



Standard Test Method for Determining the Crevice Repassivation Potential of Corrosion-Resistant Alloys Using a Potentiodynamic- Galvanostatic-Potentiostatic Technique¹

This standard is issued under the fixed designation G 192; 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 test method covers a procedure for conducting anodic polarization studies to determine the crevice repassivation potential for corrosion-resistant alloys. The concept of the repassivation potential is similar to that of the protection potential given in Reference Test Method **G 5**.

1.2 The test method consists in applying successively potentiodynamic, galvanostatic, and potentiostatic treatments for the initial formation and afterward repassivation of crevice corrosion.

1.3 This test method is a complement to Test Method **G 61**.

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

1.5 *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

B 575 Specification for Low-Carbon Nickel-Chromium-Molybdenum, Low-Carbon Nickel-Chromium-Molybdenum-Copper, Low-Carbon Nickel-Chromium-Molybdenum-Tantalum, and Low-Carbon Nickel-Chromium-Molybdenum-Tungsten Alloy Plate, Sheet, and Strip

D 1193 Specification for Reagent Water

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

G 1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens

G 5 Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements

G 15 Terminology Relating to Corrosion and Corrosion Testing

G 48 Test Methods for Pitting and Crevice Corrosion Resistance of Stainless Steels and Related Alloys by Use of Ferric Chloride Solution

G 61 Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion Susceptibility of Iron-, Nickel-, or Cobalt-Based Alloys

G 78 Guide for Crevice Corrosion Testing of Iron-Base and Nickel-Base Stainless Alloys in Seawater and Other Chloride-Containing Aqueous Environments

3. Terminology

3.1 *Definitions*—For definitions of corrosion-related terms used in this test method, see Terminology **G 15**.

4. Summary of Test Method

4.1 This anodic polarization test method combines techniques such as potentiodynamic, galvanostatic, and potentiostatic polarization methods. This test method is called the Tsujikawa-Hisamatsu Electrochemical (THE) test method to honor the two precursors of this technique (see **1** and **2**).³ The new technique will be called the THE test method. This new THE test method is more time-consuming than the already well-established cyclic potentiodynamic polarization (CPP) described in Test Method **G 61**.

4.2 The THE test method can be used with any corrosion-resistant alloy, but it was developed by studying Alloy 22 (UNS N06022). The composition and other properties of Alloy 22 are given in Specification **B 575**. Alloy 22 is a nickel-based alloy containing approximately 22wt% Cr, 13wt% Mo, 3wt% W and 3wt% Fe. The THE test method is a complement to the cyclic potentiodynamic polarization (CPP) described in Test Method **G 61**. CPP may be used as a first fast screening method and THE test method for fine-tuning the repassivation potential for crevice corrosion when the environment is not highly

¹ This test method is under the jurisdiction of ASTM Committee G01 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.11 on Electrochemical Measurements in Corrosion Testing.

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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 boldface numbers in parentheses refer to a list of references at the end of this standard.

aggressive (3-6). The THE test method has also been applied to other highly corrosion-resistant alloys, such as Titanium grade 7 (Ref 7).

4.3 The THE test method can be used with any electrolyte solution. A standard 1 M NaCl solution at 90°C or lower temperature may be used to compare alloys of interest. The round robin described in Section 15 was carried out in 1 M NaCl solution at 90°C.

4.4 The test involves in polarizing the test electrode in three steps:

4.4.1 *Step 1*—The test electrode is polarized potentiodynamically at a rate of 0.168 mV/s (as in Test Method G 61) starting at or slightly below the corrosion potential until a preset current (or current density) is reached (for example, 2 $\mu\text{A}/\text{cm}^2$). After this initial potentiodynamic polarization, the polarization control is changed to galvanostatic mode (Step 2).

4.4.2 *Step 2*—The preset current of 2 $\mu\text{A}/\text{cm}^2$ is kept constant for a 2-h period to develop and grow a crevice corroded area (if any develops). During the galvanostatic Step 2, the potential output is monitored.

4.4.3 *Step 3*—The polarization control is shifted to the potentiostatic mode. The potential at the end of the galvanostatic hold (Step 2) is read, and then 10 mV are subtracted. The resulting value of potential is applied for a 2-h period while the current output is monitored. Then successive potentiostatic treatments are applied, each time at 10 mV lower than the previous treatment. A total of 10-15 potentiostatic treatments are usually required to finish Step 3.

4.5 The crevice repassivation potential (ER,CREV) is the highest potential in Step 3 for which current density does not increase as a function of time. It is understood that at a potential below ER,CREV the alloy will not develop crevice corrosion under the tested conditions.

5. Significance and Use

5.1 The THE test method is designed to provide highly reproducible crevice repassivation potentials for corrosion-resistant alloys (for example, Alloy 22) in a wide range of environments from non-aggressive to highly aggressive. In conditions of low environmental aggressiveness (such as low temperature or low chloride concentration), corrosion-resistant alloys such as Alloy 22 will resist crevice corrosion initiation and the cyclic potentiodynamic polarization test (Test Method G 61) may fail to promote crevice corrosion mainly because it drives the alloy into transpassive dissolution instead of nucleating crevice corrosion. The THE test method provides a more controlled way of applying the electrical charge to the test electrode, which may induce crevice corrosion without moving it into transpassive potentials.

5.2 The more noble this crevice corrosion repassivation potential (ER,CREV) value, the more resistant the alloy is to crevice corrosion in the tested electrolyte. This is similar to other test methods to measure localized corrosion resistance such as Test Method G 61 and Test Methods G 48. The results from this test method are not intended to correlate in a quantitative manner with the rate of propagation that one might observe in service when localized corrosion occurs.

5.3 This test method may be used to rank several alloys by using the same testing electrolyte and temperature. It can also

be used to determine the response of a given alloy when the environmental conditions (such as electrolyte composition and temperature) change.

6. Apparatus

6.1 *Cell*—The polarization cell should be similar to the one described in Reference Test Method G 5 and Test Method G 61. Other polarization cells may be equally suitable. The cell should have a capacity of about 1 L and should have suitable necks or seals to permit the introduction of electrodes, gas inlet and outlet tubes, and a thermometer or thermocouple. The Luggin probe-salt bridge separates the bulk solution from the saturated calomel or saturated silver chloride reference electrode.

6.2 *Test Electrode (Specimen) Holder*—The test electrode holder and the mounting rod should be similar to the one described in Figure 5 in Reference Test Method G 5 (reproduced in Fig. 1). A leakproof PTFE compression gasket, as described in subsection 4.6.1 in Reference Test Method G 5, is also necessary.

6.3 *Potentiostat and Output Potential and Current Measuring Instruments*—The potentiostat and other instruments should be similar to the ones specified in Test Method G 61. Most commercial potentiostat and related instruments meet the specific requirements for these types of measurements.

6.4 *Electrodes*—The standard recommended working or testing electrode is shown in Fig. 1, which is a prismatic measuring 0.75 by 0.75 by 0.375-in. thick (approximately 20 by 20 by 10 mm). It has a drilled and tapped hole on top for the connecting rod (as in Reference Test Method G 5). The electrodes also have a 7-mm diameter hole in the center for mounting two crevice formers, one at each side using a bolt. The test electrode could be cut from any plate or extruded bar. It is recommended that the creviced faces of the test electrode correspond to the rolling or extruded direction. In certain tested conditions the test electrode may show end grain attack in the short transverse direction, but generally the crevice former provides a more active path for corrosion than the freely exposed surfaces.

6.5 *Crevice Former or Crevice Washer*—The crevice former is a multiple crevice assembly (MCA), and it is described in subsection 5.4 of Test Methods G 48, in subsection 9.2.2 in Guide G 78, and in Ref 8. This MCA crevice former should be fabricated using a hard non-conductive ceramic material such as alumina or mullite (Fig. 2). Before mounting on the test electrode (specimen), the crevice washers should be covered with a PTFE tape. This tape is 1.5-in. wide and 0.003-in. thick (standard military grade MIL-T-27730A). A corrosion-resistant fastener is used to secure the two MCA washers, one on each side of the test electrode. Crevice formers made of solid PTFE such as in Test Methods G 48 or Guide G 78 are not as effective, since they do not form a crevice gap tight enough for certain high end corrosion-resistant materials. This may result in higher and poorly reproducible repassivation potential values. Two standard metal washers are used as well (Figs. 1 and 2). The standard pressure on the MCA crevice formers may vary (depending of the study underway) but a minimum of 30-in.·lb (3.4-N·m) torque may be needed to form a tight crevice. Use a calibrated torque wrench to apply the

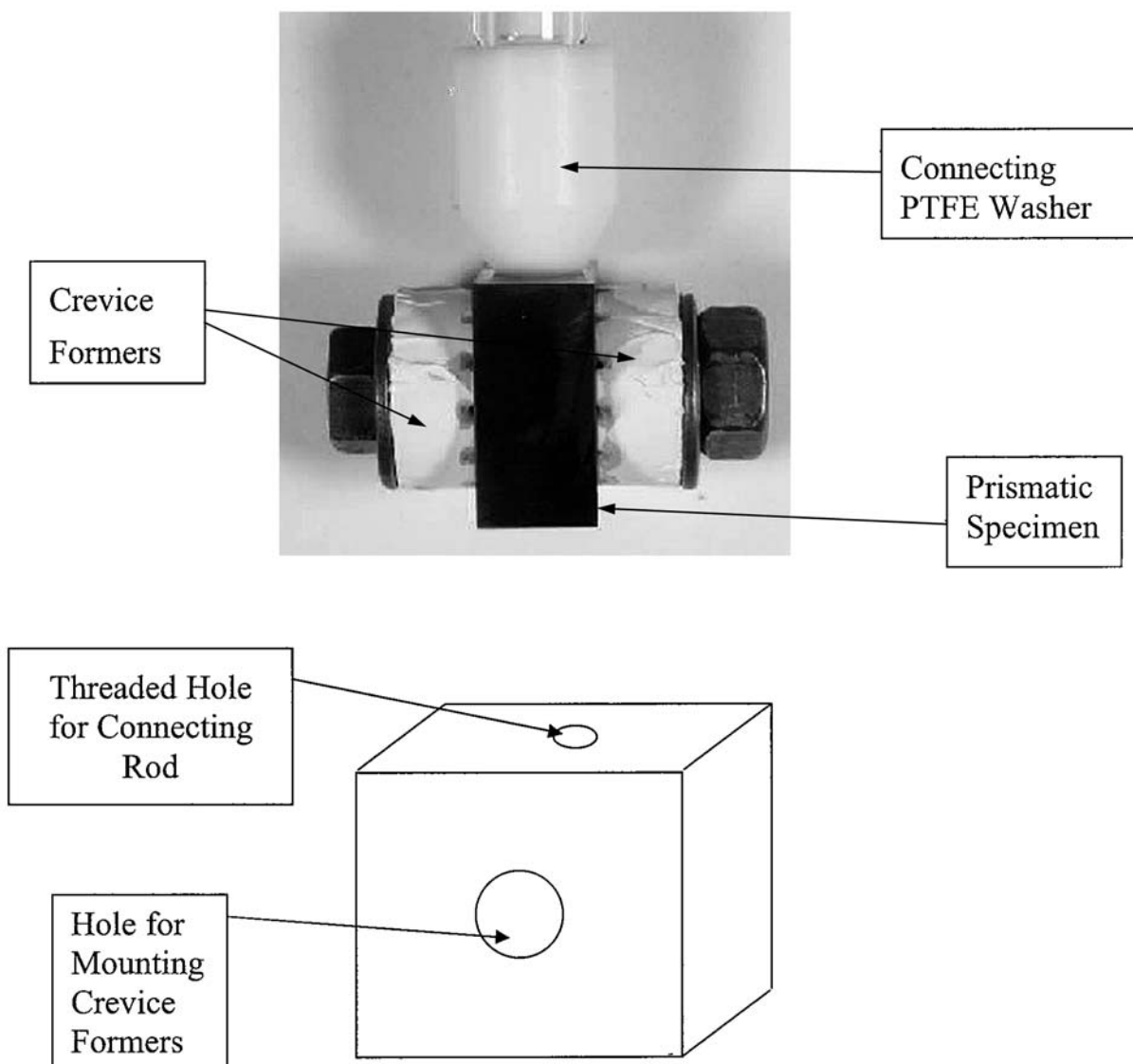


FIG. 1 Prismatic Test Electrode (0.75 by 0.75 by 0.375 in. or approximately 20 by 20 by 10 mm)

torque. Electrical contact between the bolt and the test electrode should be avoided. Effective insulation may be provided by the use of nonmetallic sleeves or by wrapping the assembly bolt with PTFE tape.

6.6 Counter Electrode—The counter electrodes may be prepared as in Reference Test Method **G 5** or may be prepared from high-purity platinum flat stock and wire. Counter electrodes could be easily fabricated by spot welding platinum wire to a platinum foil, which could be curved to adapt to the cell geometry. It is recommended that the area of the platinum counter electrode be twice as large as the one of the working electrode (test electrode or specimen).

6.7 Reference Electrode—Reference electrodes could be commercially available saturated calomel or silver-silver chloride. These electrodes are durable and reliable; however they should be maintained in the proper conditions. The potential of

the reference electrodes should be checked at periodic intervals to ensure their accuracy.

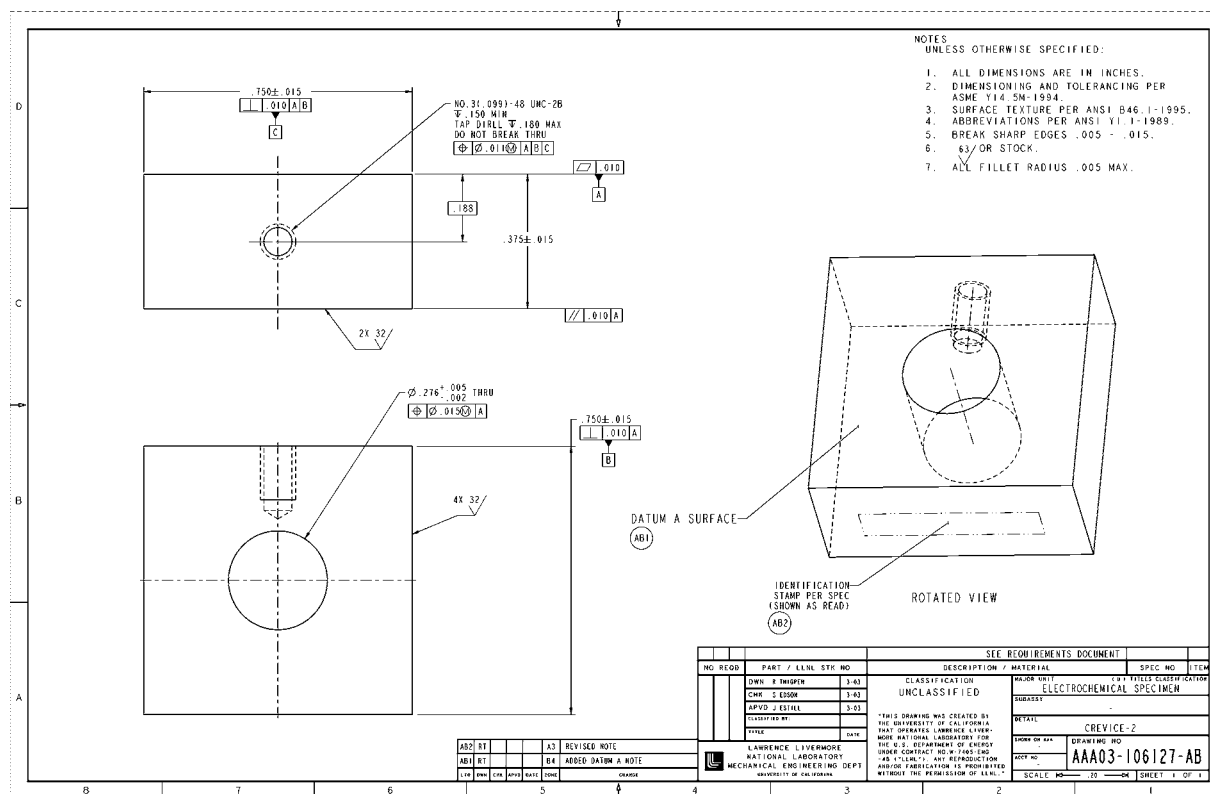
7. Reagents and Materials

7.1 Purity of Reagents—Reagent grade chemicals should be used in all tests.

7.2 Purity of Water—The water should be distilled or deionized conforming to the requirements of Specification **D 1193**, Type IV reagent water.

7.3 Sodium Chloride (NaCl)—To prepare 1 L of 1 M NaCl solution, dissolve 58.45 g of NaCl in purified water to obtain a total volume of solution of 1 L.

7.4 Purging Gas—If deaeration is necessary, nitrogen gas of a minimum 99.99 purity should be used. Tests could also be run under normal aeration conditions or under any other atmosphere.



7.5 *Prismatic-Shaped Test Electrodes of the Corrosion-Resistant Alloy*—Other type of creviced test electrodes may also be used, depending on the specific study being performed.

8. Hazards

8.1 Normal precautions for handling hot liquids should be observed.

8.2 Personal protective equipment for handling hot liquids should be used.

9. Sampling, Test Electrodes, and Test Units

9.1 Recommended test electrodes (specimens) are prismatic-shaped as shown in Fig. 1. The thickness of the material for the test electrodes is not essential, but it should be enough to handle the mounting rod mechanism. Thicker materials are easier to prepare (polish). A fresh (or 1 h prior to testing) finish wet grinding of 600 grit silicon carbide paper is recommended. If surface effects are being studied, other surface finishing may be considered.

9.2 If other than mill finishes are investigated, the test electrodes may be reused after remachining or grinding to remove all traces of previously incurred attack. The importance of maintaining parallel/prismatic surfaces cannot be overstated with regard to reproducing crevice conditions and the prevention of possible fracture of the ceramic devices.

9.3 The test electrodes could be prepared using wrought or cast material, or machined weld metal.

9.4 The bolt, nut, and flat washer must be made of a corrosion-resistant material. It is recommended to use Ti Gr 2 (UNS R52400). Fastening devices can also be fabricated using

other readily available materials such as Alloys C-276 and 625 (UNS N10276 and N06625, respectively). The crevice former is manufactured using a ceramic material according to the 12-tooth design in Test Methods G 48, Guide G 78, and Ref 8 (Fig. 2). The ceramic washer is covered by a wide PTFE tape 1.5-in. wide and 0.003-in. thick (standard military grade MIL-T-27730A).

10. Preparation of Apparatus

10.1 The testing cell and test electrode holder are described in Reference Test Method G 5.

10.2 The potentiostat and other instruments should be similar to the ones specified in Test Method G 61. Most commercial potentiostat and related instruments meet the specific requirements for these types of measurements.

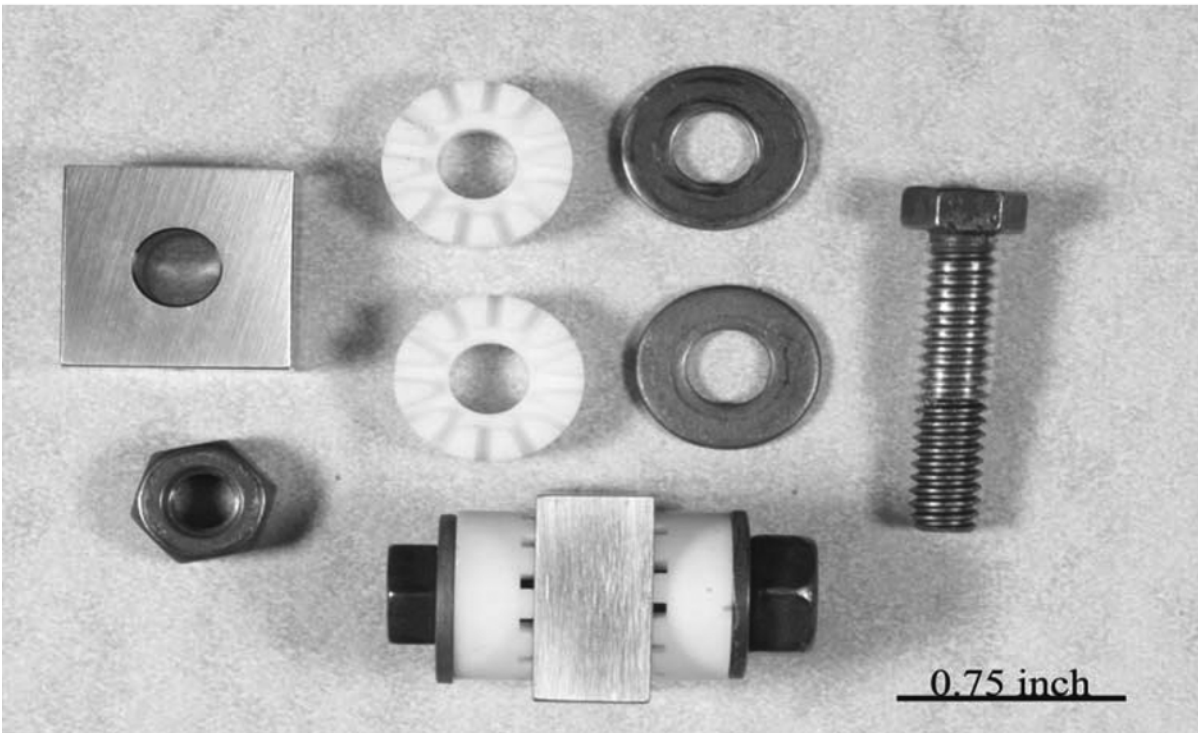
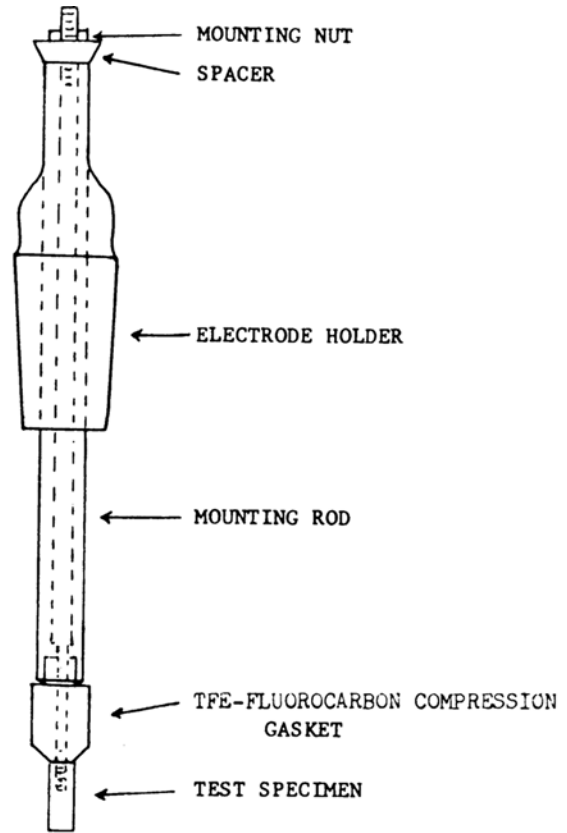
11. Calibration and Standardization

11.1 The potentiostat and its software should be calibrated in accordance with user calibration procedures. The good operating conditions of the potentiostat can also be assessed using the Reference Test Method G 5 procedure.

12. Procedure

12.1 Test electrode preparation, cleaning and mounting. Practice G 1 is to be followed where applicable, unless otherwise stated in this procedure.

12.2 Wet grind the test electrode to a surface finish of 600 grit silicon carbide paper. (Other variations may be used if the studied variable, for example, is surface finish.)



NOTE—Includes Fig. 5 from Reference Test Method G 5 to describe how the test electrode is attached to the specimen holder.

FIG. 2 Crevice Formers for the Test Electrode

12.3 Surface finishing should be carried out not longer than 1 h prior to testing. The test electrode should be then degreased in alcohol or other solvents and rinsed in deionized water and air-dried. After degreasing, handle the test electrode with clean gloves, soft clean tongs, or equivalent preventive measures to avoid surface contamination.

12.4 Mount the crevice formers on the test electrode using a calibrated wrench with torque indicator. The ceramic crevice formers should be pre-coated with the PTFE tape. Apply the specified torque (for example, 30-in.·lb or 3.4-N·m) to the securing bolts avoiding the crevice former to slide (rotate) across the surface of the test electrode.

12.5 Add 900 mL of the 1 M NaCl solution (or other testing electrolyte) to the cell and if necessary purge for 1 h with nitrogen while the cell is being brought to temperature (for example, 90°C). The purging gas rate is commonly set at 100 cc/min.

12.6 Insert the test electrode into the cell, and connect the leads to the potentiostat.

12.7 Monitor the open circuit potential for 1 h while nitrogen is purged through the electrolyte. (Other monitoring times, for example, 24 h or 1 week, could also be used for other test purposes or electrolytes.)

12.8 After the 1-h nitrogen treatment, apply a potentiodynamic anodic polarization at a scan rate of 0.167 mV/s starting 100 mV below the open-circuit potential and progressing anodically until a pre-specified current (or current density) is reached. The threshold current density is set at 2 $\mu\text{A}/\text{cm}^2$, but other values may be used depending on the type of alloy and aggressiveness of the electrolyte.

12.9 Switch the anodic polarization control mode to galvanostatic and apply the end current (for example, 2 $\mu\text{A}/\text{cm}^2$) for 2 h to grow the crevice corroded area. Monitor the output potential values.

12.10 Switch the anodic polarization control mode to potentiostatic mode and apply the end potential in the previous step minus 10 mV. That is, if at the end of step 12.9 the potential was 400 mV, in step 12.10 apply 390 mV and monitor the evolution of the current for 2 h.

12.11 If the current increases with time during the step 12.10, apply another potentiostatic period of 2 h subtracting another 10 mV to the previously applied potential. Following the example in 12.10, in this second step apply 380 mV. This treatment could be preset using commercial software for electrochemical testing.

12.12 Repeat decreasing steps of 10 mV until the output current decreases with time in the 2-h period (Fig. 3).

12.13 The highest potential step for which the current does not increase as a function of time is the Crevice Repassivation Potential, ER,CREV.

12.14 Fig. 3 shows an example of the representation of the current and potential values as a function of the testing time, outlining the potentiodynamic, galvanostatic, and potentiostatic sections of the test.

12.15 After the test is complete, carefully remove the test electrode from the cell, remove the crevice former, and rinse in running water. Let it dry in air.

12.16 Inspect the test electrode for crevice corrosion or other type of localized attack under at least a 20 $\times$ magnification stereomicroscope.

13. Calculation or Interpretation of Results

13.1 The inspection of the test electrode should show if it suffered crevice corrosion or other type of localized attack.

13.2 The value of the Crevice Repassivation Potential (ER,CREV) should be interpreted according to the evidence that the test electrode suffered or did not suffer crevice corrosion according to 13.1. The value of ER,CREV is the average value of all the creviced spots on the tested specimen, for example, if 24 sites corroded, the value of ER,CREV will represent a composed value of the 24 sites.

14. Report

14.1 At a minimum, report the following information:

14.1.1 Alloy tested, common denomination, and UNS number, if available. Also report alloy (metal) composition.

14.1.2 Metallurgical condition (for example, welded, thermally aged, wrought, or non-welded, etc.) and type of surface condition or finish (for example, fresh 600 grit paper or high temperature oxidized).

14.1.3 Torque applied to the test electrodes (see 12.4).

14.1.4 Potentiostat and software used, report also date and procedure of calibration (see 11.1).

14.1.5 Type of reference electrode used and how the reference electrode was checked for accuracy (calibrated).

14.1.6 Testing electrolyte and temperature (for example, deaerated 1 M NaCl at 90°C). Report if the electrolyte was deaerated or not (see 7.4).

14.1.7 Initial pH of the electrolyte.

14.1.8 The value of the applied current in the galvanostatic step and the duration of application (see 12.9).

14.1.9 Attach to the report a graph of potential and current versus time similar to Fig. 4 at the end of this test method.

14.1.10 Report the value of the Crevice Repassivation Potential (ER,CREV) in millivolts (mV) specifying the reference electrode used (see 6.7).

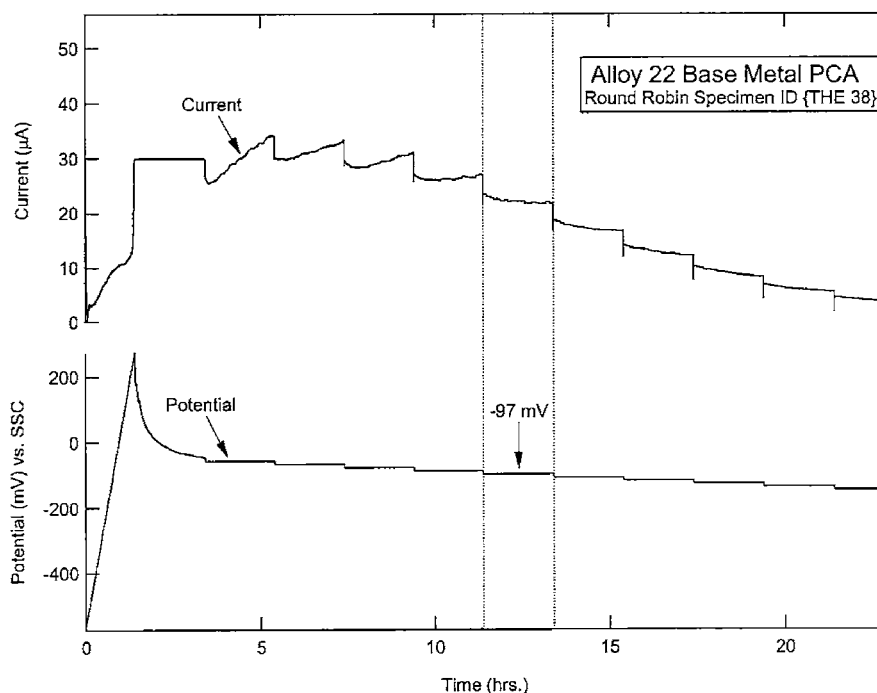
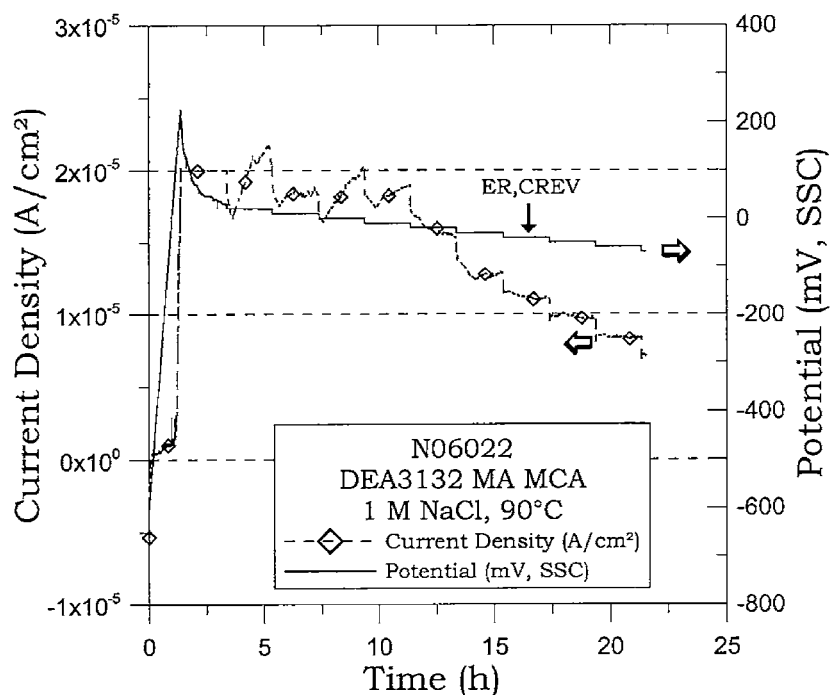
14.1.11 Characteristics of the corroded test electrode (for example, type of attack, if crevice corrosion, how many of the 24 creviced sites were attacked, etc.). It would be useful to include an image of the corroded test electrode.

15. Precision and Bias

15.1 Under some testing conditions results from this method may be comparable to results from Test Method G 61. The current THE test method is meant to be a complement to Test Method G 61 (see also Refs 3-6). The THE test method may produce crevice corrosion in conditions where Test Method G 61 has limitations.

15.2 An interlaboratory study (ILS) test program (round-robin) was completed to determine repeatability of the test results. Ten laboratories initially agreed to participate in this round-robin test to which the testing protocol and test electrodes were sent. At the end, five laboratories returned the results. Results from the round robin are in Ref 6.

15.3 For the round-robin tests, Alloy 22 was exposed in deaerated 1 M NaCl solution at 90°C. The corrosion potential

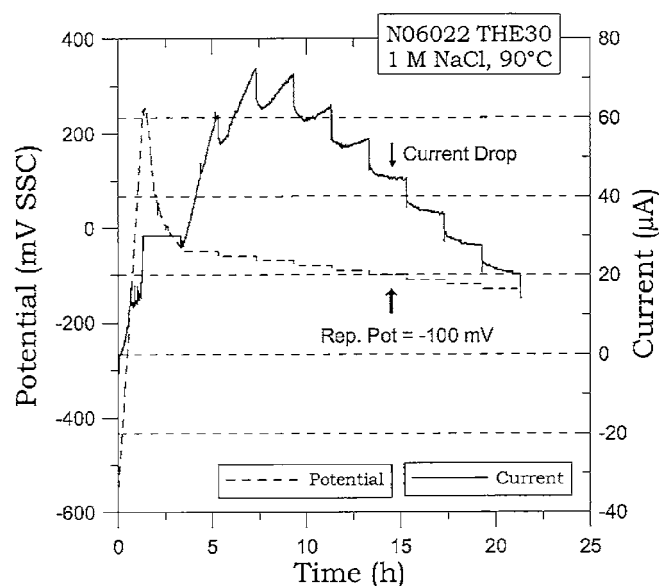


NOTE—ER, CREV is the potential at which the current density decreases as function of time. The top image is from 3.

FIG. 3 Example of a THE Representation

was waited for 1 h. The total applied current in the galvanostatic step was $2 \mu A/cm^2$ ($30 \mu A$ for a $14.06 cm^2$ surface area test electrode). The test electrode was the prismatic shape (Fig. 1) with freshly wet ground surface. The applied torque to the crevice formers was 70 in.-lb (7.9 N-m).

15.4 Prismatic test electrodes were ordered from a single supplier and were distributed five test electrodes to each of the ten different laboratories. Hardware for mounting the test electrodes (Fig. 2) were also supplied to the laboratories. Five laboratories returned the results from the round robin test. Fig.



NOTE—The drop in current at the sixth potentiostatic step clearly shows that the repassivation potential for this test was -100 mV.

FIG. 4 Repassivation Potential for Specimen THE30 from Laboratory 15

4 shows one of the typical results from the tests. This corresponded to Specimen THE30 from Laboratory 15. Fig. 4 shows the repassivation potential for the 6th potentiostatic step for which the current dropped. The repassivation potential for this test was -100 mV SSC.

15.5 The results from all five laboratories are listed in Table 1 and Fig. 5. The overall crevice repassivation potential for Alloy 22 in 1 M NaCl at 90°C from the five laboratories was -107 mV SSC and the standard deviation was 10.0 mV. This low standard deviation shows that the measurement of the repassivation potential using the THE test method is highly reproducible. Fig. 6 shows the corroded test electrode THE51 from Laboratory 6. Crevice corrosion developed under the crevice formers.

15.6 The precision of the procedure in the THE test method was determined in an inter laboratory test program with five laboratories running tests with one lot of Alloy 22. These laboratories ran replicate tests with three to six tests as shown in Table 1. The precision was calculated using a modification

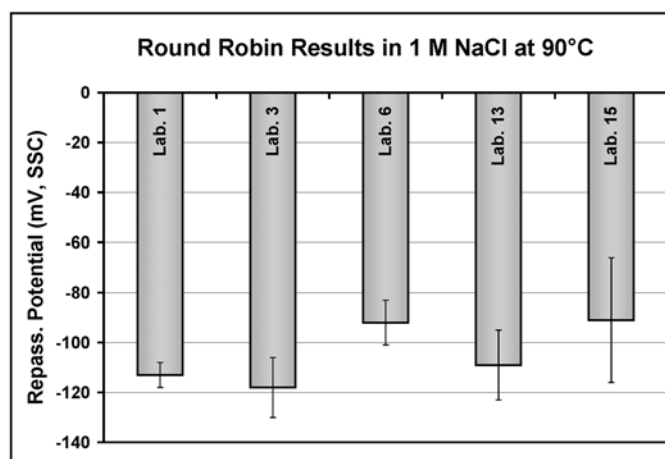


FIG. 5 Repassivation Potential from the Five Laboratories that Reported Values from the Round Robin test

of the procedure shown in Practice E 691 to account for the fact that different numbers of replicates were used.

15.6.1 Precision consists of two components:

15.6.1.1 *Repeatability*—is the agreement that occurs when identical test electrodes are run sequentially with the same test method by the same laboratory and the same operator and equipment.

15.6.1.2 *Reproducibility*—refers to the agreement that occurs when identical test electrodes are run with the same test method but in different laboratories.

15.7 The interlaboratory test program produced repeatability statistics consisting of the repeatability standard deviation s_r and the 95% confidence interval r , where $r = 2.8 s_r$. These values were found to be as shown below for the Alloy 22 test electrodes tested:

$$s_r = \pm 10.2 \text{ mV}$$

$$r = \pm 28.6 \text{ mV}$$

15.8 The interlaboratory program produced reproducibility statistics including the reproducibility standard deviation S_R and the 95% confidence interval for reproducibility, R , where $R = 2.8 S_R$. These values were found to be as shown below for the Alloy 22 test electrodes:

$$S_R = \pm 10.6 \text{ mV}$$

$$R = \pm 29.7 \text{ mV}$$

15.9 The procedure in the THE test method has no bias because the repassivation potential determined by this method is defined only in terms of this method.

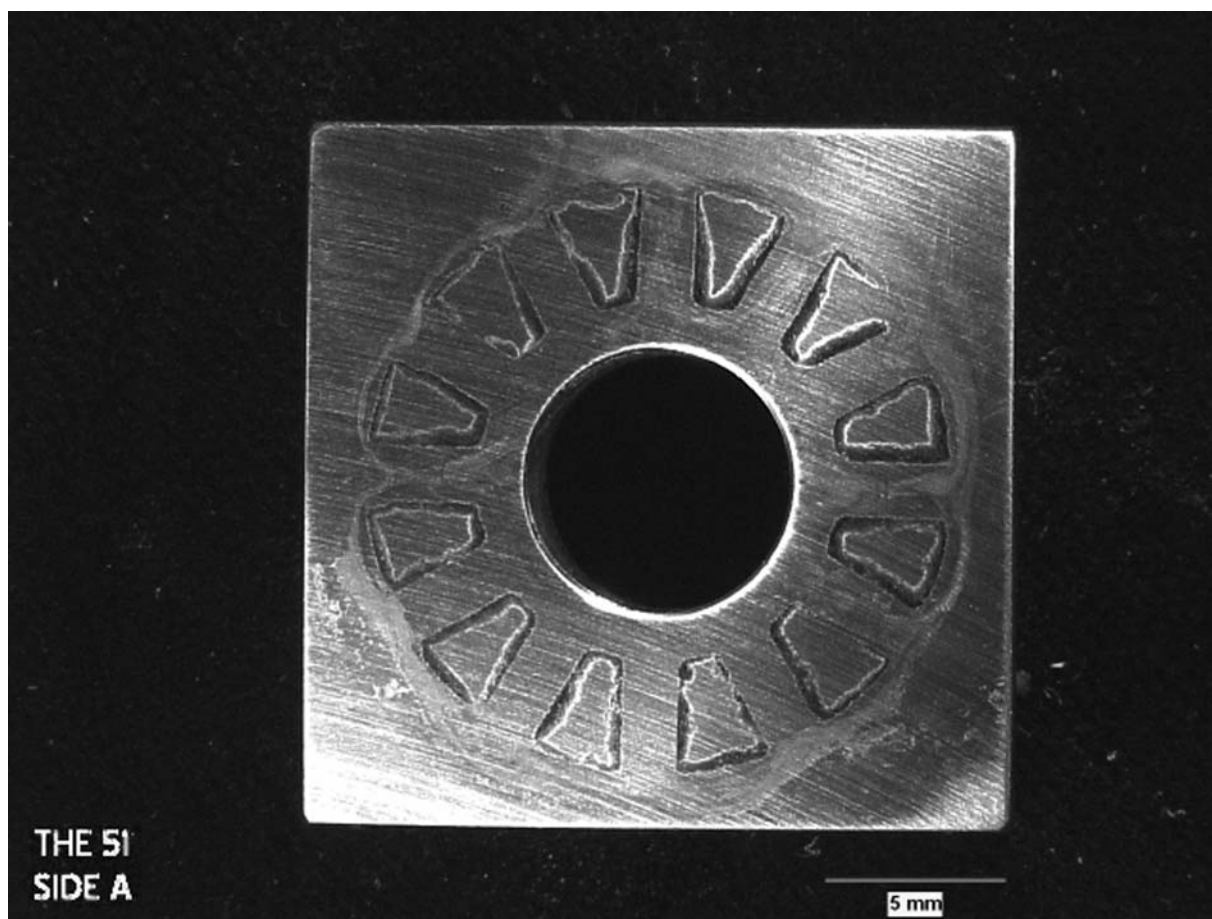
NOTE 1—Test Method G 61 also produces a repassivation potential value. However, the magnitude of the repassivation potential values obtained in Test Method G 61 may vary significantly depending upon the scan rate and other factors. In some cases significantly higher repassivation potentials were found with Test Method G 61 than with the THE test method.

16. Keywords

16.1 corrosion-resistant alloys; crevice repassivation potential; electrochemical test

TABLE 1 Repassivation Potential Results from the Round Robin

	Lab 1	Lab 6	Lab 15	Lab 13	Lab 3	Overall (all labs)
Individual Repassivation Potential Values (mV, Saturated Silver Chloride Scale)	-111	-114	-82	-102	-106	...
	-106	-135	-82	-125	-91	...
	-116	-110	-97	-100	-110	...
	-112	-112	-94	...	...	...
	-119	...	-102	...	...	...
	...	...	-95	...	...	...
Average	-112.8	-117.8	-92	-109	-102.3	-106.8
Standard Deviation	5	11.6	8.2	13.9	10.0	10.0



NOTE—Attack occurred under all of the twelve footprints of the MCA. White areas around the footprints (mainly at 4 and 8 o'clock) are deposits of salts or corrosion products. The letters THE51 represent the specimen ID.

FIG. 6 Example of Crevice Corrosion affecting Alloy 22 test electrodes in the THE Round Robin Test

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