loss to be reported in unit g/m<sup>2</sup>/h.

*Weldments:* This method can be used for, wrought stainless steel, castings and deposited metals.

#### 4.1.3 ASTM G 157: "Standard Guide for Evaluating the Corrosion Properties of Wrought Ironand Nickel-Based Corrosion Resistant Alloys for the Chemical Process Industries"

This guide covers an evaluation approach that is designed to provide information on the corrosion properties of wrought iron- and nickel alloys for the chemical process industries. It incorporates test conditions for general corrosion measurements in a variety of environments. The use of this approach will allow direct comparisons to be made among alloys from various suppliers.

This guide is intended to provide a series of evaluations that will assist engineers dealing with chemical environments in selecting appropriate alloys. It is also intended for alloy developers to assist them in choosing environments and test methods that are of particular interest to the chemical process industries.

Materials: Wrought iron and nickel alloys.

*Test solution:* The general corrosion resistance is determined in 14 test solutions (listed in the standard).

*Procedure:* For general corrosion, the test samples are immersed in 14 test solutions at various temperatures to determine the lowest temperature at which the corrosion rate exceeds 0,13 mm/year (5 mpy).

*Weldments:* The standard is very suitable for testing weldments in the as-welded condition. Where the corrosion resistance of the weld deposit is equal to or better than that of the parent metal, a welded specimen can be used in lieu of the as-manufactured specimen and thus avoid running additional tests.

#### 4.1.4 MTI-1 Test Method: "Laboratory Testing of Iron- and Nickel-Based Alloys for Corrosion Resistance in Selected Media"

This method describes the procedure for corrosion tests to determine the relative resistance of wrought iron- and nickel-based alloys to corrosion in selected media. These tests are intended to provide corrosion data suitable for preliminary evaluation prior to testing for specific chemical applications.

Each alloy is tested in the as-manufactured and aswelded condition.

The corrosion test media have been selected to provide a wide spectrum of corrosive environments which are economically important to the chemical industry.

*Materials:* Wrought iron- and nickel-based alloys in the as-manufactured and as-welded condition.

*Test solution:* Fourteen different corrosive environments are used (listed in the standard).

*Procedure:* Two kinds of specimens shall be used: (a) as-manufactured specimens and (b) as-welded specimens.

The reason for using the welded specimen is to evaluate the corrosion resistance of the weld deposit. The weldment shall be made with filler metal using the GTAW (TIG) process.

The test temperatures are to be selected from the following listed: 30, 50, 70, 90, 110 and 130 °C.

The aim is to determine the lowest temperature at which the corrosion rate exceeds 5 mpy (0,13 mm/year).

Specimen evaluation procedures provide for weight loss measurements for evaluation of general corrosion and low power surface microscopic examination for presence of localized corrosion, such as pitting, stress corrosion, intergranular attack, end grain corrosion and preferential weld attack.

*Weldments:* The MTI-1 test method is intended for both base materials and weldments.

#### 4.2 Pitting corrosion

The resistance of a stainless steel or a nickel alloy to pitting corrosion in a chloride-containing environment can be determined by two methods:

 Exposing a sample to a chloride-containing environment by immersing at a pre set temperature.

- Electrochemical determination by detecting changes of the anodic current density of a specimen.

Both methods are suitable for welded specimens and can be used on as-welded surfaces.

### 4.2.1 ASTM G 48: "Standard Test Methods for Pitting and Crevice Corrosion Resistance of Stainless Steels and Related Alloys by use of Ferric Chloride Solution"

ASTM G 46: "Standard Guide for Examination and Evaluation of Pitting Corrosion"

The G 48 standard is divided into six methods. Method A, B, C, D, E and F.

Method A is designed to determine the relative pitting resistance of stainless steels and nickel-base, chromiumbearing alloys. Recommended test temperatures are 22 °C and 50 °C, but other temperatures may be used depending on alloy and chemical composition.

Method C is used for determining critical pitting temperature for nickel-base and chromium-bearing alloys.

Method E is used for determining critical pitting temperature for stainless steels.

*Materials:* Bare solid metal specimens of stainless steels or nickel-base and chromium-bearing alloys.

#### Test solution:

Method A: Ferric chloride pitting test. 6 %  $\text{FeCl}_3$  (by mass) solution.

Method C and E: Critical pitting temperature test. 6 %  $FeCl_3$  (by mass) and 1 % HCl resulting in a pH controlled environment over the test temperatures.

*Procedure:* The test specimen is fully immersed in the test solution. The result is determined by weight loss measurements in combination with visual inspection. Specimen faces are examined for pits at low magnification (20X). Pit sites should be probed with a needle to expose sub surface attack.

For methods A and C the exposure time is 72 hours although method A is often used with a 24 hour exposure time when testing stainless steels and their weldments.

*Weldments:* Suitable method for testing of weldments. The test will give information of the weakest area of the weldment (weld metal, fusion line or HAZ). As-welded or post weld treated surfaces can be tested. As ASTM G-48 is a rather coarse method to test materials for pitting and crevice corrosion, it shall not be over-interpreted.

# 4.2.2 JIS G 0578: "Method of ferric chloride test for stainless steels"

The scope of this JIS standard is to specify the ferric chloride test method (method A) to evaluate the pitting resistance of stainless steels by determining the corrosion rate in 6 % ferric chloride solution. The standard also specifies a method (method B) to determine the critical pitting temperature (CPT) of highly corrosion resistant steels in 6 % ferric chloride solution.

#### Materials: Stainless steels.

*Test solution:* Hydrochloric acid solution containing 6 % ferric chloride.

#### Procedure:

 (Method A) The test specimen shall have a total surface that is larger than 10 cm<sup>2</sup>. Wet polishing to be carried out to grit size 600 followed by degreasing and drying.

The specimen to be weighed to the nearest 1 mg before immersing in the test solution. The quantity of the test solution shall be a minimum 20 ml/cm<sup>2</sup>. The test temperature shall be at 35 °C or 50 °C as a standard. However, the test temperature may be changed by agreement. After immersing for 24 h, corrosion products are gently removed and the specimen is dried, the loss by corrosion is determined by weighing. The depth of pitting and the density of pits are also determined. A fresh solution is prepared for each new test.

– (Method B) The sample preparation and weighing are the same as for Method A. The starting temperature is not specified but may be chosen by using the following formula as a guideline.

T (°C) = 2.5 (wt-% Cr) + 7.6 (wt-% Mo) + 31.9 (wt-% N) - 41.0

In contrast to Method A, the testing time is 72 h. After washing and drying, the pitting depth is determined and the lowest temperature producing pitting of minimum depth of 0,025 mm is the CPT. A fresh solution is used for every test.

Evaluation: For method A, the mass loss shall be expressed as  $g/m^2/h$ . For method B, the continuous immersion time, the test temperature, the maximum pitting depth and the determined CPT (°C) shall be described together.

*Weldments:* Suitable method for testing of weldments. The test will give information about the weakest area of the weldment (Weld metal, fusion line or HAZ). Aswelded or post weld treated surfaces can be tested.

#### 4.2.3e ASTM G 61: "Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion Susceptibility of Iron-, Nickel-, or Cobalt-Based Alloys"

This test method gives a procedure for conducting cyclic potentiodynamic polarization measurements to determine relative susceptibility to localized corrosion (pitting and crevice corrosion).

Materials: Iron, nickel, and cobalt alloys.

Test Solution: Chloride environment.

*Process:* An indication of the susceptibility to initiation of localized corrosion in this test method is given by the potential at which the anodic current increases rapidly. The higher this potential, obtained at a fixed scan rate, the less susceptible is the alloy to initiation of localized corrosion.

*Weldments:* The test method can be used for weldments.

#### 4.2.4e ASTM G 150: "Standard Method for Electrochemical Critical Pitting Temperature Testing of Stainless Steels"

This method covers a procedure for the evaluation of the resistance of stainless steel and related alloys to pitting corrosion based on the concept of the determination of a potential independent critical pitting temperature (CPT).

This test method applies to wrought and cast products including but not restricted to plate, sheet, tubing, bar, forgings and weldments.

*Materials:* Austenitic stainless steels, duplex (Austenitic-Ferritic) stainless steels and other related alloys.

#### Test Solution: 1M NaCl.

*Procedure:* The specimen is exposed, either entirely or in part, depending on test cell configuration, to the test solution initially at 0 °C. After an initial temperature stabilization period, the solution is heated at a rate of 1 °C/min. About 60 seconds before the temperature scan is commenced, the specimen is anodically polarized to a potential above the pitting potential range. This potential is kept constant during the whole temperature scan. The CPT is defined as the temperature at which the current increases rapidly. Pitting is confirmed visually after the test.

*Weldments:* The method is excellent for determining the CPT of as-welded surfaces of weldments.

# 4.2.5 ISO 11463: "Corrosion of metals and alloys – Evaluation of pitting corrosion"

This international standard gives guidance on the selection of procedures that can be used in the identification and examination of pits and in the evaluation of pitting corrosion.

#### 4.2.6e ISO 17864: "Corrosion of metals and alloys – Determination of the critical pitting temperature under potentiostatic control"

This international standard describes the procedure for determining the critical pitting temperature for stainless steels (austenitic, ferritic/austenitic, ferritic stainless steels) under potentiostatic control. This standard is very similar to ASTM G 150. The principal advantage is the rapidity with which the critical pitting temperature can be measured in a single test. The critical pitting temperature, as determined in this international standard, can be used as a relative index of performance, for example to compare the relative performance of different grades of stainless steel.

*Materials:* Austenitic stainless steels, duplex (Austenitic-Ferritic) stainless steels and other related alloys.

Test solution: 1,0 Mol/I NaCl.

*Procedure:* The exposed surface area of the specimen is measured. The specimen, counter electrode and salt bridge are placed in the test cell. The cell is then filled with the solution, which is stirred continuously throughout the test. When the potential has been applied for 60 s or longer, the temperature of the specimen shall be increased at a controlled rate. The CPT is defined as the temperature at which the current density reaches 100  $\mu$ A.cm<sup>-2</sup> and then remains above this level for a minimum of 60 s.

*Weldments:* The method is excellent for determining the CPT of as-welded surfaces of weldments.

# 4.2.7 ASTM A 923-C: "Ferric chloride corrosion test for classification of structures of duplex stainless steels"

NOTE This standard shall not be confused with ASTM G 48. This method does not determine the critical pitting temperature. It is designed solely for detection of detrimental intermetallic phases in duplex stainless steels.

#### 4.3 Intergranular corrosion

#### 4.3.1 ASTM A 262 Practice B: "Ferric sulphatesulfuric acid test for detecting susceptibility to intergranular attack in austenitic stainless steels"

The ferric sulphate-sulphuric acid test detects susceptibility to intergranular attack associated with the precipitation of chromium carbides in unstabilized austenitic stainless steels. It does not detect susceptibility to intergranular attack associated with sigma phase. The exception is stabilised stainless steels of type 321 (and perhaps 347) and cast austenitic stainless steels containing Mo, it also detects intergranular attack associated with sigma phase.

Materials: Austenitic stainless steels.

*Test solution:* Approximately  $3 \% \text{Fe}_2(SO_4)_3$  and  $50 \% \text{H}_2SO_4$ . Balance distilled water.

*Procedure:* Specimen to be weighed to nearest 0,001 g and immersed in boiling solution for 120 hours. After 120 hours, the specimen shall be rinsed and dried and weighed. The weight loss is calculated and the corrosion rate to be reported as millimetres of penetration per month.

Weldments: The test is suitable to use on weldments.

#### 4.3.2 ASTM A 262 Practice C (Huey test): "Nitric acid test for detecting susceptibility to intergranular attack in austenitic stainless steels"

The scope of the test is to measure the relative susceptibility of austenitic stainless steels to intergranular attack. Intergranular attack in nitric acid is associated with:

1) Intergranular precipitation of chromium carbides,

2) Sigma or transition phases in molybdenum-bearing grades, and

3) Sigma phase constituents in stabilised grades.

The test may be used to evaluate the heat treatment accorded "as-received" material. It is also used to check the effectiveness of stabilising elements and of reductions in carbon content in preventing susceptibility to rapid intergranular attack.

Note that the boiling nitric acid test should not be used for extra low carbon molybdenum-bearing grades, unless the material tested is to be used in nitric acid service.

Specimens of extra low carbon and stabilised grades are tested after a sensitising heat treatment at 675 °C for 1 hour. This practice may be applied to wrought products (including tubes), castings, and weld metal of the various grades of stainless steels.

Materials: Austenitic Stainless Steels.

Test solution: Nitric acid 65 %.

*Procedure:* The maximum convenient weight of specimens is 100 g. Specimens containing welds should be cut so that no more than 13 mm width of base metal is included on either side of the weld.

The specimen should be measured, including the inner surfaces of any holes, and the total exposed area calculated. The specimen shall be weighed to the nearest 0,001 g.

The specimen is fully immersed in the boiling acid for five periods of 48 hours each.

The loss of weight is determined after each testing period and for the total of the test periods. The corrosion rate is usually reported as millimetres per month.

*Weldments:* This practice can be applied to wrought products, castings and weld metal of various grades intended for use in strongly oxidizing environments.

The test indicates the susceptibility of intergranular attack associated with the precipitation of chromium-rich carbides. It does not detect susceptibility associated with sigma phase.

Materials: Austenitic stainless steels.

*Test solution:* 6 weight-% of anhydrous  $CuSO_4$  and 16 weight-% of  $H_2SO_4$ . Balance water. Electrolytic grade copper shot or grindings are added to the solution in a sufficient quantity (the effective galvanic coupling between copper and the test specimen may have importance).

*Procedure:* No particular size of the test specimen is specified. The testing apparatus dictates the final size and shape of the test specimen.

Each specimen shall be degreased prior to being tested.

Specimens of extra low carbon and stabilised grades are tested after sensitising heat treatment at 675 °C for 1 hour.

Specimen to be completely immersed in the test solution at ambient temperature, which is then brought to boil and maintained boiling throughout the test period. The time of the test shall be minimum of 24 hours.

The test specimen shall be bent through 180° over a diameter equal to the thickness of the specimen being bent. The bent specimen shall be examined under low magnification (5 to 20X). The appearance of fissures or cracks indicates the presence of intergranular attack.

*Weldments:* The method is suitable for testing of weldments. The weld – base metal interface shall be located approximately at the centreline of the bend.

Face, root or side bend tests may be performed. The bend radius shall not be less than that required for mechanical testing in the appropriate material specification (for base metal) or in ASME Code Section IX (for welds).

#### 4.3.4 ASTM A 262 Practice F: "Copper – copper sulfate – 50 % sulfuric acid test for determining susceptibility to intergranular attack in Austenitic Stainless Steels"

This test may be used to evaluate the susceptibility of an austenitic stainless steel to intergranular corrosion caused by chromium carbide precipitation. The test does not detect susceptibility to intergranular attack associated with sigma phase.

Materials: Austenitic Stainless Steels.

*Test Solution:* Approximately 10 % CuSO<sub>4</sub> and 50 %  $H_2SO_4$ . A piece of copper with dimensions of  $3.2 \times 19 \times 38$  mm, or an equivalent area of copper shot or chips, may be used.

*Procedure:* Specimens of extra low carbon and stabilised grades are tested after a sensitising heat treatment at 675 °C for 1 hour.

The specimen to be tested is immersed in the boiling test solution for 120 hours. The weight loss of the specimen is determined by weighing (before and after immersing) on an analytical balance to the nearest 0,001 g. The corrosion rate should be reported as millimetres of penetration per month. No bending is involved.

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*Weldments:* It is possible to use this test to evaluate the resistance of extra low carbon grades to sensitisation, caused by welding, and to estimate the susceptibility to intergranular attack.

#### 4.3.5 ISO 3651-1: "Determination of resistance to intergranular corrosion of stainless steels – Part 1: Austenitic and ferritic-austenitic (duplex) stainless steels – Corrosion test in nitric acid medium by measurement of loss in mass (Huey test)"

This standard is very similar to ASTM A 262 Practice C (4.3.2 above). There are, however, minor differences in the following areas.

*Materials:* Austenitic and ferritic-austenitic (duplex) stainless steels (4.3.2 ASTM A262 Practice C, is intended for austenitic stainless steels only).

#### Test solution: Identical with 4.3.2.

*Procedure:* Sensitisation to be made for stabilised steels and steels with C  $\leq$  0,03 %. 700 °C  $\pm$  10 °C for 30 min. Welded pieces shall not be submitted to a sensitizing heat treatment.

Testing procedure in corrosive solution is identical to 4.3.2.

The corrosion rate to be reported as mm/year or in g/m<sup>2</sup>/h.

Weldments: The same applicability as for 4.3.2.

#### 4.3.6 ISO 3651-2: "Determination of resistance to intergranular corrosion of stainless steels – Part 2: Ferritic, austenitic and ferritic-austenitic (duplex) stainless steels – Corrosion test in media containing sulphuric acid"

This part of ISO 3651 specifies methods for the determination of resistance to intergranular corrosion of ferritic, austenitic and ferritic-austenitic (duplex) stainless steels in media containing sulphuric acid. The test methods included are:

- Method A: the 16 % sulphuric acid/copper sulphate test (Strauss test).

- Method B: the 35 % sulphuric acid/copper sulphate test.
- Method C: the 40 % sulphuric acid/ferric sulphate test.

The methods are applicable to stainless steels supplied in the form of castings (welds), rolled or forged products and tubes and intended for use in mildly oxidizing acid medium.

*Materials:* Ferritic, austenitic and ferritic-austenitic (duplex) stainless steels.

#### Test solution:

- Method A: 16 % H<sub>2</sub>SO<sub>4</sub> + ~ 10 % CuSO<sub>4</sub>.
- Method B: 35 % H<sub>2</sub>SO<sub>4</sub> + ~ 11 % CuSO<sub>4</sub>.
- Method C: 40 %  $H_2SO_4$  + ~ 2.5 %  $Fe_2(SO_4)_3$ .

*Procedure:* Sensitisation heat treatment to be performed on stabilized steels and steels with low carbon content.

Test pieces, which are not solution annealed after welding, shall be tested in the as-welded condition. No additional sensitisation heat treatment shall be performed. Sensitisation by welding applies to all the stainless steels covered by this part of ISO 3651.

For corrosion testing, a test piece is prepared according to the standard, and is immersed in a solution according to A, B or C for a specified time. The test piece is then subject to a bend test. The convex surface of the test piece is examined after bending in order to reveal any cracks caused by intergranular corrosion. The duration of the test shall be 20 hours.

For the bend test following the 20 hour boiling period, cylindrical and flat pieces from wrought products shall be bent to at least 90° over a mandrel with a radius not exceeding the thickness of the test piece.

For evaluation, the bent test piece shall be examined under low magnification (about 10 X), in order to detect cracking.

*Weldments:* The standard is, among others, intended for testing intergranular corrosion caused by sensitisation during welding.

#### 4.3.7 JIS G 0572: "Method of ferric sulphatesulphuric acid test for stainless steels"

This Japanese Industrial Standard specifies a method for testing for intergranular corrosion by measurement of weight loss of an austenitic stainless steel specimen in a boiling solution of sulphuric acid and ferric sulphate.

Materials: Austenitic stainless steels.

*Test solution:*  $50 \pm 0.3$  % sulphuric acid + ~ 4 % ferric sulphate.

*Procedure:* The test specimen, which can be taken from castings, wrought material or deposited metal, shall have a total area of 10 - 30 cm<sup>2</sup>. The surfaces shall be dry polished to grit size 120 minimum or wet polished to size 80 minimum.

Sensitisation of the specimen shall be carried out for stabilised steels and for steels containing a maximum of 0,030 % carbon. The sensitisation shall be carried out before polishing at a temperature of 650 °C for 2 h followed by air cooling.

The specimen is weighed to the nearest 1 mg and immersed in the boiling test solution for 120 h. Other durations of testing can be agreed upon.

After the test, corrosion products are gently removed and the specimen is weighed after drying. No bending of the specimen shall be carried out.

The loss of mass is reported in unit g/m<sup>2</sup>/h.

*Weldments:* Applicable for weldments in austenitic stainless steels.

# *4.3.8 JIS G 0573: "Method of 65 per cent nitric acid test for stainless steels"*

This Japanese Industrial Standard was revised in 1999 to conform to ISO 3651-1. The parts compatible with

corresponding ISO standard were adopted without any modification in their technical content.

# 4.3.9 JIS G 0574: "Method of nitric-hydrofluoric acid test for stainless steels"

This Japanese Industrial Standard specifies a method for evaluating the susceptibility of molybdenum – bearing austenitic stainless steels to intergranular corrosion by immersion of a test specimen in 10 % nitric and 3 % hydrofluoric acid solution at 70 °C.

*Materials:* Molybdenum-bearing austenitic stainless steels.

Test solution: 10 % nitric acid + 3 % hydrofluoric acid.

*Procedure:* The test specimen, which can be taken from castings, wrought material or deposited metal, shall have a total area of 10 - 30 cm<sup>2</sup>. The surfaces shall be dry, polished to grit size 120 minimum or wet polished to size 80 minimum. After polishing, the specimen shall be degreased with a suitable solvent and dried.

Steels of normal carbon content shall, prior to polishing, be solution treated at 1 100  $^{\circ}$ C for 30 min followed by quenching.

Low carbon steels shall be sensitised at 650 °C for 2 h followed by air cooling.

Tests on as-received material shall be carried out in parallel with the sensitised specimens.

The specimen to be weighed to the nearest 1 mg and immersed in the test solution for 2 h. The temperature of the test solution shall be  $70 \pm 0.5$  °C.

The result shall be reported as corrosion rate ratio between corrosion rate of as-received test specimens and corrosion rate of heat treated (sensitised) specimens.

*Weldments:* May be used for weldments and all-weld-metal.

#### 4.3.10 JIS G 0575: "Method of copper sulphatesulphuric acid test for stainless steels"

The JIS standard G 0575 was revised in 1999 to conform with ISO 3651-2. As a result, the parts compatible with the corresponding ISO standard were adopted without any modification in the technical content.

However, the portions in the ISO standard, which were found difficult to incorporate into JIS 0575, at the stage of its review for conformity, are adopted in Annexes. Annex A (normative) "Streicher test". Annex B (informative) "The 35 % Sulphuric acid / copper sulphate test". Annex C (informative) "Examples of application".

Weldments: Comments, see 4.3.6.

# 4.3.11 GOST 6032-89: "Methods for determination of intercrystalline corrosion resistance"

The GOST standard specifies methods for determining the resistance to intergranular corrosion for stainless steels. Forms of stainless steels to be tested are castings, wrought material, weldments, and all-weld metal. The standard is based on ISO 3651-1 and ISO 3651-2. It is a combined standard for intergranular corrosion and also contains the oxalic acid test similar to ASTM A 262A.

*Materials:* Austenitic, austenitic-martensitic, austenitic-ferritic (duplex) and ferritic stainless steels.

*Weldments:* The standard is similar to the ISO 3651 standards adapted for the testing of weldments.

#### 4.3.12 ASTM A763-93: "Standard Practices for Detecting Susceptibility to Intergranular Attack in Ferritic Stainless Steels"

These practices cover the following four tests:

- Practice W Oxalic acid etch test.
- Practice X Ferric sulphate-sulphuric acid test.

Practice Y – Copper-copper sulphate-50 % sulphuric acid test.

Practice Z – Copper-copper sulphate-16 % sulphuric acid test.

*Materials:* Ferritic stainless steels. The practice contains a table listing the alloys for which each respective method is appropriate.

Test solutions:

- Practice W: 10 % oxalic acid solution (reagent grade).
- Practice X:  $H_2SO_4 + Fe_2(SO_4)_3 + H_2O + boiling chips.$ - Practice Y:  $H_2SO_4(50 \%) + CuSO_4 + H_2O + piece of copper + boiling chips.$

- Practice Z:  $H_2SO_4(16 \%)$  + CuSO<sub>4</sub> +  $H_2O$  + copper shots or grindings.

*Procedure:* For practices W - Z, a specimen having a total surface area of  $5 - 20 \text{ cm}^2$  is recommended. Aswelded specimens should be cut so that no more than 13 mm (1/2 in.) width of unaffected base metal is included on either side of the weld and heat – affected zone. The intent is to test a specimen representing, as nearly as possible, the surface of the material as used in service. Specimens from materials that are going to be welded or heat treated should be welded or heat treated in as nearly the same manner as the material will experience in service.

*Weldments:* All practices may be applied on wrought products, as well as weldments.

#### 4.3.13 ISO 9400: "Nickel-based alloys – Determination of resistance to intergranular corrosion"

This international standard specifies four methods for determination of the susceptibility of nickel alloys to intergranular corrosion.

- Method A: Iron (III) sulphate-sulphuric acid test.

Method B: Copper – copper-sulphate – 16 % sulphuric acid test.

- Method C: Hydrochloric acid test.
- Method D: Nitric acid test.

*Materials:* As a guide, the methods specified in this international standard should be applicable to those nickel alloys used for corrosion service and listed in ISO 6207. Test solutions:

- Method A:  $H_2SO_4 + Fe_2(SO_4)_3 + H_2O +$  boiling chips. - Method B:  $H_2SO_4 + CuSO_4 + H_2O +$  copper shots or turnings.

- Method C: HCl +  $H_2O$  + boiling chips (For Ni-Cr-Mo alloys the same test procedure can be used except that a 10 % solution of HCl should be used).

- Method D:  $HNO_3 + H_2O$  (65 % solution).

#### Procedure:

A specimen having a total surface area of  $20 - 30 \text{ cm}^2$  is recommended. As-welded specimens should be cut so that no more than a 13 mm width of unaffected base metal is included on either side of the weld and the heat-affected zone. It is intended to test a specimen representing, as nearly as possible, the surface of the mate-rial used in service. Specimens from material that is intended to be welded or heat treated shall be welded or heat treated in nearly the same manner as the mate-rial will experience in fabrication or service.

*Weldments:* All practices may be applied on wrought products, as well as weldments.

## 4.3.14 ASTM G 28: "Standard Test Methods of Detecting Susceptibility to Intergranular Corrosion in Wrought, Nickel-Rich, Chromium-Bearing Alloys"

The method covers two tests:

- Method A: Ferric Sulphate-Sulphuric Acid Test.
- Method B: Mixed Acid-Oxidizing Salt Test.

Only method A is suitable for weldments and cast products.

*Materials:* Certain nickel-rich, chromium-bearing alloys with UNS numbers:

N06007	N06455	N08020
N06022	N06600	N08367
N06030	N06625	N08800
N06059	N06686	N08825
N06200	N06985	N10276

Test solution: Method A.

 $Fe_2(SO_4)_3 + 50 \% H_2SO_4 + Bal.$  Water.

*Procedure:* A specimen having a total surface area of 5 to  $20 \text{ cm}^2$  is recommended. The intent is to test a specimen representing as closely, as possible, the material as used in service.

The specimen is immersed in the boiling solution for a time between 24 and 120 hours depending on the alloy. The loss of mass during the period is used to calculate the rate of corrosion.

*Weldments:* This method may be used to evaluate asreceived material and to evaluate the effects of subsequent heat treatment. The method can be used for wrought material, weldments and cast products of the alloys in the table above.

#### 4.3.15e GOST 9.914:"Electrochemical methods for determination of inter crystalline corrosion resistance"

The standard specifies accelerated electrochemical methods for determination of resistance to intergranular corrosion. Four different methods are described: Potentiostatic pickling (PP-method), drop method (PK-1 method), corrosion potential measurement (PK-2 method) and potentiodynamic reactivation (PDR-method).

Materials: Austenitic stainless steels.

*Weldments:* PP, PK-1, PK-2 and PDR methods are suitable for the assessment of welded joints. Sampling and sample preparation to be carried out according to GOST 6032.

#### 4.4 Crevice corrosion

#### 4.4.1 ASTM G-48: "Standard Test Methods for Pitting and Crevice Corrosion Resistance of Stainless Steels and Related Alloys by use of Ferric Chloride Solution"

This test method covers procedures for the determination of the resistance of stainless steels and related alloys to pitting and crevice corrosion, when exposed to chloride environments.

 Method B: Ferric chloride crevice test for nickel-base and chromium-bearing alloys.

 Method D: Critical crevice temperature test for nickelbase and chromium-bearing alloys.

 Method F: Critical crevice temperature test for stainless steels.

As Method B has many technical drawbacks e.g. the result is dependent on specimen thickness, edge attacks influence the initiation of attacks under the crevice formers etc. It is advised to use Methods D and F instead. Methods D and F allow for the ranking of alloys by the minimum (critical) temperature required to cause initiation of crevice corrosion.

*Materials:* Stainless steels and nickel-base, chromium-bearing alloys.

#### Test solution:

Method B: Ferric chloride crevice test. 6 % FeCl<sub>3</sub> (by mass) solution.

– Methods D and F: Critical crevice temperature test. 6 %  $FeCl_3$  (by mass) and 1 % HCl resulting in a pH controlled environment throughout the test temperature range.

#### Procedure:

- Crevice formers - Method B:

1. Cylindrical TFE – fluorocarbon blocks, two for each test specimen. Each block shall be 12.7 mm in diameter and 12.7 mm high with perpendicular grooves.

2. Fluorinated Elastomeric O – rings or rubber bands.

Recommended test temperatures are 22 °C and 50 °C but other temperatures may be used depending on alloy and chemical composition.

- Crevice formers - Methods D and F:

1. A Multiple Crevice Assembly (MCA), consisting of two TFE - fluorocarbon segmented washers, each having a number of grooves and plateaux, shall be used.

The test specimen is fully immersed in the test solution. The result is determined by weight loss measurements in combination with visual inspection.

Exposure time for methods B and D is 72 hours.

Exposure time for method F is 24 hours.

Weldments: The method can be used for welds but flat specimens are needed in order to get a well defined crevice.

#### 4.4.2 ASTM G 78: "Standard Guide for Crevice Corrosion Testing of Iron-Base and Nickel-Base Stainless Alloys in Seawater and other Chloride-**Containing Aqueous Environments**"

This guide covers procedures for crevice corrosion testing of iron and nickel stainless alloys in seawater. The guidance provided may also be applicable to crevice corrosion testing in other chloride-containing natural waters and various laboratory-prepared aqueous chloride environments.

Materials: Iron and nickel alloys.

Test Solution: Chloride-containing aqueous solutions including seawater.

Process: Crevice assemblies exposed to chloride-containing environments. Test duration at least 30 days. Evaluation can be based on a number of criteria including mass loss.

Weldments: May be used for testing of weldments when reinforcements are removed.

#### 4.4.3 MTI-2: "Test Method: Laboratory Testing of Iron- and Nickel-Based Alloys for Relative Resistance to Crevice Corrosion in a 6 % Ferric Chloride (FeCl<sub>3</sub>) Solution"

MTI-2 Test Method provides a means for ranking the crevice corrosion resistance of a new alloy in a 6 % ferric chloride solution. The procedure is patterned after ASTM G 48 with the added feature of varying the temperature of the ferric chloride solution to determine the critical crevice corrosion temperature (CCT).

The method is designed to determine the relative resistance of alloys to crevice corrosion in an oxidizing chloride environment by measuring the minimum (critical) temperature for crevice corrosion.

The MTI-2 Test Method is also used to determine the probability for initiation and rate of crevice corrosion for duplex stainless steels compared to austenitic stainless steels.

Materials: Wrought Iron- and Nickel-Based Alloys.

*Test solution:* 6 % FeCl<sub>3</sub> solution.

Process: The test specimen shall be made from sheet, plate or strip produced by commercial methods.

The crevice blocks are fastened to the test specimens and tightened by a torque of 0.28 Nm. After exposure in the FeCl<sub>3</sub> solution for 24 hours the assembly is removed from the solution and disassembled. The areas under the crevice blocks are examined for crevice attack at low power (20X) magnification. Crevices are considered corroded if the local attack is 1.0 mil (0.025 mm) or greater.

Weldments: The method is difficult to use on as welded surfaces but can be applied on flat surfaces after the reinforcement is removed.

### 4.5 Stress corrosion (Environmentally assisted cracking)

This section covers the test procedures developed for all types of environmentally assisted cracking (EAC), including stress corrosion cracking and also cracking in the absence of external loads. No distinction has been made whether stress corrosion cracking is hydrogenrelated or not. For the description of the various EAC types, the ISO 15156-Part 1 standard is recommended.

#### 4.5.1 ASTM G 36: "Standard Practice for Evaluating Stress-Corrosion-Cracking Resistance of Metals and Alloys in a Boiling Magnesium Chloride Solution"

This practice describes a procedure for conducting stress corrosion cracking test in a boiling magnesium chloride solution containing approximately 45 % MgCl<sub>2</sub>. The boiling temperature shall be kept constant at 155.0 ± 1.0 °C.

(This practice should also be compared to ASTM G 123, in 4.5.8 below.)

Materials: Wrought, cast and welded stainless steels and related alloys. It is a method for detecting the effects of composition, heat treatment, surface finish, microstructure and stress on the susceptibility of these materials to chloride stress corrosion cracking.

Test solution: Approximately 45 % MgCl<sub>2</sub> solution.

Procedure: Any type of stress corrosion test specimen can be used (See ASTM G 30: "Making and Using Ubend Stress-Corrosion Test Specimens"). The specimen must be thick enough so that the applied stress does not cause mechanical rupture when the cross section is reduced by general or pitting corrosion.

After the boiling point is set to 155 ± 1.0 °C by adjusting with water, the specimen is immersed in the solution. The starting time is recorded and the time required to initiate cracks or the crack rate and/or the time to failure may be of importance, depending of the purpose of the test.

Weldments: The boiling magnesium chloride test is applicable to wrought, cast and welded stainless steels and related alloys.

#### 4.5.2 ISO 7539-1 through ISO 7539-9: "Corrosion of metals and alloys - Stress corrosion testing"

The ISO 7539 standard has been developed for the assessment of the resistance of metals and alloys to

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stress corrosion. The different test procedures are described in parts 1 through 9.

ISO 7539-1: "General guidance on testing procedures"

This part of ISO 7539 describes the general considerations which apply when designing and constructing tests to assess susceptibility of materials to stress corrosion.

**ISO 7539-2**: "Preparation and use of bent beam specimens"

This part covers procedures for designing, preparing and using bent beam test specimens.

Bent beam specimens may be used to test a variety of product forms such as sheet, plate, castings, wire, rod or flat, extruded materials. They can also be used for parts joined by welding.

Materials: Metals and alloys.

Test Solution: To be selected.

*Procedure:* The test consists in applying a bending stress to a beam specimen of rectangular or circular cross section and exposing the stressed specimen to a specific test environment. Bent beam specimens are used only for testing at stress levels below the elastic limit.

The time required for cracks to appear, after exposure of stressed specimens to the test environment or the threshold stress below which cracks do not appear, can be used as a measure of the stress corrosion resistance of the material in the test environment at the stress level employed.

*Weldments:* The method can be used for testing overlay welds, welded joints and all-weld metal. (See ISO 7539-8).

ISO 7539-3: "Preparation and use of U-bend specimens"

This part of ISO 7539 covers procedures for designing, preparing and using U-bend test specimens for investigating the susceptibility of a metal to stress corrosion.

U-bend specimens may be used to test a variety of product forms, such as sheet, plate or flat, extruded materials. They may also be employed for materials in the form of wire or rod and also for parts joined by welding.

The U-bend test is frequently used to establish whether an alloy is susceptible to stress corrosion cracking in a given environment. It is often used in laboratories to screen materials for susceptibility in specific applications and service environments and to assess the risk of failure.

The principle advantages of the test are its simplicity and adaptability for use in plant and process environments. A disadvantage is that stresses cannot be quantified with accuracy. The main objective of the test is to establish whether a metal is suitable for a proposed application.

Materials: Metals and alloys.

Test Solution: To be selected.

*Procedure:* The test consists in exposing to the corrosive environment a piece of metal bent into a U-shape and held in a manner which ensures that there are initial tensile stresses ranging up to the yield point over a portion of the surface. Varying amounts of cold work may be introduced and this deformation may influence the tendency to stress corrosion when compared to that of the material in the original condition.

The test should be regarded as basically a "go/no go" test. Minor differences in behaviour, e.g. time to first crack, or in size of crack, should not be considered significant.

*Weldments:* The method can be used for testing, overlay welds, welded joints and all-weld metal samples. (See ISO 7539-8).

**ISO 7539-4**: "Preparation and use of uniaxially loaded tension specimens"

This part of ISO 7539 covers procedures for designing, preparing and using uniaxially loaded tension test specimens for investigating susceptibility to stress corrosion.

Tension test specimens are adaptable for testing a wide variety of product forms, e.g. plate, rod, wire, sheet and tubes, as well as parts joined by welding.

Materials: Metals and alloys.

Test Solution: To be selected.

*Procedure:* The test consists in subjecting a specimen to constant load, constant strain, or increasing load or strain, during exposure in a corrosive environment. The environment selected should ideally be the same as the one prevailing for the intended use of the alloy or comparable to the anticipated service condition. A number of standard environments are also used for ranking purposes.

The most frequently used parameter for assessing cracking susceptibility is the time to total failure, which is easy to measure with reasonable accuracy.

*Weldments:* The method can be used for testing welded joints as well as all-weld metal samples (See ISO 7539-8).

ISO 7539-5: "Preparation and use of C-ring specimens"

This part of ISO 7539 covers procedures for designing, preparing, loading, exposing and inspecting C-ring test specimens for investigating the susceptibility to stress corrosion.

The C-ring is a versatile, economic specimen for determining the susceptibility to stress corrosion cracking of all types of metals in a wide variety of product forms including parts joined by welding.

C-ring specimens may be stressed to predetermined levels, using simple equipment for application of either constant load or constant strain.

Materials: Metals and alloys.

Test Solution: To be selected.

*Procedure:* The test involves subjecting a specimen to constant load, or to constant strain, with a view to determining stress corrosion susceptibility by immersing in a corrosive environment. Because of the small size of the specimen and the simple method of stressing, it can be exposed to almost any kind of corrosive environment.

The time required for cracks to appear after exposure of stressed specimens to the test environment or the threshold stress below which cracks do not appear can be used as a measure of the stress corrosion resistance of the material in the test environment at the stress level employed.

*Weldments:* The method can be used for testing welded joints as well as all-weld-metal samples. (See ISO 7539-8).

**ISO 7539-6**: "Preparation and use of pre-cracked specimens for tests under constant load or constant displacement"

This part of ISO 7539 covers procedures for designing, preparing and using pre-cracked specimens for investigating susceptibility to stress corrosion.

Pre-cracked specimens are not suitable for the evaluation of thin products such as sheet or wire. They are generally used for thicker products including plate bar and forgings. They can also be used for parts joined by welding.

Pre-cracked specimens allow data to be acquired from which critical defect sizes, above which stress corrosion cracking might occur, can be estimated for components of known geometry subjected to known stresses. They also enable rates of stress corrosion crack propagation to be determined. The latter data may be taken into account when monitoring parts containing defects during service.

Materials: Metals and alloys.

Test Solution: To be selected.

*Procedure:* Special equipment is needed for testing. The test involves subjecting a specimen, in which a crack has been developed by fatigue from a machined notch, to either a constant load or displacement during exposure to a test environment. The objective is to quantify the conditions under which environmentally assisted crack extension can occur in terms of stress intensity for stress corrosion cracking,  $K_{ISCC}$ , and examine the kinetics of crack propagation.

*Weldments:* The method can be used for testing welded joints as well as all-weld metal. (See ISO 7539-8).

ISO 7539-7: "Slow strain rate testing"

This part of ISO 7539 covers procedures for conducting slow strain rate tests for investigating susceptibility of a metal to stress corrosion cracking, including hydrogeninduced failure. The test will be described together with the NACE TM 0198-98 standard.

**ISO 7539-8**: "Preparation and use of specimens to evaluate weldments"

This part of ISO 7539 covers the procedures available for stress corrosion testing of welded specimens. In particular, it gives recommendations for the choice of specimens and test procedures to determine the resistance of a metal or an alloy to stress corrosion when it is welded.

Weld regions are more likely than the parent metal to contain defects which may influence corrosion and

stress corrosion behaviour, e.g. microcracking, lack of fusion and porosity. It is therefore recommended that the weldment be characterized with regard to residual welding stresses, surface condition and weld defects prior to testing. Other factors that can influence stress corrosion are:

- changes in microstructure;
- non metallic inclusions;
- stress concentrations.

Specimens can be prepared from weldments in the aswelded or post weld heat-treated conditions. It is recommended that specimens be tested in the same condition of heat treatment as that of the intended application.

Materials: Metals and alloys.

Test solution: To be selected.

*Procedure:* The test procedures follow ISO 7539-2 to ISO 7539-7. ISO 7539-8 highlights the considerations that have to be taken when testing weldments.

**ISO 7539-9**: "Preparation and use of pre-cracked specimens for tests under rising load or rising displacement"

This part of ISO 7539 covers procedures for designing, preparing and using pre-cracked specimens for investigating the susceptibility of metal to stress corrosion cracking by means of a test conducted under rising load or rising displacement, i.e. specimens to be tested under ISO 7539-2 to ISO 7539-7.

#### 4.5.3 ASTM G 38: "Standard Practice for Making and Using C-Ring Stress – Corrosion Test Specimens"

This practice involves the preparation of and the quantitative stressing of a C-ring stress corrosion test specimen by application of a bending load. Characteristics of the stress system and the distribution of stresses are discussed. Guidance is given for methods of exposure and inspection.

The C-ring is a versatile, economical specimen for quantitatively determining the susceptibility to stress corrosion cracking of all types of alloys in a wide variety of product forms.

Materials: Metals and alloys.

Test Solution: To be selected.

*Procedure:* Exposure of a pre-loaded specimen to a corrosive environment.

*Weldments:* Suitable for testing of weldments either with or without a notch.

#### 4.5.4 ASTM G 39: "Standard Practice for Preparation and Use of Bent-Beam Stress-Corrosion Test Specimens"

This practice involves the quantitative stressing of a beam specimen by application of a bending stress. The applied stress is determined from the size of the specimen and the bending deflection. The stressed specimens are then exposed to the test environment and the time required for cracks to develop is determined. This cracking time is used as a measure of the stress corrosion resistance of the material in the test environment at the stress level utilised.

The bent-beam specimens are designed for testing at stress levels below the elastic limit of the alloy. For testing in the plastic range, U-bend specimens should be employed. See 4.5.5, ASTM G 30 below.

Materials: Metals and alloys.

Test Solution: To be selected.

*Procedure:* Exposure of a pre-loaded specimen to a corrosive environment.

*Weldments:* The method can be used for testing overlay welds, welded joints and all-weld metal samples.

#### 4.5.5 ASTM G 30: "Standard Practice for Making and Using U-bend Stress-Corrosion Test Specimens"

This practice is concerned only with the test specimen and not the environmental aspects of stress corrosion testing.

U-bend specimens usually contain both elastic and plastic strain. In some cases it is possible to form a U-bend and produce only elastic strain. However, bent beam specimens are normally used to study stress corrosion cracking under elastic strain only.

Materials: Metals and alloys.

Test Solution: To be selected.

*Procedure:* The practice involves the stressing of a specimen bent to a U-shape. The applied strain is estimated from the bend conditions. The stressed specimens are then exposed to the test environment and the time required for cracks to develop is determined. This cracking time is used as a measure of the stress corrosion resistance of the material in the test environment.

*Weldments:* The method can be used for testing overlay welds, welded joints and all-weld metal.

#### 4.5.6 ASTM G 49: "Standard Practice for Preparation and Use of Direct Tension Stress-Corrosion Test Specimens"

This practice covers the use of axially loaded, quantitatively stressed ASTM standard tension test specimens for investigating the resistance to stress corrosion cracking of metallic materials in all types of product forms. Consideration is given to important factors in the selection of appropriate specimens, the design of loading equipment, and the effects of these factors on the state of stress in the specimen as corrosion occurs.

Axially loaded tension specimens provide one of the most versatile methods of performing stress corrosion testing because of the flexibility permitted in the choice of type and size of test specimen, stressing procedures and range of stress levels. The uniaxial stress system is simple; hence, this test method is often used for studies of stress corrosion mechanisms. Test Solution: To be selected.

*Procedure:* A uniaxially stressed sample is exposed to a test environment, either gaseous or liquid.

*Weldments:* Tension test specimens are adaptable for testing a wide variety of product forms, as well as parts joined by welding.

### 4.5.7 ASTM G 58: "Standard Practice for Preparation of Stress Corrosion Test Specimens for Weldments"

This practice covers procedures for the making and utilization of test specimens for the evaluation of weldments in stress corrosion cracking environments.

Test specimens are described in which:

- a) Stresses are developed by the welding process only.
- b) Stresses are developed by an externally applied load, in addition to the stresses due to welding.

c) Stresses are developed only by an externally applied load, with residual welding stresses removed by annealing.

This practice is concerned only with the welded test specimen and not with the environmental aspects of stress corrosion testing.

The actual stress in test specimens removed from weldments is not precisely known and is not comparable to that in a similar component weld, because it depends on the level of residual stress resulting from the welding and subsequent cooling process from the welding operation, combined with any other residual stresses.

The intent of the practice is to indicate standard welded specimens and welding procedures for evaluating the stress corrosion cracking characteristics of weldments in corrosive environments.

Materials: Weldable materials and alloys.

*Test Solution:* The test medium and exposure times for the stress corrosion for testing of weldments may vary from long-term tests in plant equipment under operating conditions or in outdoor environments through to various laboratory test media.

*Process:* Preparation of various test specimens for stress corrosion cracking tests.

*Weldments:* This standard is particularly developed for preparing samples for evaluation of the susceptibility to stress corrosion cracking of weldments.

#### 4.5.8 ASTM G 123: "Standard Test Method for Evaluating Stress-Corrosion Cracking in alloys with Different Nickel Contents in Boiling Acidified Sodium Chloride Solution"

This boiling sodium chloride test method is used to evaluate wrought stainless steels, including austenitic-ferritic (duplex) stainless steels and alloys containing up to 33 % nickel. It may also be employed to evaluate these types of materials in the cast and welded conditions.

This test method detects major effects of composition,

heat treatment, microstructure, and stress on the sus-

Materials: Metals and alloys.

ceptibility of materials to chloride stress corrosion cracking.

This test method is also designed to provide better correlation with chemical process industry experience for stainless steels when compared with the more severe boiling magnesium chloride test of Practice G 36.

*Materials:* Stainless steels including duplex (Austenitic-Ferritic) stainless steels and alloys with up to 33 % nickel.

*Test Solution:* 25 % NaCl +  $H_3PO_4$  for pH adjustment to pH 1,5.

*Procedure:* A U-bend specimen (or other stressed specimen) of the material to be tested is put into the boiling solution. The test may be continued for as many weeks, as necessary, but six weeks (about 1 000 h) or less is expected to be sufficient to crack susceptible materials. It is recommended that samples of a susceptible material, for example type 304 or 316 are included as a control, when more resistant materials are evaluated.

*Weldments:* Suitable for testing stress corrosion susceptibility of weldments.

#### 4.5.9 NACE TM0177: "Laboratory Testing of Metals for Resistance to Specific Forms of Environmental Cracking in H<sub>2</sub>S Environments"

# EFC Guideline No. 17 "Corrosion Resistant Alloys for Oil and Gas Production: Guidance on General Requirements and Test Methods for $H_2S$ Service"

Both standards have been issued for evaluating material behaviour under stress corrosion conditions with respect to sour service environments, particularly in oilfield applications. The standards differ in the specified test solutions. However, the mechanical loading conditions are broadly similar for the different specimen types which are usually subjected to tensile stresses. Four different test specimen types are considered:

Method A: Standard tensile test.

Method B: Standard bent beam test.

Method C: Standard C-ring test.

Method D: Standard double – cantilever-beam (DCB) test.

Since these standards represent the most common procedures for the evaluation of stress corrosion cracking, the various specimen types are described in some detail.

Materials: Corrosion resistant alloys and steels.

*Test solution*: For the NACE test, a 5 % NaCl + 0.5 %  $CH_3COOH$  solution, saturated with various  $H_2S$  contents, is used as a test environment. The pH values must be controlled in the range 2.7 to 4.0. The lower pH of approximately 2.7 is recommended. The test solution should be maintained at  $24 \pm 3$  °C. The pressure and temperature of the test solution can be varied in order to study their effects on stress corrosion cracking behaviour of materials. Increasing temperature above 75 °C may reduce the stress corrosion sensitivity of a given material. However, the electrolyte specified in this standard is sometimes considered as being too aggressive

and is replaced by aqueous solutions, which are designed to provide more realistic conditions, similar to those specified in the EFC guideline. Careful selection of the electrolyte, regarding the pH and the buffering capacity, is therefore recommended.

The procedures, briefly described below, are increasingly applied using more realistic solutions, like the formation water compositions of real oil and gas fields.

*Procedure*: Stressed specimens are immersed in the electrolytic test solution saturated with  $H_2S$  at ambient temperature and pressure. The test duration varies, but 30 days is the most common practice. Convenient increments of mechanical load can be simulated to obtain stress corrosion cracking data. More information about the testing procedure is found in Appendix III.

Weldments: Can be used for testing of weldments.

#### 4.5.10 ASTM G 129: "Standard Practice for Slow Strain Rate Testing to Evaluate the Susceptibility of Metallic Materials to Environmentally Assisted Cracking"

NACE Standard TM 0198-98: "Slow Strain Rate Test Method for Screening Corrosion Resistant Alloys (CRAs) for Stress Corrosion Cracking in Sour Oilfield Service"

### (4.5.2 ISO 7539: "Corrosion of Metals and Alloys – Stress Corrosion Testing – Part 7: Slow Strain Rate Testing")

*Procedure:* The procedure involves the application of very slow strain rates to tension or notched or fatigue pre-cracked specimens exposed to a predetermined environment. The slow strain rates are achieved by a constant extension rate on the specimen while monitoring load and extension of the specimen. The Slow Strain Rate (SSR) test always produces fracture to the test specimen. The degree of susceptibility to EAC is generally assessed through observation of the differences in the behaviour of the material in tests conducted in the test environment from that obtained from tests conducted in the control environment.

Slow strain rate tests are adaptable for testing a wide variety of product forms, including plate, rod, wire, sheet and tubes, as well as composites of these and parts joined by welding. Notched or pre-cracked specimens may be used, as well as initially plain specimens. This standard does not cover corrosion fatigue.

The principal advantage of the test is the rapidity with which susceptibility to stress corrosion cracking of a particular metal or alloy/environment combination can be assessed. Thus, the slow rate test has been used for rapid screening or comparative evaluation, or both, of environmental, processing or metallurgical variables, that can affect the resistance of a material to stress corrosion cracking. For example, this testing technique has been used to evaluate materials, heat treatments, chemical constituents in the environment, temperature and chemical inhibitors. Corrosive environments may cause a degradation of the properties of stressed materials beyond those observed in the same environment at constant load over a limited period. Thus, and in contrast to tests with constant loading, an additional advantage of the test is to show hidden degradation of a specific material in a specific environment.

Moreover, this standard is also one of the appropriate methods to be used for evaluation of stress corrosion cracking behaviour of welded components.

*Materials*: Corrosion resistant alloys and steels, light alloys.

*Test solution*: Frequently, the NACE TM 0177-96 solution, 5 % NaCl + 0.5 % CH<sub>3</sub>COOH with various H<sub>2</sub>S saturations is employed if the steel components are designed for exposure to sour service environments. However, increasingly media corresponding to real environments are specifically adopted for evaluating material resistance against stress corrosion cracking.

*Procedure*: The test consists in subjecting a specimen to an increasing, mainly unidirectional, strain whilst exposed to a specified environment with a view to determining stress corrosion susceptibility.

Tests may be conducted in tension or in bending, on plain, notched or pre-cracked specimens. The most important characteristic of the test is the relatively slow strain rate generated at the region of crack initiation or growth in the metal. Such strain rates in a range of  $10^{-3}$  to  $10^{-7}$  s<sup>-1</sup>, or even lower, should be controlled in order to avoid failure of the components by mechanical overload without any effects of stress corrosion cracking. A constant strain rate of  $10^{-6}$  s<sup>-1</sup> is recommended for testing plain CRA specimens. Additionally, temperature and pressure during testing, particularly at increasing strain rates influencing the characteristic of stress corrosion cracking must also be specified.

After complete rupture of the specimen, visual observation and stereo microscopy should at least be applied to verify whether the specimen failed by stress corrosion cracking or simply by mechanical overload. Furthermore, electron microscopic observations should be performed to get a better understanding of cracking behaviour in the specific region and of the fracture topography. In particular, such additional evaluations distinguish the slow strain rate test from simple crack-no-crack and ranking procedures and can provide some insight into cracking mechanisms.

*Weldments*: This standard can be adopted for testing weldments. For better evaluation of the results, the specimens should have a gauge length in which the welds are included. It should be noted, that the different alloying compositions and the thermal history of the weld metal during welding may result in significantly different mechanical properties and stress corrosion cracking resistance and the test results should be interpreted bearing this in mind. Notched or pre-cracked specimens may be used when it is desirable to restrict cracking to a particular location, for example, when testing the heat-affected zone of a weldment.

#### 4.5.11 NACE TM 0284-96: "Evaluation of Pipeline and Pressure Vessel Steels for Resistance to Hydrogen-Induced Cracking"

This test applies to a special form of environmentally assisted cracking called Hydrogen Induced Cracking (HIC) and covers all formerly used expressions for cracking symptoms like blistering, stepwise cracking etc. caused by hydrogen absorption from aqueous sulphide environments. In contrast to the procedures described above, the test consists in exposing unstressed test specimens to one or two standard test solutions saturated with  $H_2S$  at ambient pressure and temperature. The test is not intended to reproduce real conditions, but serves as a ranking test distinguishing between different materials with respect to their HIC susceptibility.

Materials: Pipeline steels.

*Test Solution:* Solution A (5 % NaCl + 0.5 % CH<sub>3</sub>COOH) or solution B (Synthetic seawater), saturated with H<sub>2</sub>S.

*Procedure:* Test specimens cut out of the vessel (pipe) parent material or cross sectional to respective welds are placed in the test vessel with the wide faces separated from the test vessel and other specimens by glass or other non-metallic rods. The specimens are subjected to the previously de-aerated test solution which is purged with  $H_2S$  for 96 hours at ambient temperature. After the test, the specimens are sectioned and the crack sensitivity can be measured by different ratios. Most frequently, the crack length is related to the width of the specimen (crack length ratio).

*Weldments:* Suitable for testing HIC susceptibility of weldments.

#### 4.5.12 NACE MR 0175/

#### ISO 15156: "Petroleum and natural gas industries – Materials for use in H<sub>2</sub>S containing environments in oil and gas production"

This standard combines the efforts of the EFC and NACE to establish procedures and standards to evaluate the cracking resistance of materials in  $H_2S$ -containing environments. The standard includes and refers to the different test procedures developed by NACE and EFC, but does not describe them in detail. It represents more of an overview to guide materials selection in the oil and gas industries.

Part 1 provides requirements and gives recommendations for the selection of metallic materials for service in equipment used in oil and gas production and natural gas sweetening plants in  $H_2S$ -containing environments. In particular it describes all the mechanisms of cracking that can be caused by  $H_2S$ -containing environments.

Part 2 refers particularly to carbon and low alloy steels as well as to cast irons, while Part 3 focuses on CRAs. These two parts contain very helpful information about weldments with respect to hardness limitations for the materials to be used. However, when compared to the original test standards they do not provide additional information on the testing of weldments, weld sections or welded components.

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#### 4.5.13 MTI-3 Test Method: "Laboratory testing of Iron- and Nickel-Based Alloys for Relative Resistance to Stress Corrosion Cracking in a Boiling Magnesium (MgCl<sub>2</sub>) Solution"

This method describes the procedure for corrosion tests to determine the relative resistance of wrought iron- and nickel-based alloys to stress corrosion cracking in a boiling magnesium chloride solution. It also results in a time to failure number which can be used to rank alloys for their relative resistance to stress corrosion cracking in this environment. This ranking may or may not be applicable to other chloride environments.

*Materials:* Wrought materials of Iron- and Nickel-Based Alloys

*Test solution:* Magnesium chloride solution with boiling point  $155 \pm 1.0$  °C.

*Procedure:* The test specimens shall be U-bend specimens. After the solutions boiling temperature has been adjusted to the specific value of  $155 \pm 1.0$  °C, the test specimen(s) are immersed in the solution. Three separate tests shall be made. One of 24-hour duration, one of 96, and a third of 500-hour duration. The specimens shall be examined at the end of each test for signs of cracking.

*Weldments:* The test can be performed on weldments with the weld in the centre of the U-bend.

#### 4.5.14 MTI-5 Test Method: "Laboratory Testing of Iron- and Nickel-Based Alloys for Relative Resistance to Stress Corrosion Cracking in a Sodium Chloride (NaCl) Drop Evaporation System"

This method describes the procedure for corrosion tests to determine the relative resistance of wrought iron- and nickel-based alloys to stress corrosion cracking in a drop evaporation system. The result is presented as a minimum stress to failure number which can be used to rank alloys for their relative resistance to stress corrosion cracking in a sodium chloride solution. This ranking may or may not be applicable to other chloride environments.

*Materials:* Wrought materials of Iron- and Nickel-Based Alloys.

Test solution: 0.6 % NaCl solution.

*Procedure:* The test specimen shall be tension specimens with a circular cross section in the gage length. The specimen is attached to the gripping devices and the specified load is applied by direct loading in tension. The load applied shall be one of the following fractions of the 0,2 percent offset yield strength of the alloy at  $200 \,^\circ$ C: 0.1, 0.3, 0.5, 0.7, 0.9.

The specimen is resistance heated to a temperature of 300 °C. The test solution is dripped onto the test section of the specimen at a rate of 40 drops per minute. The test is continued until the specimen ruptures, up to a maximum time of 500 hours. The time to rupture is recorded.

Weldments: The test can be applied to weldments.

#### 4.6 Corrosion fatigue

#### 4.6.1 ISO 11782-1: "Cycles to failure testing"

The study of cycles to failure testing uses plain or notched specimens to provide data on the corrosion fatigue behaviour of a metal or alloy and can be used to develop criteria for engineering design to prevent fatigue failures. It can be applied to a wide variety of product forms including plate, rod, wire, sheet and tubes including parts joined by welding. The testing environment can be aqueous or gaseous.

The test may also be used as a guide to the selection of materials for service under conditions of repeated applied stress under known environmental conditions.

Materials: Metals and alloys.

Test Solution: To be selected.

*Procedure:* In the presence of an aggressive environment the fatigue strength of a metal or alloy may be reduced to an extent which depends on the nature of the environment and the test conditions. The tests involve subjecting a series of specimens to the number of stress cycles required for a fatigue crack to initiate and grow large enough to cause failure during exposure to a corrosive or otherwise active environment at progressively smaller alternating stresses. This is done to define either the fatigue strength at N cycles,  $S_N$  from an S-N diagram or the fatigue strength limit as the fatigue life becomes extended.

*Weldments:* The method can be used for testing welded joints as well as all-weld metal samples.

# 4.6.2 ISO 11782-2: "Crack propagation testing using pre cracked specimens"

This part of ISO 11782 describes the fracture mechanics method of determining the crack growth rates of preexisting cracks under cyclic loading in a controlled environment. It includes the measurement of the threshold stress intensity factor range for crack growth, below which the rate of crack advance falls below some defined limit.

Materials: Metals and alloys.

Test Solution: To be selected.

*Procedure:* A fatigue pre-crack is induced in a notched specimen by cyclic loading.

The corrosion fatigue crack propagation tests are then conducted using cyclic loading under environmental and stressing conditions relevant to the particular application. During the test, crack length is monitored as a function of elapsed cycles.

*Weldments:* The method can be used for testing welded joints as well as all-weld metal samples.

## 4.7 Galvanic corrosion

#### 4.7.1 ASTM G 71: "Standard Guide for Conducting and Evaluating Galvanic Corrosion Tests in Electrolytes"

This guide is used for conducting and evaluating galvanic corrosion tests to characterise the behaviour of

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two dissimilar metals in electrical contact in an electrolyte under low-flow conditions. It can be adapted to wrought or cast metals and alloys.

It also covers the selection of materials, specimen preparation, test environment, method of exposure, and method for evaluating the results to characterise the behaviour of galvanic couples in an electrolyte.

This standard is presented as a guide for conducting galvanic corrosion tests in liquid electrolyte solutions, both in the laboratory and for in-service environments.

Galvanic corrosion tests are conducted in the laboratory for several purposes:

1. Inexpensive screening to reduce expensive field testing.

2. Study of the effects of environmental variables.

3. Study of the corrosion accelerating or protective effects of various anode/cathode surface area ratios.

The materials proven in the laboratory to be the most promising should also be tested in the field.

Materials: Metals and alloys.

*Test Solution:* To be selected. Commonly a process solution.

*Process:* A coupled assembly is immersed in a test electrolyte for a certain time of exposure. Evaluation is usually based on mass loss.

Weldments: This guide can be used on weldments.

## 4.8 Field testing

# 4.8.1 ASTM G4. Standard Guide for Conducting Corrosion Coupon Tests in Field Applications

This guide covers procedures for conducting corrosion coupon tests in plant and equipment under operating conditions to evaluate the corrosive attack upon engineering materials. It does not cover electrochemical methods for determining corrosion rates.

While intended primarily for immersion tests, general guidelines provided can be applicable to exposure of test coupons in plant atmospheres.

Observations and data derived from coupon testing are used to determine the average rate of corrosion and the type of attack that occurs during the exposure interval. The data may be used as part of an evaluation of potential construction materials for use in similar service environments or for replacement materials in existing facilities.

Materials: Metals and alloys.

Test Solution: Process environment.

*Process:* Suitable coupons of various shapes and sizes are made for immersion at a desired location in the process vessel or pipework. After exposure for a chosen length of time, the specimen should be carefully examined using low power magnification to establish the type and uniformity of surface attack such as etching, pitting, de-alloying or parting, tarnishing, filming, scaling etc.

*Weldments:* Since welding is a principal method of fabricating equipment, welded coupons should be included as much as possible in the test programme. Major areas of interest are the effects of residual stresses from welding, the relative corrosion resistance of the weld bead and heat-affected zone and galvanic effects between base metal and weld metal.

# APPENDIX I ENVIRONMENTALLY ASSISTED CRACKING

Historically, the causes of in-service damage arising from stress corrosion cracking have primarily been associated only with constant mechanical loading. For this reason, in earlier test procedures, both plain and notched specimens were principally subjected to consistent uniaxial tensile stresses.

As many of these test procedures take up a considerable amount of time, two kinds of faster testing technology have been developed during the nineteen-sixties. The first kind is based on the application of pre-cracked specimens which, in linear-elastic fracture mechanics tests, are exposed to a constant load. The second kind uses plain or notched specimens tested under a constant, low strain rate. Both test types are currently specified methods, but the evolution of microelectronics has enabled test machines of higher precision to be developed. Therefore, a variety of non-standardised test methods is available in addition to those mentioned above. One of these advanced methods, for example, consists in testing high-strength low-alloyed steels using elastic-plastic fracture mechanics specimens under low cyclic strain.

All test methods for determining the resistance of a material to stress corrosion cracking have principally been developed regardless of whether it is caused by anodic processes or under the action of hydrogen. In the following analysis, however, the suitability of these test methods is dealt with exclusively with a view to assessing the stress corrosion resistance of a material.

The stress corrosion test methods can furthermore be used to establish several different criteria for the assessment of the risk of cracking. Five categories of prime importance are briefly outlined below:

## I Time To Failure (TTF)

Many test methods use "Time To Failure" (TTF) as a criterion for the resistance of a material in a particular environment. This time includes crack initiation, as well as crack propagation, and finally the failure of the entire specimen due to unstable fracture of the remaining cross-sectional ligament. These three stages of stress corrosion cracking cannot be accurately differentiated on plain specimens by means of the TTF-value. By contrast, this value is a suitable means for precisely determining the period of stable crack propagation in notched specimens. In addition, the real service conditions for gaps and flanges can be more realistically tested by reproducing the respective notch geometry.

#### II Limit values of the bearable stress

Plain specimens are loaded within a defined stress range. This allows the lowest stress,  $\sigma_i$ , at which cracking occurs, or the highest stress,  $\sigma_{H}$ , at which cracking no longer occurs, to be determined. The value of the highest stress is usually confirmed by using at least three specimens. Generally, the mean value between the two values of  $\sigma_{\!\scriptscriptstyle L}$  and of  $\sigma_{\!\scriptscriptstyle H}$  is determined as the threshold value of the bearable stress,  $\sigma_{\text{thres}}.$  However, the bearable stress is only a value for characterizing the stress corrosion susceptibility of a material in a particular medium. It cannot be used for structural design of components because of the relatively poor transferability of mechanical stressing and corrosion exposure from laboratory tests to real conditions. Since the threshold value,  $\sigma_{thres}$ , is always determined under a constant mechanical stress, the values obtained are insufficiently conservative for real component loading where stresses are variable with time.

#### III Specimen failure rate

Another possible assessment criterion is the specimen failure rate. A number of specimens are subjected to same stress and corrosion conditions and for a defined period of time. The criterion is based on the percentage of failed specimens after a given period of time compared with the number of specimens being tested. The data can be plotted graphically as a percentage failure rate against time.

#### IV Limit values of the stress intensity factor

The three stages of crack growth are frequently determined with the help of diagrams in which the crack velocity, V<sub>crack</sub>, is plotted versus the stress intensity factor, K. From those diagrams, a critical stress intensity factor, K<sub>ic</sub>, can be derived at which unstable specimen fracture starts independently of the environment. In tests with decreasing stress intensity factor, crack arrest may take place. The limit value of the stress intensity factor for the occurrence of stress corrosion cracking is, in the following, referred to as  $K_{ISCC}$ . Concerning the test procedures, it must be stated that the phase of stable stress corrosion crack growth is investigated at a crack velocity which is constant and hence independent of the stress intensity factor, i.e. taking account of the limit values KISCC and KIC. However, the separation between the crack initiation and the crack propagation phases, with the help of the K<sub>ISCC</sub>-value, should be taken into consideration. This aspect has been proposed in the literature, but has, as yet, not been validated by any acceptable model.

#### V Material properties and loss of ductility

Apart from the usual strength and toughness values of a material, the loss of ductility (*LD*) is a third parameter especially in the testing of hydrogen-charged specimens. Assessment is based on reduction of area (*RA*). The loss of ductility is determined from the reduction of area in a specific medium,  $RA_{env}$  compared with the reduction of area under inert conditions,  $RA_o$ , according to the following equation:

Eq. 1 
$$LD = 1 - \frac{RA_{env}}{RA_o}$$
,

where

*LD* is in most cases multiplied by 100 and indicated in per cent.

Alternatively, the normalized loss of ductility,  $RA_n$ , can be determined:

Eq. 2 
$$RA_n = \frac{RA_{env}}{RA_n}$$
,

which is also, in most cases, multiplied by 100 and indicated in per cent.

Unlike the precisely specified mechanical parameters, the test solutions vary significantly, not only between the individual test procedures, but also between the various standards. In NACE TM 0177-96, for example, an electrolyte with 5 % NaCl and 0.5 % CH<sub>3</sub>COOH saturated with H<sub>2</sub>S is specified, which is a highly sour medium with a pH range of  $2.7 \le pH \le 3.0$ . In contrast, the European Federation of Corrosion (EFC) proposes a test medium for the same test methods of 5 % NaCl and 4 % CH<sub>3</sub>COONa, with the pH-value being set to correspond to the real environment.

ISO 7539 states that all tests be conducted in electrolytes with compositions that exist in real environments. This may involve completely different conditions for the crack and hence exert an influence on the time to specimen failure. However, the application of real electrolyte compositions might yield more representative test results.

It is not the role of this document to enter the debate on testing environments, particularly since the issues of test pressure and temperature and oxygen content, when compared with real environments, have still to be resolved.

#### APPENDIX II FULL SCALE TESTING

Not only during fabrication, as for instance welding, but also during service components are subjected to multiaxial loads which, in addition, might vary with time. The inadequacy of conventional test methods in simulations of such real mechanical loading and frequently also the nature of the corrosive environment has therefore originated *Full Scale (FS) Test* procedures, of which the objective is maximum possible realistic coverage of fabrication- and service-specific mechanical stressing and respective corrosive environments. Such test procedures are not standardized up to the present, but are included into this document, because they have been designed particularly to investigate the life cycle behaviour of welded components, as for instance for investigating crack phenomena in welded pipelines for the transport of corrosive media. Full Scale Tests are now increasingly being used for examinations in sour service in order to draw correct conclusions about the construction and maintenance costs, and thus about the profitability of a tested material and also about that of the welding process, if necessary.

A series of requirements can be derived from the procedures developed up to the present:

#### I Selection of realistic samples

Pipes in the condition of their subsequent application should serve as test pieces for pipeline construction. This implies that they should have the same dimensions and the commonly used surface condition in order to realize the same mechanical loading and corrosion system. The specimen dimensions should contain at least one of the joints intended for practical application, whether these are welds, flanges, threaded or bayonet couplings. A Full Scale Test not only represents a combined corrosion test of the welded component, predominantly aiming at the evaluation of the stress corrosion cracking resistance, but also accounts for other forms of corrosion at welded components. Thus for example, local corrosion and, in particular, the effects of such corrosion forms on stress corrosion cracking will also be simulated. Potential concomitant phenomena such as cathodic protection of large areas, resulting from local crevice corrosion of flanges, can thus also be covered in such procedure. Additionally, tubular components can be subjected to different internal and external environments. Effects arising from different types of joints can additionally be identified. From the literature, appropriate test lengths are indicated to be:  $L \ge 1$  m for pipes with an outside diameter of up to  $D_0 \le 100 \text{ mm}$ and 4 m  $\leq$  L  $\leq$  12 m for pipes with D<sub>o</sub>  $\geq$  100 mm.

#### II Mechanical loading

The mechanical loading of the test specimen should cover as completely as possible the full spectrum of fabrication and service load. Regarding welded components, this means that the welds should be carried out at the same shrinkage restraint as provided by the real welded structure, in order to achieve the same distribution of residual stresses and strains in the welds. Also additional loading during fabrication of real structures as, for instance, bending, corresponding to reeling and de-reeling processes, should be considered. Finally, the real service loads should be superposed to such fabrication loads, as they might arise from pressurizing, free spans and shut downs etc., for instance in pipeline systems.

The transfer of the mechanical loads to the test procedure demands great care in determining the real loads. To cope with this task, computational simulation using the finite element method is, in many cases, an obvious choice.

#### III Corrosive environment

Full Scale Tests, particularly those designed for a pipeline application spectrum of a specific reservoir, require accurate determination also of the natural transport media before starting a test. Regarding sour service, apart from the Cl ion concentration, the variation range of the H<sub>2</sub>S- and CO<sub>2</sub>-content should for most of the part be defined. Some authors recommend additions of respective quantities of gas, water, oil and sand to the fluid such that the composition is, in some cases, identical with that of the natural medium conveyed. For reproducing extremely harmful conditions, e.g. during flow-standstill, the surface-protecting oil constituents should, however, be removed, if possible.

In addition, for the investigation of welded pressurizing pipes made of high strength steels for hydro power plants, for instance, realistic formation water composition should be employed.

The reservoir of the test solution should have enough capacity to fill the assembly up with the fluid and to provide sufficient reserves for losses and mixture preparation. Use of 1 m<sup>3</sup> of electrolyte in a Full Scale Test, of which however only half is contained in the test assembly itself, seems to be a good choice for welded pipes having a diameter of about 12 inches. Besides, the reservoir should be closed, but provided with a safety valve to prevent leakage of gases which, if they are toxic, must be passed over to an appropriate separator. The individual gases required for the mixture preparation must be separately stored to provide most manifold variation possibilities for the fluid composition in Full Scale Testing.

In order to simulate pipeline applications in which the material is also subjected to an outside environment, the total test length should be buried or submerged in a receptacle with a covering which can be filled, for example, with earth or water corresponding to the real environment. In order to create biochemical conditions as experienced by buried or submerged pipelines, the receptacle could additionally be flushed with corresponding gas mixtures. Respective quantities of the various acid-forming and sulphate-reducing bacteria should also be added and monitored, if such situations might be encountered in practice. Enclosing the test pipe in a receptacle also confers additional protection against explosion and escape of toxic gases (H<sub>2</sub>S) into the laboratory environment, when material failure occurs under the high pressures which are frequently used.

#### IV Test assembly

For reproducing real flow conditions of oil flowlines, the test assembly should be incorporated into a flow loop. This equipment should include a hydrodynamic run-in length to keep equipment-specific turbulence away from the test length itself. In addition, the representative flow should be established ahead of the test length, in order to provide constant flow conditions inside of the test specimen. The research activities concerning the influence of corrosive media flow characteristics on stress corrosion cracking are still in their initial stages and are therefore not yet reflected in practical Full Scale Tests. However, it is a proven fact that specifically the creation of so-called "slug flow", i.e. turbulent flow producing upright backward waves, must be considered as particularly harmful with respect to stress corrosion cracking. The reproduction of such flow opens up far-reaching interdisciplinary research perspectives for Full Scale Tests. Concerning the execution of such flow reproduction tests, it should be noted at this stage that quick and versatile closed-loop controlled pumps for a wide pressure range are needed to generate timely variable fluid flow.

Further examples of flow turbulence, which may occur during service, e.g. in bent areas, T-joints and pipe manifolds, should be examined using an additional test length to exclude interactions.

All the other conduits, pumps, measuring instruments and reservoirs, in which the test medium flows, must be more corrosion resistant than the test length. Special attention must be given to this aspect in order to secure galvanic separation of the test material from the rest of the assembly and to prevent unwanted side effects resulting from contact corrosion. The materials to be used for the separation should also be heat and corrosion resistant. PTFE exhibits these properties and is therefore an obvious choice.

Generally, the structural design of the assembly should be flexible enough to allow, as far as possible, simultaneous testing of several test pieces using different load levels, for example. The equipment should also include special devices capable of accommodating different test piece configurations, such as pipe bends, flanges and manifolds.

#### **V** Measurements

In view of the fact that Full Scale Tests procedures are already very cost-intensive and time-consuming, the test assembly should be instrumented as generously as possible with measurement devices to get a maximum of results from one test phase. The mechanical stressing should be measured at a location as near as possible to the test piece, i.e. the weld, in order to suppress losses and transmission errors. To accomplish the same purpose, it is advisable to measure also the pressure, the temperature and the flow rate of the fluid shortly before it enters the test pipe. However, the pH-value, the potential and the concentration of the fluid should be measured in the reservoir, where the flow rate of the medium is lower.

Full Scale Tests represent today the most realistic laboratory test procedures. The last step that remains to be taken towards supremely realistic conditions in materials testing is to use pipes in lengths representative of those used by the oil and gas industry.

The above recommendations can be summarized by the key headings as for the other test procedures:

Materials: All weldable materials.

*Test solution*: Corrosive media in Full Scale Tests also should correspond as far as possible to the real conditions. This is the reason why standard electrolytes are generally not used in these test procedures. These experiments should be performed with the natural composition of the medium which should also be at its real temperature and pressure. Flow rate and turbulence of the electrolyte must also be taken into account. Furthermore, the material surface in the Full Scale Test should also correspond to the true service conditions.

*Procedure*: Much more attention than in standard tests, in which less crack-critical parameters are predominantly determined, must be paid in Full Scale Tests to keeping the predefined test conditions constant over the full test period, to allow for separation of different effects. Therefore, the test procedure should utilise real conditions in which welded components may be degraded by stress corrosion cracking.

*Weldments*: Full Scale Tests probably provide the most comprehensive data and insight into the corrosion resistance and service behaviour of welded components. They are particularly suitable for assessing the resistance of weldments in comparison to the base material.

# APPENDIX III APPLICATIONS OF STRESS CORROSION STANDARDS

### <u>Method A – Uniaxial tensile specimens</u> (Tensile test)

#### I Constant Load Test

One of the most widely used is the Constant Load Test (CLT) which corresponds to a creep rupture test in a specific corrosive medium.

This test procedure is applied predominantly in the oil and gas industry for evaluating the susceptibility of materials to stress corrosion cracking. In particular, it is used to investigate materials of non-welded components for transporting  $H_2S$ -containing media in pipes and pipelines. In all these applications, EFC recommends this test as a *Fitness for Service Test*.

In many cases, the Constant Load Test is conducted under 'real' conditions, e.g. at elevated temperature and high pressure.

These tests are carried out by selecting a specimen loading as high as the expected in-service loading which, in the case of pipelines and oil production plants, is near the yield strength or proof stress of the material to be used. Hence, the criterion for the acceptance of materials for pipework and pipelines in oil and gas production following the EFC-guidelines equates to an allowable stress of  $\sigma_{th}\approx 0.9\cdot R_{\text{po}.2}$ .

Unlike frequent real multiaxial loading in practice, the stress in this test procedure, can only be imposed unidirectionally. It has to be appreciated that the tensile specimens are able to creep during the Constant Load Test in an uncontrolled manner, thus changing this test into a type of test operating with constantly decreasing plastic deformation. This means that the limit stress called for in many specifications for this test, e.g. 90 % of the yield strength or proof stress, is perfectly reasonable.

EFC considers a test duration of 720 hours to be sufficient for assessing the resistance of a material to stress corrosion cracking. The reason for this recommendation is that exceeding this time limit could lead to pitting corrosion of the specimen surface, which could invalidate the test result, if local stress concentrations and hydrogen were present.

It should be noted that materials employed in oil and gas production are usually exposed for several months or years to  $H_2S$ -containing media. Consequently, if a material does not fail in the Constant Load Test, this is not an assurance that no damage will occur, later, in service. Furthermore, the limited test duration may result in any possible relationship between pitting corrosion and stress corrosion cracking going unrecognised. It is therefore recommended the test duration be prolonged to at least the time when pitting corrosion begins to take place, in order to establish whether or not it has any influence on stress corrosion cracking.

#### **II** Sustained Load Test

The NACE standard differentiates between the CLT and the Sustained Load Test (SLT). In the latter test, a tensile specimen is axially clamped into a ring-shaped frame. Another variant consists in pre-loading the specimen using a calibrated spring. In this procedure, the global stress,  $\sigma_{\mbox{\tiny alob}},$  remains unchanged, at first, as in the Constant Load Test. However, when crack initiation occurs, the specimen extends in an axial direction causing a decrease of the load imposed by the frame. The global stress  $\sigma_{\mbox{\tiny glob}}$  remains constant only until the crack is initiated. Hence, in order to avoid a decline in the local stress  $\sigma_{loc}$  as the crack grows, the elastic clamping in the test rig must be appropriately high. To summarise this, it appears that the SLT is more realistic than the CLT, because real components in service will also show some compliance or relaxation when a crack has been initiated.

But in this context the question arises of how realistic an extrinsic compliant axial tensile loading is with increasing crack propagation, as it is simulated in a Sustained Load Test. Realistic loading would only be conceivable in cases where the crack, and the region in the component through which it passes, are relatively large compared to the global structure. Usually, failure by hydrogen-assisted stress corrosion cracking takes place in regions with the highest stress levels, i.e. in notched thin cross-sections. Systems consisting of very stiff specimens and soft frames are therefore not very representative of structures in which stress corrosion cracking -susceptible areas are surrounded by thick walls.

### III Constant Strain Test (CST)

If the test frame is very stiff, the specimen undergoes plastic straining whilst the frame itself remains elastic. Once a crack has initiated, the stress at the crack tip will first increase on account of the local stress concentration. However, finally, it will decline, as the specimen extends within the stiff frame. This means that with a very stiff frame, straining will be limited and may be insufficient to cause tearing (i.e. crack extension). Thus, in Constant Strain Tests, the test frame is much stiffer than the specimen, whilst in a Sustained Load Test, the test frame is softer than the specimen. On the basis that the test frame takes over the function of a real component, Constant Strain Tests are more typical of real structures than Sustained Load Tests and therefore reflect engineering applications more frequently.

This makes it clear that when deciding between carrying out tests with constant load or with constant strain, the decision must be related to specific applications, i.e. whether, in service, the component will be subjected to a constant load or a fixed displacement.

As compared to Constant Load Tests, Constant Strain Tests in conformity with the EFC-guidelines are regarded as less severe, thus requiring specimen loading up to the proof stress or yield strength. For the purposes of determining loads in a Constant Strain Test, it is necessary to take account of the influence of temperature on material strength.

For the case of elevated temperature testing, it is possible that uncontrolled creep may occur during a Constant Strain Test. This effect assumes special significance when the test material and the frame material have different thermal expansion coefficients, or are physically separated from each other by plastic material, which may experience significant creep, even at room temperature.

Microcreep, however, may also occur at lower loads than those specified and cannot, therefore, be excluded in any test using constant loading of plain specimens. For this reason, it is reasonable to carry out several tests under the same conditions using various amounts of mechanical loading.

It is reasonable, furthermore, to check each completed test, which has been carried out on plain specimens with constant loading, by subsequently conducting a standard tensile test under normal conditions. This would reveal any possible degradation of mechanical properties resulting from corrosion reactions during the original test.

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### Method B - Bending specimens (Bend Tests)

In contrast to uniaxial tensile loading, the loading of the test pieces may also be performed by bending. Tests using bent specimens are less elaborate and are preferred when space is limited for the accommodation of large test machines. It should however be considered that only half of the material surface is under tension, which additionally exhibits a gradient in the plate thickness direction and becomes zero at the neutral axis.

Consequently, the results of tests with test pieces subjected to bend loading may differ significantly from those using axial loading of tensile specimens. In addition, the various shapes of bending specimens involve different stress distributions over the length and width of the specimens as well as a two-dimensional stress condition in some specimens. Therefore, the results of tests using bending specimens are also not directly comparable to each other. In the Bend Beam Test (BBT), the first type of test set-up is based on bending beam-shaped test strips which primarily allows testing of materials available in the form of plates or disks. But it is also possible in this test to prepare test material from pipes or round billets.

Following the principle of constant strain, a relatively soft specimen is provided with stiff fixturing such that the global strain,  $\sigma_{glob}$ , decreases with crack extension. For each test, the highest tensile stress at the surface of the test piece is selected as the test stress. Only tests with elastic specimen loading can be evaluated, since test stresses above the yield strength or proof stress, are difficult to predict by computation.

Specimen geometries available include: Two-, Threeand Four-Point Bend Test (2, 3 and 4PBT), the Welded or Bolt Loaded Double Beam Test (WDBT or BLDBT, resp.) and the Constant Moment Beam Test (CMBT).

The specimen dimensions and further specific features of the tests are shown in the standards. The Two-, Three- and Four-Point Bend Tests are most frequently used. In both the Two- and the Three-Point Bend Test. the maximum tensile stress is in the beam centre and drops to zero at the specimen ends. In practice, the 2PBT and the 4PBT are preferred over the 3PBT, since crevice corrosion is a potential hazard at the contact points in the 3PBT or also in the Shell Type Test (STT) described in detail by Ikeda et al. and takes place at the point of maximum mechanical loading. The electrochemical conditions present at this location may differ in such cases from those on the rest of the surface. In comparison to the 2PBT and the 3PBT, the 4PBT offers the advantage of constant stress between the two internal loading points. This is also true for the WDBT in which constant stress prevails in the beams at the height of the wedge. But this test method, as compared to the 4PBT, suffers from the drawback that weld defects at the ends may cause inhomogeneous stress distributions in both beams. For this reason, an alternative is suggested in the EFC-guideline allowing bolting of the ends.

*Weldments:* Among all tests using bent beam specimens, only the CBMT configuration provides equal

stress over the entire length of the test piece. This effect makes this test particularly suitable for situations in which the quantity of available test material is limited. In conformance with the EFC guidelines the 4PBT is considered to be suited also to weld testing in view of the susceptibility to hydrogen-assisted stress corrosion cracking. In this test, the weld is preferably loaded in transverse direction, but longitudinal loading may be applied as well. However, such load imposition may at best only approximately reproduce the in-service loading of a welded component.

This is not at all sufficient, particularly since additional residual stresses are present in a weld as a result of the welding process. The level of the reaction stresses resulting from restrained weld shrinkage depends on the respective component stiffness, and is unlikely to be transferred in direction and height to the simple configuration. This means that the real shrinkage reaction stresses of welds cannot accurately be simulated in the 4PBT and that its suitability for weld testing has thus to be called into question.

# <u>Method C – Bending specimens</u> (C-Ring Specimens)

Ring specimens bent with the help of screws are above all suited to pipe testing, since they can easily be prepared and have no size limit. For accurate specimen preparation and loading, it should be ensured that the outside diameter is  $D_0 \ge 20$  mm.

C-ring specimens are often used to investigate the susceptibility of pipeline materials to stress corrosion cracking, since ring expansion produces inside tension stress and outside compression stress which largely corresponds to real radial pipe wall pressure loading.

C-rings are typical specimens for constant straining. But with the help of a calibrated spring it is also possible to impose a largely constant load on them. Another option is to produce a multiaxial stress condition by providing the specimen with a notch which makes it necessary to determine the stress prevailing in the notch root with the help of the respective stress concentration factor.

But what applies to bend beam specimens is also true for ring specimens, namely that the global stresses,  $\sigma_{glob}$ , decrease with increasing crack length as soon as crack initiation occurs. Likewise, the circumferential stress is not uniformly distributed. Maximum stressing takes place in the bow centre and drops to zero in the two fixturings. The most accurate method of determining the specimen loading relies on the application of strain gauges along the tensile loaded surface. Analogously to bend beam specimens, plastic loading of C-rings is also not recommended, unless an exact correlation of deformation can be established between the strain gauge measurements and a stress-strain diagram of the tested material.

*Weldments:* Testing of welds appears to be doubtful due to the equally poor transferability of the real shrinkage reaction stresses, as described for the bend beam specimens.

Unlike C-rings, so-called O-rings are subjected to uniform stress over the entire circumference by pulling an oversize plug over the ring. The O-Ring Test is regarded as more realistic than the other bending specimens, since it permits the reproduction of in-service loadings of structures with tight fits. In order to ensure maximum possible uniformity of circumferential stress, it should be made sure that the O-ring width does not exceed the four-fold wall thickness.

Varying tensile stress versus wall thickness suggests that this test should also be used for examining stress corrosion cracking in circumferentially welded pipelines in order to reproduce, for instance, stresses in the pipe wall produced by overpressure. But right from the beginning, practical limits are set to this application profile on account of crevice corrosion to be anticipated between bolt and ring.

#### Method D – Double Cantilever Beam Test

#### I Cantilever Bend Specimens

In analogy to the Bend Beam Test, single-edge notched cantilever-type specimens are used in the Cantilever Bend Test (CBT) which are bent around three or four contact points. But unlike plain specimens, they are subjected to a constant load such that they represent a typical test set-up for exact determination of stress intensity factors increasing with the crack length. Crack growth is usually measured with the help of strain gauges at the point of force introduction. The specified specimen geometry is shown in both standards.

#### **II Compact Specimens**

The test with compact specimens (Compact Test, CT) is normally carried out by providing the specimen with a constant crack opening using a wedge, even though

constant loading of such specimens is stated in some literature references. This means that the stress intensity factor will decrease with increasing crack length.

Equations for calculating the critical stress intensity factors  $K_{\rm ISCC}$  and  $K_{\rm Ic}$  can be adopted from the relevant standards.

#### III Double cantilever beam specimens

Among all fracture mechanics tests, the Double Cantilever Beam Test (DCBT) is most frequently used for determining the stress corrosion cracking resistance of a material. In the seventies and eighties, this test method was often used to assess the resistance of lowalloyed martensitic steels to H<sub>2</sub>S-containing media. In contrast to the similar CT-specimens, such samples are suitable for studying crack growth across a broad spectrum of stress intensity factors. In addition, the specified low height/length ratio allows sampling from thinwalled components and even from larger pipelines. In order to assure a plane strain condition, prediction of the K<sub>ISCC</sub>-value is necessary. Like with the CT-specimens, the opening of the test piece is usually kept constant with the help of a wedge, although constant loading is also possible. For reasons of symmetry, however, the starter crack required in the test should, even in aluminium specimens, not be generated with the help of the wedge. Various equations are given in the specifications and guidelines for crack propagation calculation.

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