



Designation: G100 – 89 (Reapproved 2021)

Standard Test Method for Conducting Cyclic Galvanostaircase Polarization¹

This standard is issued under the fixed designation G100; 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 cyclic galvanostaircase polarization (GSCP) to determine relative susceptibility to localized corrosion (pitting and crevice corrosion) for aluminum alloy 3003-H14 (UNS A93003) (1).² It may serve as guide for examination of other alloys (2-5). This test method also describes a procedure that can be used as a check for one's experimental technique and instrumentation.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*³

D1193 Specification for Reagent Water

G1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens

G5 Reference Test Method for Making Potentiodynamic Anodic Polarization Measurements

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

³ 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.

G59 Test Method for Conducting Potentiodynamic Polarization Resistance Measurements

G69 Test Method for Measurement of Corrosion Potentials of Aluminum Alloys

3. Significance and Use

3.1 In this test method, susceptibility to localized corrosion of aluminum is indicated by a protection potential (E_{prot}) determined by cyclic galvanostaircase polarization (1). The more noble this potential, the less susceptible is the alloy to initiation of localized corrosion. The results of this test method are not intended to correlate in a quantitative manner with the rate of propagation of localized corrosion that one might observe in service.

3.2 The breakdown (E_b), and protection potentials (E_{prot}) determined by the cyclic GSCP method correlate with the constant potential corrosion test (immersion-glassware) result for aluminum (1, 6, 7). When the applied potential was more negative than the GSCP E_{prot} , no pit initiation was observed. When the applied potential was more positive than the GSCP E_{prot} , pitting occurred even when the applied potential was less negative than E_b .

3.2.1 Severe crevice corrosion occurred when the separation of E_b and E_{prot} was 500 mV or greater and E_{prot} was less than -400 mV vs. SCE (in 100 ppm NaCl) (1, 6, 8). For aluminum, E_{prot} determined by cyclic GSCP agrees with the repassivation potential determined by the scratch potentiostatic method (1, 9). Both the scratch potentiostatic method and the constant potential technique for determination of E_{prot} require much longer test times and are more involved techniques than the GSCP method.

3.3 DeBerry and Viebeck (3-5) found that the breakdown potentials (E_b) (galvanodynamic polarization, similar to GSCP but no kinetic information) had a good correlation with the inhibition of localized corrosion of 304L stainless steel by surface active compounds. They attained accuracy and precision by avoiding the strong induction effect which they observed by the potentiodynamic technique.

3.4 If this test method is followed using the specific alloy discussed it will provide (GSCP) measurements that will reproduce data developed at other times in other laboratories.

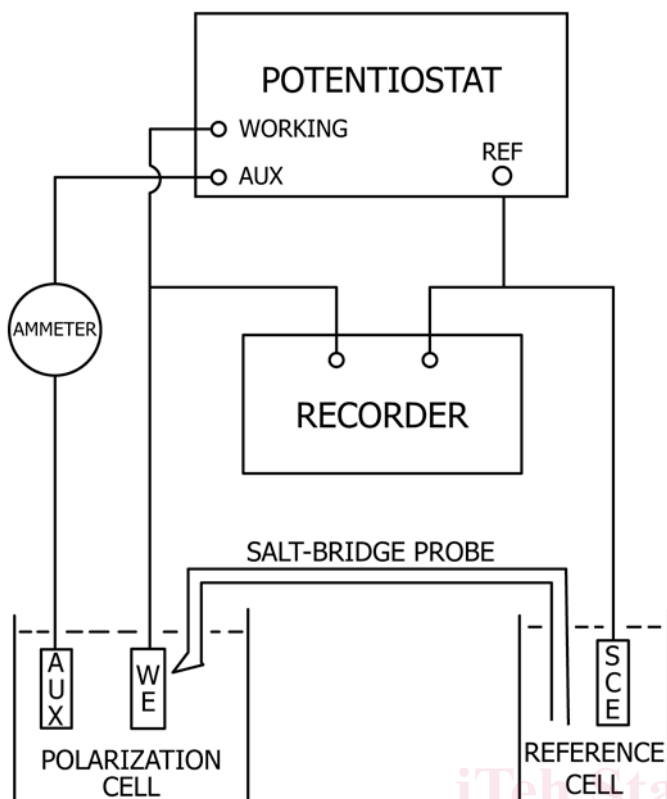


FIG. 1 Schematic Wiring Diagram for Galvanostaircase Polarization

3.5 E_b and E_{prot} obtained are based on the results from eight different laboratories that followed the standard procedure using aluminum alloy 3003-H14 (UNS A93003). E_b and E_{prot} are included with statistical analysis to indicate the acceptable range.

4. Apparatus

4.1 *Cell*—The cell should be constructed of inert materials such as borosilicate glass and PTFE fluorocarbon. It should have ports for the insertion of a working electrode (1 cm² flat specimen holder (Note 1) is very convenient), two auxiliary electrodes, salt bridge for reference electrode, and a thermometer or a thermostat probe for temperature control. The figure in Reference Test Method G5 would be satisfactory, but a flat bottom cell is also satisfactory provided that all of the essential ports are provided. (See Ref (10) for details.)

NOTE 1—These specific recommendations and conditions were followed to improve the interlaboratory precision during the round robin for galvanostaircase polarization.

4.2 *Current Staircase Generator and Recorder*—The schematic diagram of the apparatus is given in Fig. 1. The recorder may be replaced by a plotter if the current staircase signal is generated with the aid of a computer. The current staircase may be generated manually (Note 2) but this is not recommended. The most convenient current staircase generators are found in recent commercial potentiostats where the software is available. The electrical equipment may be checked in accordance with the procedure in Test Method G59.

NOTE 2—The current staircase signal was generated manually in the round robin because automated system or software was not available when this project was started.

4.3 Electrodes:

4.3.1 *Working Electrode*—For generating data to be compared to the reference data included herein, use type 3003-H14 (UNS A93003) A1 in sheet form. Cut 1.55 cm diameter circles and prepare in accordance with Practice G1 using 600 grit diamond slurry on a flat lapping machine. Install in flat specimen holder using PTFE gasket (no crevice type) (Note 1) so that 1 cm² is exposed to the test solution. Apply 29 m-g of torque.

4.3.2 *Auxiliary Electrodes*—Graphite, (ultrafine grade) (Note 3).

NOTE 3—Coarse grades of graphite should be avoided because they absorb solute impurities. Ultrafine grades are available from spectrographic supply companies.

4.3.3 *Reference Electrode*—Saturated calomel (Note 1). It should be checked against another reference which has not been exposed to test solutions and they should be within 3 mV of each other. Test Method G69 round robin test conducted by G01.11 (unpublished results) indicate that potential difference should not exceed 2 mV or 3 mV. The reference electrode is connected to the test bridge solution which consists of 75 % saturated KCl, prepared by adding 1 part (by volume) of distilled water to 3 parts saturated KCl. When the bridge is in active use, the bridge solution should be replaced once each day and the bridge tip immersed in this solution when not in use. Any test solution that does not deposit films may also be used in the bridge. (The VYCOR⁴ tip **should not** be allowed to go to dryness.)

4.4 Magnetic Stirrer.

5. Procedure

5.1 Test solution, 3000 ppm ± 30 ppm (0.0513 M) NaCl. For example, transfer 6.000 g reagent grade NaCl to a 2 L volumetric flask. Dissolve in ASTM Type IV water (demineralized or distilled) and dilute to the mark. (See Specification D1193.)

5.2 Assemble cell with the electrodes described in Section 4. Place the reference bridge probe about 2 probe tip diameters away from the working electrode.

5.3 Fill the cell with the test solution so that the level is about 25 mm above the working electrode.

5.4 Maintain a temperature of 25 °C ± 1 °C.

5.5 Do not deaerate.

5.6 Turn on the magnetic stirrer to a maximum speed that will maintain a smooth vortex above the specimen without whipping air bubbles into the solution.

5.7 Apply a current staircase signal from 0 μA/cm² to 120 μA/cm² using a step height of 20 μA/cm² and step duration of 2 min; reverse the current staircase scan to 0 current. Record the voltage transients on an X-Y or X-T recorder or plotter as

⁴ VYCOR is a trademark of Corning Incorporated, One Riverfront Plaza, Corning, NY 14831, Code No. 7930 glass.

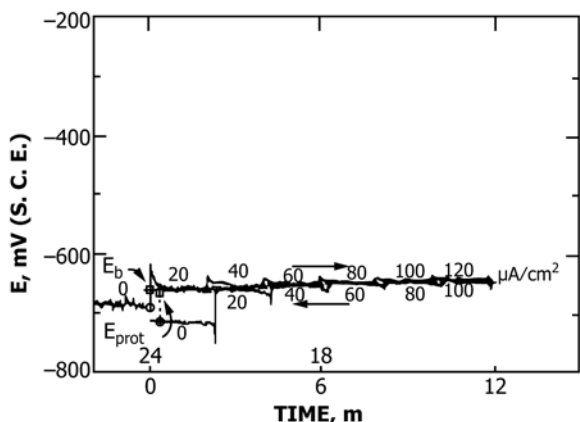


FIG. 2 Cyclic GSCP Curve of 3003 A1 in 3000 ppm NaCl (Taken from Ref 8)

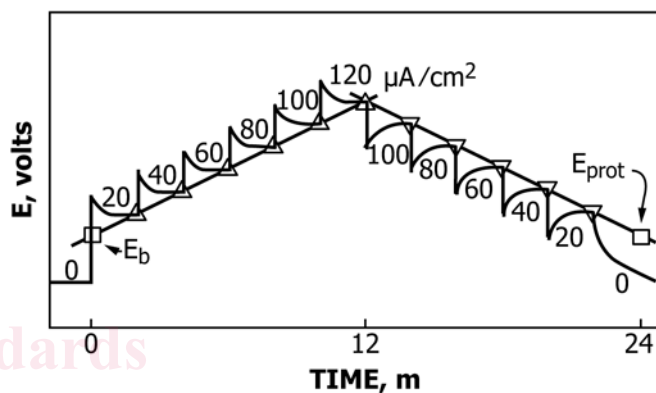
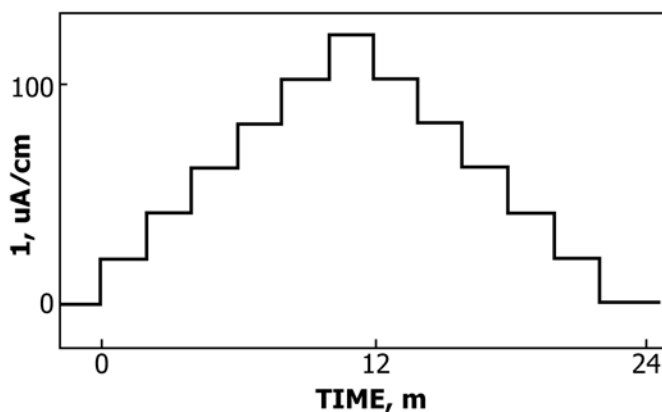


FIG. 3 Relationship of a Schematic GSCP Curve (lower) to the Current Staircase Signal (upper)

shown in Fig. 2 (Note 4). In order to differentiate between the steady-state potential values of the forward scan from those of the reverse scan, it would be helpful to (1) delay the actual reversal of the pen about 12 s after dropping from 120 $\mu\text{A}/\text{cm}^2$ to 100 $\mu\text{A}/\text{cm}^2$ so that there will be a separation of about 24 s between the forward and reverse steady state points and (2) change the pen color in the reverse scan.

NOTE 4—Fig. 2 can be elucidated with the help of Fig. 3. The upper curve in Fig. 3 shows the current staircase signal applied in 5.7 and the lower curve gives schematic voltage response transients with the current density given for each transient. The current is selected at the end of each step (even though current is constant during a step) because the steady state voltage is obtained at the end of the step. This allows extrapolation to zero current which is a discrete current value at each end of the lower curve. In Fig. 2, the down-steps are reversed with a slight delay to separate up-step (triangles pointing upward) from the down-step (triangle pointing downward) steady state voltage.

5.8 *Extrapolation*—Extrapolate the up-step points to zero current to obtain E_b . Similarly, extrapolate the down-step points to obtain E_{prot} . Fig. 2 and Fig. 3 give examples of these extrapolations.

6. Precision and Bias

6.1 *Precision*—The precision information is based on data obtained by the GSCP Task Group with eight laboratories participating. Each laboratory ran duplicate results on the one test solution. The mean value for E_b was -636 mV with a standard deviation of 15.8 mV. The mean value for E_{prot} was -652 mV with a standard deviation of 14.8.

6.2 The repeatability of this technique was 3.5 mV for E_{prot} and 7.3 mV for E_b in terms of the pooled standard deviation. (See Note 5.)

6.3 *Bias*—This procedure has no bias because the values of E_b and E_{prot} can be defined only in terms of this method. If the voltage transients are omitted from Fig. 2 and Fig. 3, typical quasi-stationary galvanostatic polarization plots are obtained.

However, the kinetic and noise information derived from the voltage transients are desirable attributes of GSCP.

NOTE 5—The standard deviation was derived from:

$$S^2 = \sum_{i=1}^N \frac{(Y_i - \bar{Y})^2}{N - 1} \quad (1)$$

where:

- Y = the i^{th} result,
- \bar{Y} = the average of all Y_i values, and
- N = is the total number of results.

The pooled standard deviation was derived from:

$$(\text{Spooled})^2 = \sum_{i=1}^K \frac{(J_{1i} - J_{2i})^2}{2K} \quad (2)$$

where:

- K = the number of laboratories and J_{1i} and J_{2i} are the duplicate results from the i^{th} laboratory.

7. Keywords

7.1 aluminum; corrosion; electrochemical measurement; galvanostaircase; localized corrosion; polarization