



Designation: G108 – 94(Reapproved 2010)

# Standard Test Method for Electrochemical Reactivation (EPR) for Detecting Sensitization of AISI Type 304 and 304L Stainless Steels<sup>1</sup>

This standard is issued under the fixed designation G108; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers a laboratory procedure for conducting an electrochemical reactivation (EPR) test on AISI Type 304 and 304L (UNS No. S30400 and S30403, respectively) stainless steels. This test method can provide a non-destructive means of quantifying the degree of sensitization in these steels (**1**, **2**, **3**).<sup>2</sup> This test method has found wide acceptance in studies of the effects of sensitization on intergranular corrosion and intergranular stress corrosion cracking behavior (see Terminology **G15**). The EPR technique has been successfully used to evaluate other stainless steels and nickel base alloys (**4**), but the test conditions and evaluation criteria used were modified in each case from those cited in this test method.

1.2 The values stated in SI units are to be regarded as the standard. The inch-pound units given in parentheses are for information only.

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

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>3</sup>

- [A262 Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels](#)
- [D1193 Specification for Reagent Water](#)
- [E3 Guide for Preparation of Metallographic Specimens](#)
- [E7 Terminology Relating to Metallography](#)

<sup>1</sup> 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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<sup>2</sup> The boldface numbers in parentheses refer to a list of references at the end of this standard.

<sup>3</sup> 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.

- [E112 Test Methods for Determining Average Grain Size](#)
- [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 \(Withdrawn 2010\)<sup>4</sup>](#)
- [G28 Test Methods for Detecting Susceptibility to Intergranular Corrosion in Wrought, Nickel-Rich, Chromium-Bearing Alloys](#)
- [G61 Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion Susceptibility of Iron-, Nickel-, or Cobalt-Based Alloys](#)

## 3. Terminology

### 3.1 Definitions of Terms Specific to This Standard:

3.1.1 *integrated charge ( $Q$ )*—the charge measured, in coulombs, during reactivation as given by the time integral of current density below the reactivation peak of the curve.

3.1.2 *maximum anodic current density ( $I_r$ )*—the current density measured at the peak of the anodic curve during reactivation.

3.1.3 *normalized charge ( $P_a$ )*—the integrated current normalized to the specimen size and grain size.  $P_a$  represents the charge (in coulombs/cm<sup>2</sup>) of the grain-boundary area. The method for calculating  $P_a$  is given in **9.2**.

3.1.4 *reactivation*—in the electrochemical reactivation (EPR) test, the potential sweep from the passivation potential returning to the corrosion potential.

3.1.5 *scan rate*—the rate at which the electrical potential applied to a specimen in a polarization test is changed.

## 4. Summary of Test Method

4.1 The EPR test is accomplished by a potentiodynamic sweep from the passive to the active regions of electrochemical potentials in a process referred to as reactivation. The EPR test

<sup>4</sup> The last approved version of this historical standard is referenced on www.astm.org.

measures the amount of charge associated with the corrosion of the chromium-depleted regions surrounding chromium carbide precipitated particles. Most of these particles in a sensitized microstructure are located at grain boundaries (see Terminology E7). Discrete particles located within the grain (referred to as intragranular precipitates) will also contribute to the total measured charge. Therefore, it is important to examine the alloy microstructure following an EPR test, to determine the relative proportion of corrosion site associated with intergranular versus intragranular precipitates.

4.2 The chromium-depleted zones around carbide precipitates in sensitized steels are particularly susceptible to corrosion in oxidizing acid solutions. Corrosion at chromium-depleted grain boundary sites causes a rapid rise in the current density when the electrochemical potential is changed from the passive to the active region.

4.3 A sensitized steel produces a curve similar to the active portion of the polarization curve during the reactivation from the passive region back to the rest potential ( $E_{corr}$ ) as shown in Fig. 1. A nonsensitized (solution annealed) steel polarized under the conditions given in this test method will produce a curve with lower current densities than a sensitized steel.

4.4 The EPR test results are readily reproducible, as long as the electrolyte temperature, electrolyte composition, and scan rate are carefully controlled. The EPR test is significantly affected by the composition, thermomechanical condition and surface finish of the specimen as well as the presence of non-metallic inclusions, that result in pitting of the etched microstructure.

NOTE 1—Various cutting and grinding operations can promote sensitization of Type 304 (5). Superficial carbide precipitation can occur during cutting and grinding or during subsequent low temperature heat treatments, such as 24 h at 500°C.

4.5 The criteria used to distinguish between sensitized and solution annealed samples are the activation charge density,  $Q$  (given by the time integral of current density below the reactivation peak of the curve), or the maximum anodic current density,  $I_r$ , in the active state. Sensitized steels are easily

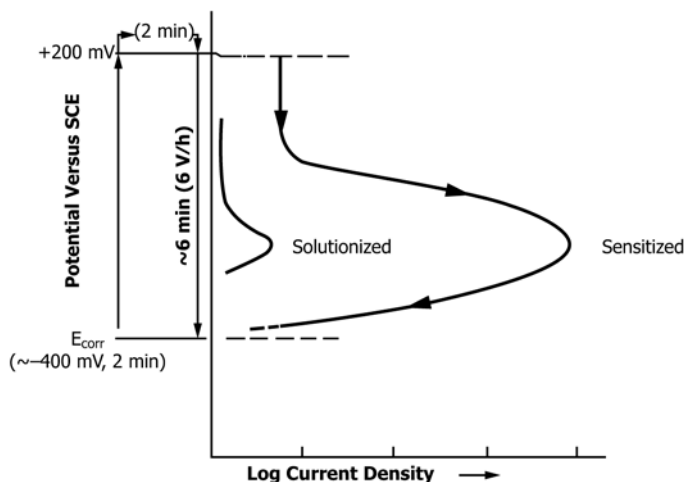


FIG. 1 Schematic EPR Curves for Sensitized and Solutionized AISI Type 304 Stainless Steel

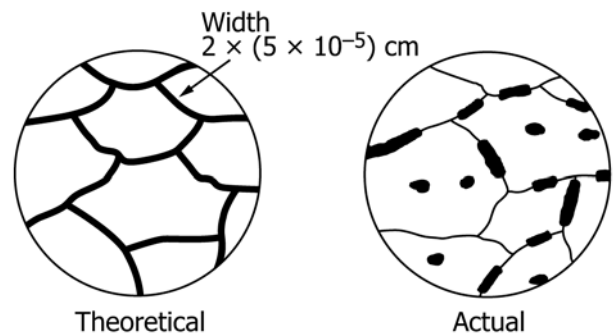
activated and show higher  $Q$  and  $I_r$  values than solution annealed steels, that are not susceptible to intergranular corrosion. The value  $Q$  is normalized for both specimen size and grain size. The value normalized in this fashion is called  $P_a$  and represents the charge (in units of coulombs) per unit grain-boundary area. This normalization permits direct comparisons of different heats of material that exhibit different  $Q$  values solely as a result of differences in grain size.

## 5. Significance and Use

5.1 This test method describes an EPR test method for quantitatively determining the relative degree of sensitization in AISI Type 304 and 304L stainless steels. The EPR test has found wide use as a means to provide a numerical level of sensitization in studies of the effects of sensitization on intergranular corrosion and intergranular stress corrosion cracking behavior. The results of this test method correlate with other test methods (for example, Practices A262 and Test Methods G28) that are commonly used to assess sensitization in stainless steels.

5.2 The EPR test can also be used for product acceptance, service evaluation, regulatory statutes, and manufacturing controls providing that both the supplier and user have agreed upon appropriate acceptance criteria and a sensitizing treatment. The test is not intended for design purposes since the test conditions accelerate corrosion in a manner that does not simulate any actual service environment.

5.3 The EPR test involves the measurement of the amount of charge resulting from the corrosion of the chromium-depleted regions surrounding the precipitated chromium carbide particles. Most of these particles in a sensitized microstructure are located at the grain boundaries. However, discrete particles located within grains (referred to as intragranular precipitates) will also contribute to the total measured charge. (See Fig. 2.) Therefore, it is important to examine the alloy microstructure following an EPR test to determine the relative proportion of corrosion sites associated with intergranular versus intragranular precipitates. Sites of intergranular attack will appear similar to grain boundary ditching as defined in Practice A of Practices A262.



NOTE 1—The calculation of  $P_a$  is based on the assumptions illustrated at left. Mild cases of sensitization usually result in a combination of intergranular attack and pitting as illustrated at right (6).

FIG. 2 Schematic Microstructures After EPR Testing

## 6. Apparatus

6.1 The apparatus necessary for obtaining EPR data consists of electronic instruments and a test cell. These instruments may be integrated into one instrument package or may be individual components. Either form of instrumentation can provide acceptable data.

6.2 Typical apparatus, as illustrated in Fig. 3, shall consist of the following: scanning potentiostat (or potentiostat/voltage ramp generator combination), potential measuring instrument, current and current integration measuring instruments, and test cell and specimen holder.

6.2.1 *Scanning Potentiostat*—Requirements shall be in accordance with 4.2 of Test Method G5 with the following refinements: the potentiostat shall control the potential within  $\pm 5$  mV accuracy over the range of potential and current density encountered in the EPR measurements. The potentiostat shall be operable in a potential range of  $-600$  to  $+500$  mV (SCE) and a current density range of  $1 \mu\text{A}$  to  $100 \text{ mA/cm}^2$ . The applied potential is changed either automatically or manually in the following manners:

6.2.1.1 Shifting the potential from the open circuit potential to a potential in the passive range, and

6.2.1.2 Scanning back to the open circuit potential (re-activation) at a voltage scan rate of  $1.67 \text{ mV/s}$  ( $6 \text{ V/h}$ ).

6.2.2 *Potential Measuring Instruments*—Requirements shall be in accordance with 4.3 of Test Method G5 except that the potential range is as stated above.

6.2.3 *Current Measuring Instruments*—Requirements shall be in accordance with 4.4 of Test Method G5. However, current measurements are essential for passivation assessment and other intermediate checks of system stability. The currents encountered in EPR for a specimen with the dimensions given in 7.3 are in the range of  $1 \mu\text{A}$  to  $100 \text{ mA/cm}^2$ . For samples of less than  $100 \text{ mm}^2$  test area, currents above about  $20 \text{ mA/cm}^2$  rarely have been reported.

6.2.4 *Current Integration Measurement Instruments (Optional)*—Current integration, or charge, can be measured by an electronic device incorporated into the potentiostat, or by a separate electronic device, such as a coulometer. If a coulom-

eter is used, it shall be capable of measuring charges from  $0.001$  to  $2$  coulombs. The use of a coulometer shall be considered optional. Charge can also be measured by using a chart recorder, as illustrated in Fig. 3, to record a current versus time trace and then, subsequently, integrating it by various methods. When potentiostat measurements are available in a digitized format, an appropriate computer integration routine can also be used to obtain a value for charge.

6.2.5 *EPR Test Cell*—Requirements shall be in accordance with 4.1 of Test Method G5. A deaeration tube is not required and only one counter electrode is required for EPR testing. A suitable cell and electrode arrangement is shown in Fig. 4.

6.2.6 *Electrode Holder*—Requirements shall be in accordance with 4.6 of Test Method G5 or 4.2.1 of Test Method G61. The requirements for the working electrode (specimen) and counter electrode holders are that the holders be made of an inert material and any seals must not allow leakage of the electrolyte. When using the Test Method G5-type holder the working electrode can be mounted as shown in Fig. 5 and described in Appendix X1.

6.2.7 *Auxiliary (Counter) Electrodes*—Requirements are in accordance with 4.7.2 of Test Method G5 except that only one counter electrode is necessary for EPR testing. However, two auxiliary electrodes can provide for a more uniform distribution of current. Titanium or high-purity carbon may be used in place of platinum for the counter electrode since it is always the cathode.

6.2.8 *Calomel Reference Electrode*—Requirements are in accordance or equivalent to 4.7.3 of Test Method G5.

## 7. Sampling, Test Specimens, and Test Units

### 7.1 Sampling:

7.1.1 When using this test method to meet product acceptance criteria, the means of sampling of a test specimen shall be

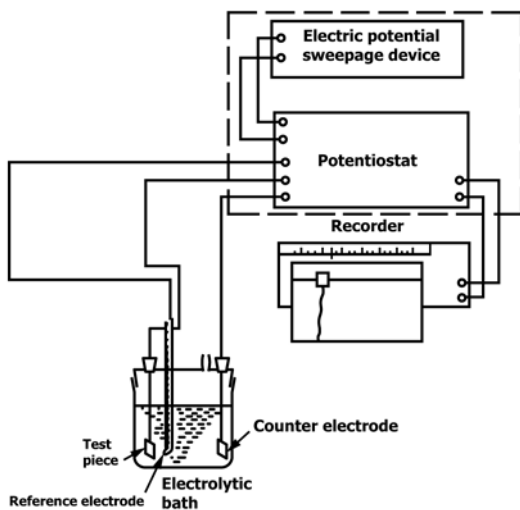
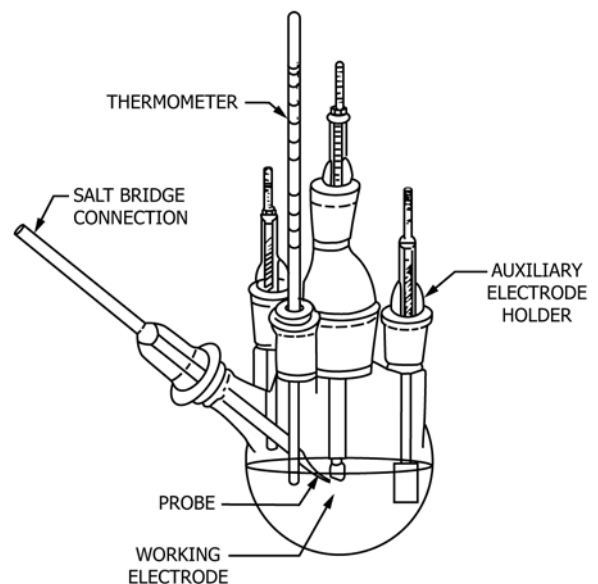


FIG. 3 Schematic Diagram of an EPR Test Apparatus



NOTE 1—The sample face is completely immersed but the connection to the electrode holder is not immersed.

FIG. 4 Schematic Diagram of an Electrochemical Cell for EPR Testing