Designation: G111 – 97 (Reapproved 2013)

Standard Guide for
Corrosion Tests in High Temperature or High Pressure Environment, or Both[1]

This standard is issued under the fixed designation G111; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers procedures, specimens, and equipment for conducting laboratory corrosion tests on metallic materials under conditions of high pressure (HP) or the combination of high temperature and high pressure (HTHP). See 3.2 for definitions of high pressure and temperature.

1.2 Tests conducted under HP or HTHP by their nature have special requirements. This guide establishes the basic considerations that are necessary when these conditions must be incorporated into laboratory corrosion tests.

1.3 The procedures and methods in this guide are applicable for conducting mass loss corrosion, localized corrosion, and electrochemical tests as well as for use in environmentally induced cracking tests that need to be conducted under HP or HTHP conditions.

1.4 The primary purpose for this guide is to promote consistency of corrosion test results. Furthermore, this guide will aid in the comparison of corrosion data between laboratories or testing organizations that utilize different equipment.

1.5 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E8 Test Methods for Tension Testing of Metallic Materials
G1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens
G3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing
G5 Reference Test Method for Making Potentiodynamic Anodic Polarization Measurements
G15 Terminology Relating to Corrosion and Corrosion Testing (Withdrawn 2010)[3]
G30 Practice for Making and Using U-Bend Stress-Corrosion Test Specimens
G31 Guide for Laboratory Immersion Corrosion Testing of Metals
G34 Test Method for Exfoliation Corrosion Susceptibility in 2XXX and 7XXX Series Aluminum Alloys (EXCO Test)
G38 Practice for Making and Using C-Ring Stress-Corrosion Test Specimens
G39 Practice for Preparation and Use of Bent-Beam Stress-Corrosion Test Specimens
G46 Guide for Examination and Evaluation of Pitting Corrosion
G49 Practice for Preparation and Use of Direct Tension Stress-Corrosion Test Specimens
G59 Test Method for Conducting Potentiodynamic Polarization Resistance Measurements
G78 Guide for Crevice Corrosion Testing of Iron-Base and Nickel-Base Stainless Alloys in Seawater and Other Chloride-Containing Aqueous Environments
G106 Practice for Verification of Algorithm and Equipment for Electrochemical Impedance Measurements

3. Terminology

3.1 *Definitions*—The definitions of terms given in Terminology G15 shall be considered as applying to this guide.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *high pressure*—a pressure above ambient atmospheric pressure that cannot be contained in normal laboratory glassware. Typically, this is greater than 0.07 MPa (10 psig).

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[1] This guide is under the jurisdiction of ASTM Committee G01 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

Current edition approved May 1, 2013. Published July 2013. Originally approved in 1992. Last previous edition approved in 2006 as G111–97 (2006). DOI: 10.1520/G0111-97R13.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

3.2.2 *high temperature*—temperatures above ambient laboratory temperature where sustained heating of the environment is required.

4. Summary of Guide

4.1 This guide describes the use of corrosion coupons, stressed SCC specimens, and electrochemical electrodes in HP and HTHP environments. It also includes guidelines for the use of high pressure test cells with these specimens to conduct reproducible, accurate corrosion test data.

4.2 Typically, HP and HPHT tests involve exposure of test specimens to a liquid (aqueous or non-aqueous), gaseous or multiphase environment, or both, in an appropriate test cell. The test cell must be able to resist corrosion and environmental cracking in the test environment while containing the pressurized, heated environment. Furthermore, the test specimens in the HP or HPHT test, or both, can be exposed in either stressed or unstressed condition in either the free corroding state or under electrochemical polarization.

5. Significance and Use

5.1 HP and HTHP corrosion tests are commonly used to evaluate the corrosion performance of metallic materials under conditions that attempt to simulate service conditions that involve HP or HTHP in combination with service environments. Examples of service environments where HP and HTHP corrosion tests have been utilized include chemical processing, petroleum production and refining, food processing, pressurized cooling water, electric power systems and aerospace propulsion.

5.2 For the applications of corrosion testing listed in 5.1, the service involves handling corrosive and potentially hazardous media under conditions of high pressure or high temperature, or both. The temperature and pressure usually enter directly into the severity of the corrosion process. Consequently, the laboratory evaluation of corrosion severity cannot be performed in conventional low pressure glassware without making potentially invalid assumptions as to the potential effects of high temperature and pressure on corrosion severity.

5.3 Therefore, there is a substantial need to provide standardized methods by which corrosion testing can be performed under HP and HTHP. In many cases, however, the standards used for exposure of specimens in conventional low pressure glassware experiments cannot be followed due to the limitations of access, volume and visibility arising from the construction of high pressure test cells. This guide refers to existing corrosion standards and practices as applicable and then goes further in areas where specific guidelines for performing HP and HTHP corrosion testing are needed.

6. Apparatus

6.1 The test cell shall be constructed to applicable standards and codes so that it will have an adequate pressure rating to safely handle the test pressure.

6.2 The test cell shall be made of materials that are corrosion resistant and effectively non-reactive with the test environment.

6.2.1 The term effectively non-reactive shall mean that the test cell shall be free of significant mass loss or localized corrosion, SCC or other embrittlement phenomena in the test environment, not contaminate the test environment with corrosion or other reaction products, and not consume or absorb reactive chemical species from the test environment.

6.3 The test cell shall have a seal mechanism that can withstand both the pressures, temperatures, and corrosive environment to be used in the test. Periodic hydrostatic testing of the test cell is recommended to ensure pressure capabilities.

6.4 The test cell shall be designed to have the necessary inlet and outlet ports to allow the test environment to be established in a controllable manner, monitored and sampled during the exposure period, released in a controlled manner at the completion of the test, and if over temperature or pressure conditions may occur, adequate over pressure release and over temperature control equipment should be utilized.

6.5 In cases where external loading fixtures are used for stressing specimens in the HP and HTHP test environment, specially designed feed-throughs shall be used which provide for a minimum of friction force.

6.6 Test cell feed-throughs required for external stressing may be designed to balance the internal pressure in the test vessel.

6.7 Any frictional or pressure forces (or thermal expansion) acting on the specimen through the stressing fixtures must be taken into account when determining the actual load on the specimen.

6.8 Stressing and electrode feed-throughs shall be designed so that the electrodes or stressing rods and specimens cannot be ejected from the test cell under pressure. Furthermore, they shall provide for electrical isolation of the specimen from the test cell unless galvanic coupling is specifically desired.

6.9 Gripping devices shall be designed such that they are in compliance with Test Method E8 where application of load to the specimen is required.

7. Reagents

7.1 In corrosion testing, providing a reproducible chemical environment in which to expose the corrosion test specimens is necessary.

7.2 In cases where the test environment is established by the mixing of chemicals in the laboratory, chemicals of reagent grade purity with known contaminant levels are recommended. Simulations of service environments can be formulated in which laboratory corrosion tests can be conducted.

7.3 In HP/HTHP corrosion testing, a common practice is to conduct tests in environments that have been sampled and retrieved from field or plant locations. In both cases described in 7.2 and 7.3, detailed information as to the chemical composition of the environment should be obtained. Particular attention should be given to the levels of impurities and contaminants that may be in the environment. Furthermore, under some conditions, these environments may be prone to changes after sampling or during testing which can affect the corrosion test results.

7.4 In many cases, the test cells used to conduct HP tests are limited in volume and may not be designed to accommodate replenishment of the environment. Therefore, monitoring the chemical composition of the environment during the exposure may be necessary to identify if depletion of reactive constituents or concentration of constituents has occurred. In some cases, replenishment or changing of the test environment may be necessary so that a valid corrosion test can be conducted.

7.5 In all cases, it is recommended that the test environment be fully documented with respect to its chemical composition.

8. Test Specimens

8.1 *Preparation of Specimens:*

8.1.1 The primary objective is to prepare a reproducible metallic surface with an absolute minimum of coldworking followed by cleaning and degreasing.

8.1.2 Since test cells for HP and HTHP tests are usually of metallic construction, care must be taken to electrically isolate the specimens from the test cell unless galvanic coupling is specifically desired in the test. In cases where the test cell is used as a member of a galvanic couple, care must be taken to ensure that the galvanic action (anodic or cathodic) does not degrade the integrity of the test cell.

8.2 *Corrosion Specimens:*

8.2.1 Prepare specimens used in HP or HTHP corrosion tests in accordance with Practices G1 and G31. Commonly, test cells used for HP and HTHP exposure tests are restricted in volume. The available volume in the test cell often decreases with increasing pressure rating. Therefore, it is frequently necessary to restrict the size and surface area of corrosion coupons used in HP and HTHP corrosion tests.

8.2.2 The ratio of solution volume-to-specimen surface area is important and a minimum ratio of 30 mL/cm^2 should be maintained, where possible. If the ratio drops below this level, it should be shown that there will not be an unacceptably high depletion rate of important environmental constituents, or there will not be an undesirable amount of metal ion impurities added into the test environment during the period of exposure. In all cases, the solution volume-to-specimen surfaces area used in the test should be stated. If the test cell, specimen holders or stressing fixtures can contribute to the conditions stated above then they should be included in the calculation of specimen surface area.

8.3 *Stressed Corrosion Specimens:*

8.3.1 Both self stressed and externally stressed specimens are acceptable for testing at HP and HTHP. Methods for the fabrication and use of appropriate stressed specimens are given in the referenced documents. These include tension, bent beam, C-ring, and U-bend specimens in accordance with Practices G49, G39, G38, and G30, respectively. Fracture mechanics specimens can also be accommodated.

8.3.2 For similar reasons given in 8.2, when testing multiple specimens, it is recommended that the size of the specimens be restricted to the smallest applicable specimen provided for under the appropriate standards.

8.3.3 Due to the limited access of the specimens in HP and HTHP tests, self stressed specimens are usually more convenient than specimens that require external stressing fixtures.

8.3.4 In cases such as direct tension and fracture mechanics tests, use of external loading frames and fixtures in conjunction with HP and HTHP corrosion tests may be desirable. In these cases, take both the frictional (sealing) forces and pressure forces acting on the specimens into account when determining the effect of applied stress.

8.4 *Electrochemical Electrodes:*

8.4.1 Prepare electrodes for use in HP and HTHP corrosion studies as described in Practice G3, Test Method G5, and Practices G59 and G106.

8.4.2 Cylindrical electrode specimens where only the lower portion of the electrode is exposed to the liquid phase of test environment and where the electrical connections are made externally to the test cell are a convenient geometry. Care must be taken to electrically isolate the electrodes from the test cell. Other electrode geometries and designs may be used that facilitate feed-through and electrical isolation.

8.4.3 A critical portion of the HP or HTHP electrochemical system is the design and construction of the reference electrode. It is common to use external reference cells that use stable reference systems such as Ag/AgCl or other stable electrochemical reference system that can be enclosed in a separate pressure containing compartment. This cell is then connected to the test cell via a salt bridge and is pressure balanced with the test cell to minimize ingress of contaminants into either the test cell or the reference electrode. Alternatively, an inert or corroding metal electrode can be used as a pseudo-reference electrode in some cases. Examples of such pseudo-reference electrodes include platinum, graphite, or other metal with known stable corrosion potential. However, one problem that can occur with this technique is a drift in reference potential with time. Care should be taken when employing such methods. These pseudo-reference electrodes can effectively give a measure of relative potential even if the absolute potential is not known.

9. Test Environment

9.1 Choose the test environment to either simulate the most accurate representation of the service environment possible under the constraints of the equipment available or provide for a simple screening environment. In the case of service environment simulation, accurate monitoring for depletion and concentration of chemical species in the test cell is required so that the environment can be controlled within a specified range of composition. In the case of simple screening environments, allowance for greater latitude in the variance of the test environment from service conditions is acceptable. In this case, simple solutions are commonly utilized and chemical monitoring is not conducted.

9.2 *Test Temperature:*

9.2.1 The test temperature should be controlled to within ±1.0 % of the specified temperature or ±2.5°C, whichever is greater, unless otherwise specified.

9.2.2 Temperature of the liquid phase can be measured in one location if the specimens are totally contained therein. However, for large test cells substantial temperature gradients can exist and care should be taken to monitor the temperature close to the specimens using thermocouples contained in corrosion resistant sheaths.

9.2.3 In tests where the specimens are exposed to the gaseous or vapor phase, care must be taken to obtain direct measurements of specimen temperature. When the test vessel is heated externally, the vessel temperature may greatly exceed the specimen temperature. Internally heating the specimen may be possible in the gaseous environment. Such a procedure is particularly useful when conditions of heat transfer are being simulated.

9.3 *Pressure:*

9.3.1 The pressure should be monitored continuously or periodically during the exposure period using either a pressure gage or pressure transducer. Care shall be taken to properly select materials of construction for these measurement devices if exposed directly to the test environment. Methods to minimize corrosion of pressure monitoring equipment are to provide for an isolation valve between the monitoring equipment or to utilize a diaphragm seal that transmits the pressure from the test cell to the monitoring equipment via a chemically inert media.

9.4 *Liquid Constituent(s):*

9.4.1 In single phase liquid environments, the solution is often static with only convection to provide agitation. However, it can be stirred or mixed, particularly if decomposition or separation of phases may occur, or if effects of velocity are desired.

9.4.2 Special magnetic or mechanical stirring attachments are available for use in pressurized systems. Care shall be taken to ensure that the components of these stirring attachments are inert to the test environment and that they will not contaminate the test environment particularly from wear of bearing materials.

9.4.3 In multiphase liquid systems, HP and HTHP tests can be conducted in either static or agitated conditions. The agitation can be accomplished by either stirring as mentioned in 9.4.2 or by rotating the test cell to provide for mixing of the constituents.

9.5 *Gaseous Constituent(s):*

9.5.1 Gases can be used separately as a test environment or in combination with liquid environments to provide aeration, deaeration, saturation of soluble gases, and pressurization.

9.5.2 Care should be taken to add the correct amount of gas at room temperature so that the desired pressure is attained at the test temperature. An estimate of this starting pressure can be made taking into account the reduction in vapor space from expansion of the liquid, the PVT characteristics of the gas, the vapor pressure of the liquid, and the solubility of the gas in the liquid constituents.

9.5.3 In some cases, it is necessary to add liquefied gas constituents at room temperature that then convert to a pressurized gas upon heating, to elevated temperature. In this case, a quantity of the liquefied gas is weighed that will convert to the desired pressure at the test temperature.

9.5.4 If deaerated conditions are required, care should be taken to adequately remove air from the test vessel and the liquid constituents. This usually requires cyclic vacuum and inert gas purging of the test vessel. Inert gas purging of the liquid phase is also commonly utilized along with a vacuum cycle only if the vapor pressure of the liquid at room temperature is low (that is, ≤1 psia).

9.5.5 Where reactive constituents are present in the test gas, it may be necessary to allow for sufficient vapor space to act as a reservoir of the reactive species or replenish the gas in the test cell on either a periodic or continuous basis. If this condition is suspected, detailed chemical analysis of the test environment versus time may be required. This procedure will allow for the frequency of replenishment to be determined. Control of the environment to ±10 % of the intended level of constituent is usually adequate for conventional HP and HTHP tests.

10. Procedure

10.1 Clean, weigh, and assemble the test specimens on suitable fixtures with electrical isolation. Care should be taken to secure the specimens and position them in the test cell. They can often experience movements and vibrations during the sealing of the test cell.

10.2 Electrical isolation is usually obtained using ceramic or TFE washers depending on the intended test temperature. TFE washers have a temperature limit of approximately 280°C. The performance of ceramics can be pH dependent except for ZrO_2.

10.3 Place the specimens in the test cell.

10.4 If environment deaeration is not required, the liquids can be added either before or after the specimens.

10.5 If deaeration is required, seal the vessel following placement of the specimens inside. Then vacuum the test cell and purge inert gas to the degree required. Add the constituents (that have been pre-deaerated by a similar technique) in a closed system excluding air.

10.6 Leak test the test cell either with inert or test gas to the intended test pressure at room temperature.

10.7 Heat the test cell to the desired test temperature and adjust the pressure to the desired level.

10.8 Run the test for a specified period. Typical durations may be as short as several hours or may last, over 10 000 hours depending on the intended use of the data. Screening tests commonly run for shorter durations (1 to 30 days) while service simulations usually last for many months.

10.9 As a minimum, check the composition of the test environment (liquid or gas, or both) in the test vessel at the start and completion of the test. This will help to determine the extent of changes in the environment and possible contamination or depletion of the test environment. The test cell may be configured to have ports that allow for venting of gas or liquid, or both, from the vessel under pressure for the purpose of analysis. Take care in taking such samples as it requires handling chemical environments at high pressures and temperatures.

10.10 Upon completion of the test exposure, vent the test cell to release the pressure. Where possible this venting should be conducted after the vessel has cooled to below the boiling point of any liquid phases and also less than the flash point of any potentially flammable chemicals. Furthermore, the venting process should be conducted in a slow, controlled manner unless the aim of the test is to achieve rapid decompression and evaluate its effects on the test specimens. All venting should be conducted in a manner whereby any reactive or hazardous constituents can be scrubbed or contained for proper disposal.

11. Evaluation of Specimens

11.1 Specimens exposed in HP and HTHP test should be evaluated for corrosion in a similar manner as specimens exposed to conventional corrosion tests in glassware. It is recommended that Practice G1 be consulted for details on specimen cleaning and the procedures in Guide G46, Test Method G34, and Guide G78 be used for evaluation of localized corrosion.

12. Report

12.1 Report the following information for all HP and HTHP corrosion tests:

12.1.1 *Materials Characterization*—Including composition, mechanical properties, product form, heat treatment, section size, and sampling procedures.

12.1.2 *Specimen Characterization*—Including location, orientation, type, size, configuration, number of specimens, surface preparation, and specimen surface area to environment volume ratio.

12.1.3 *Test Characterization*—Including test type, duration, stress level, strain rate, galvanic coupling, impressed current, electrochemical potential or scan rate, description of equipment (test vessel dimension and volume, materials of construction, location of ports and feed-throughs), and solution volume.

12.1.4 *Environment Characterization*—Including analysis of environment (start, interim, completion, as necessary), liquid constituent(s), gaseous constituent(s), aeration/deaeration/replenishment procedure, pH, temperature, agitation—method and description, and pressure.

12.1.5 *Test Results*—Including mass loss, pitting, crevice attack, SCC "failure/no failure," crack growth rate, linear polarization (polarization resistance), polarization curve, critical pitting/crevice temperature, and electrochemical impedance.

13. Keywords

13.1 corrosion tests; high pressure; high temperature