Standard Test Method for
Electrochemical Reactivation (EPR) for Detecting
Sensitization of AISI Type 304 and 304L Stainless Steels[1]

1. Scope

1.1 This test method describes 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 nondestructive means of quantifying the degree of sensitization in these steels **(1, 2, 3)**.[2] 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 G 15). 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 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:*

A 262 Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels[3]

D 1193 Specification for Reagent Water[4]

E 3 Methods of Preparation of Metallographic Specimens[5]

E 7 Terminology Relating to Metallography[5]

E 112 Test Methods for Determining Average Grain Size[5]

G 1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens[6]

G 3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing[6]

G 5 Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements[6]

G 15 Terminology Relating to Corrosion and Corrosion Testing[6]

G 28 Test Methods for Detecting Susceptibility to Intergranular Attack in Wrought Nickel-Rich, Chromium-Bearing Alloys[6]

G 61 Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion Susceptibility of Iron-, Nickel-, or Cobalt-Based Alloys[6]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *integrated charge (Q)*—the charge measured, in couloumbs, 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^2) 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 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 E 7). 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 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

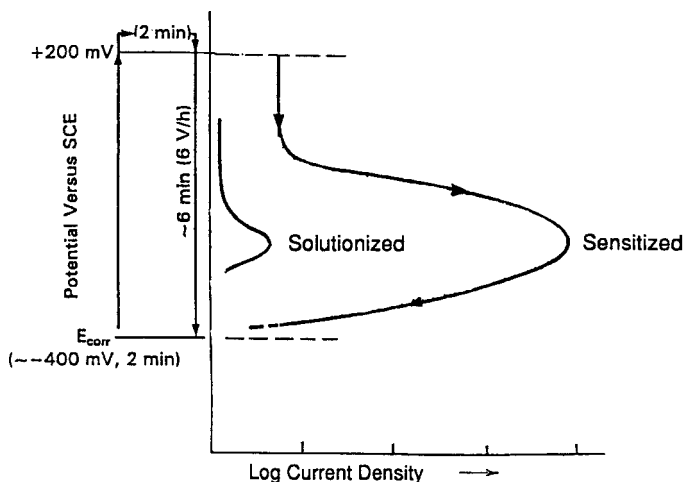5.1 This test method describes an EPR test method for quantitatively determining the relative degree of sensitization in AISI Type 304 and 304L stainless steels. The EPR test has found wide use as a means to provide a numerical level of sensitization in studies of the effects of sensitization on intergranular corrosion and intergranular stress corrosion cracking behavior. The results of this test method correlate with other test methods (for example, Practice A 262 and Test Method G 28) 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 A 262.

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 Practice G 5 with the following refinements: the potentiostat shall control the potential within ± 5

FIG. 1 Schematic EPR Curves for Sensitized and Solutionized AISI Type 304 Stainless Steel
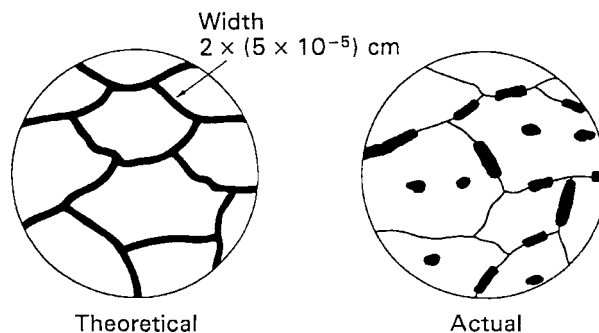
NOTE 1—The calculation of P_a is based on the assumptions illustrated at left. Mild cases of sensitization usually result in a combination of intergranular attack and pitting as illustrated at right (**7**).

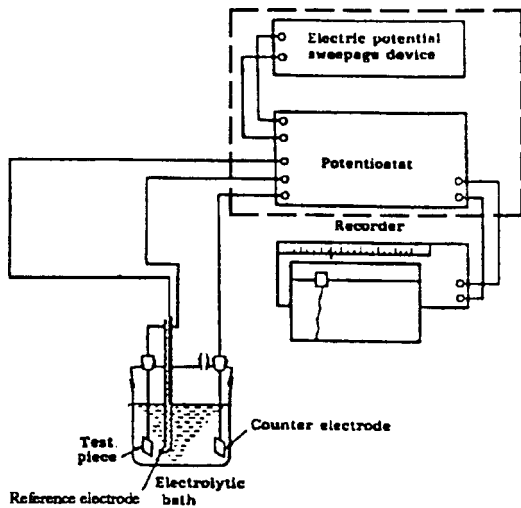FIG. 2 Schematic Microstructures After EPR Testing

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

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 μA to 100 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 (reactivation) at a voltage scan rate of 1.67 mV/s (6 V/h).
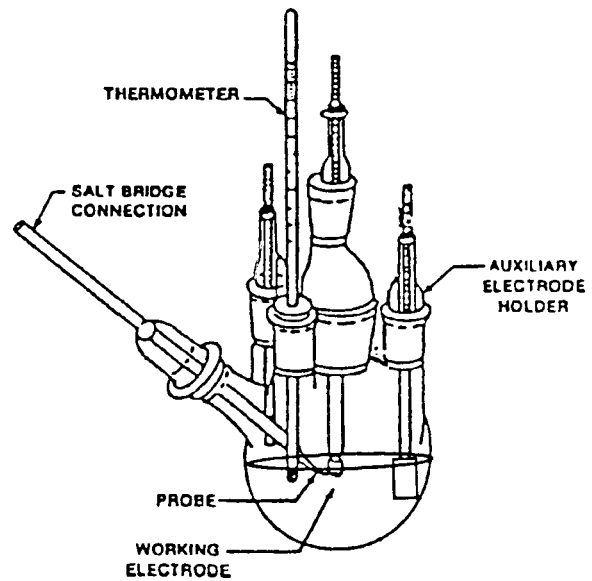
6.2.2 *Potential Measuring Instruments*—Requirements shall be in accordance with 4.3 of Practice G 5 except that the potential range is as stated above.

6.2.3 *Current Measuring Instruments*—Requirements shall be in accordance with 4.4 of Practice G 5. 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 μA to 100 mA/cm^2. For samples of less than 100 mm^2 test area, currents above about 20 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 coulometer 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 Practice G 5. A deareation 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 Practice G 5 or 4.2.1 of Test Method G 61. 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 Practice G 5-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 Practice G 5 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 Practice G 5.

7. Sampling, Test Specimens, and Test Units
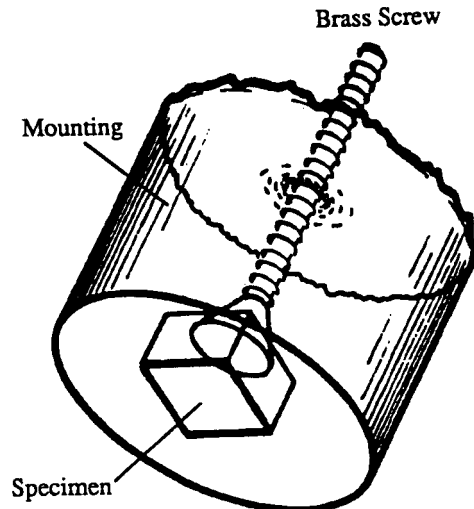
7.1 *Sampling*:

FIG. 5 A Method of Mounting Specimens for EPR Testing (7)

7.1.1 When using this test method to meet product acceptance criteria, the means of sampling of a test specimen shall be decided by agreement between the parties involved; for instance, but not limited to, a user and a supplier.

7.1.2 Specimens removed form a piece of AISI Type 304 or 304L steel by shearing, cutting, burningtc, and so forth shall have the affected edges removed by grinding or machining.

7.2 *Sensitization of Test Specimens*—Specimens can be given a sensitizing treatment when it is desired to assess the influence of a thermal exposure during fabrication on corrosion resistance.

7.2.1 Specimens may be tested in a condition simulating that of the product as installed in service. Specimens may be welded or heat treated in as nearly the same manner as the product will experience in fabrication or service. The user and supplier must agree to the use and conditions of a sensitization treatment. The most common sensitizing treatment is 1 h at 675°C (1250°F) according to 15.3 of Practices A 262.

7.2.2 Heat treatment, particularly carburization, may alter the surface to be tested and may invalidate the EPR test results. Precautions shall be taken to ensure that the specimen surface is representative of the product form in service. Refer to Section 6 of Practice G 1 for descaling procedures and 7.3 as well as Section 5 of Practices A 262 for guidance in preparing specimens.

7.2.3 Expose specimens to be given a sensitization treatment prior to EPR testing in a furnace at the required temperature and for the required time and then water-quench. Use a thermocouple and a timer to ensure that the entire specimen cross sections are at the specified temperature for the specified amount of time. The number of thermocouples needed to obtain a reliable reading for all specimens exposed in a furnace at a given time is left to the discretion of the user.

7.3 *Specimen Preparation*:

7.3.1 Test specimens can be any shape but shall be at least 3.2 mm (0.125 in.) in diameter or on a side dimension and of a suitable thickness. Specimens shall not be larger than 130 mm^2 (0.2 in.^{2}) in area since such specimens will not fit into the recommended mold for mounting (see Appendix X1). A mounted specimen is illustrated in Fig. 5.

7.3.2 Remove any oxides or grease from the specimen as such film may promote loss of adhesion between the mounting compound and the specimen that could cause a crevice to form thereby producing erroneously high current densities during the EPR measurement.

7.3.3 The front surface of the specimen will be evaluated in the EPR test. The back surface of the test specimen is used to establish electrical contact with the specimen (see Note 2).

NOTE 2—A convenient way to make this attachment may be either by spot welding or by using a conducting cement to fasten a stainless steel machine screw (for example, NC4-40 × 0.3 cm (0.75 in.) long) to the back surface of the specimen. This assembly is mounted in a suitable compound that is inert in the EPR electrolyte (see Appendix X1) such that the front surface upon immersion in the EPR electrolyte is fully in contact with the electrolyte.

7.3.4 Measure the surface area of the front surface of the test specimen within 0.1 mm^2 precision and record on the EPR data record sheet (see Appendix X2).

7.3.5 Specimens can be in any shape that will not be susceptible to crevice corrosion in the solution. Test surface area shall be at least 10 ± 0.1 mm^2 (0.016 in.^{2}). It is occasionally useful to mask the area to be measured leaving an opening for exposure to the electrolyte. One suitable masking method uses precut pieces of an acid resistant tape. Care must be taken not to introduce undercutting of the tape during the EPR measurement because it will cause erroneously large currents.

8. Procedure

8.1 *Metallographic Preparation*:

8.1.1 Polish and attach the test specimen, mounted in a suitable inert compound, to the electrode holder following the procedures and cautions described below:

8.1.2 Exercise care since any crevice between the specimen and the mounting compound could lead to erroneously large current densities.

8.1.3 Prepare the surface within 1 h of the experiment, or store the prepared specimen in a suitable desiccating cabinet. Wet grind with 240-grit and 400-grit silicon carbide papers, and wet polish with 600-grit silicon carbide paper until all coarse scratches are removed. Rinse with water and dry. Polish the specimens in two additional stages with 6 and 1 μm diamond paste on a low speed polishing wheel in accordance with Methods E 3.

8.1.4 Polishing specimens on automated, high speed wheels using aluminum oxide slurries is not recommended. Specimens tend to retain an alumina impregnated surface layer that gives erroneous results during the EPR test.

8.1.5 Attach the specimen to the specimen holder as described in either 4.6.1 of Practice G 5 or 4.2.1 of Test Method G 61. In the case of the Practice G 5-type holder, tighten the assembly by holding the upper end of the mounting rod in a vise or clamp while turning the mounting nut until the gasket is properly compressed. Similarly for the Test Method G 61-type holder, it is important to properly compress the TFE-fluorocarbon gasket to minimize the potential for crevice corrosion.

8.1.6 Clean the specimen just before immersion in the electrolyte by degreasing with a suitable detergent, rinsing in distilled water, then reagent grade methanol, and air drying.

8.2 *Test Solution Preparation*:

8.2.1 Prepare a mixture of reagent grade sulfuric acid (H_2SO_4) and potassium thiocyanate (KSCN) in reagent water as follows: 1 L of 0.5 *M* H_2SO_4 +0.01 *M* KSCN and Type IV reagent water (in accordance with Specification D 1193). The solution can be made up in bulk and stored for one month at room temperature. Transfer approximately 500 to 600 mL of solution to a clean test cell.

8.3 *Initiating the Test*:

8.3.1 Bring the temperature of the solution to 30 ± 1°C by immersing the cell in a controlled temperature water bath or by other convenient means.

8.3.2 Place the specimen, counter electrodes, salt bridge probe, and other components in the test cell. Ensure the salt bridge is filled with the test solution and contains no air bubbles, particularly in the restricted space at the tip.

8.3.3 Record the open circuit potential (OCP) of the test specimen after 1 to 2 min of immersion. If the OCP is not

consistent with typical values for the given alloy (for example, − 350 to −450 mV versus SCE for AISI Type 304), cathodically polarize the specimen to −600 mV versus SCE for 0.1 to 1 min and recheck the rest potential. If the rest potential is still abnormal (relative to the usual value around −200 mV for solutionized Type 304 and 304L), the specimen must be removed from the cell and repolished (back to the step for polish with 1 or 6 μm diamond paste is usually sufficient).

8.3.4 Passivation is accomplished by applying the potential to +200 mV versus standard calomel electrode and holding for 2 min. For specimens 1 cm^2 or less in area, a current density of 10 μA/cm^2 or less indicates that the specimen has passivated.

8.4 *Reactivation Scan*:

8.4.1 Set the current integration to zero and start the current integrator instrument. (Some instruments perform these steps automatically.) Start the potential scan in the active direction at the rate of 1.67 ± 0.08 mV/s (6 V/h). During the reactivation scan, the current density will decay quite rapidly.

8.4.2 Record the reading on current integrator when potential reaches 50 mV above (more positive) the initial E_{corr}. This reading is the integrated current or charge value in coulombs. (Some instruments are capable of ending the experiment automatically.) The test is complete once this reading has been obtained.

8.4.3 Once the test is complete, put all electrochemical polarization equipment on standby. Remove the specimen from the cell and holder, rinse it in water, clean with alcohol or detergent, rinse again, and then air dry.

8.4.4 *Optional E Versus Log I Plot*—The recorder automatically plots the anodic polarization data on semilogarithmic paper in accordance with Practice G 3. A strip chart recorder may also be used since potential is linear with time.

8.5 *Metallographic Inspection*:

8.5.1 Photograph surface of each specimen after testing (without additional preparation or etching) at a suitable magnification to determine grain size and to document the microstructures and extent of grain boundary attack. If the specimen is not sufficiently etched after the EPR test to delineate the microstructure for grain size determination, then the specimen shall be etched by either electrolytic 10 % oxalic acid (in accordance with Practice A of Practices A 262), 60 % HNO$_3$-40 % H$_2$O (**6**), or by other suitable means to delineate the grain boundaries.

8.5.2 Examine the microstructure after the EPR test to ensure that the bulk of the integrated current Q value actually represents attack of the grain boundary areas (that is, "ditching" in terms of Practice A of Practices A 262 has occurred). Reactivation of intragranular (matrix) precipitates (principally chromium carbides) that may be present in substantial quantities in some specimens (**7, 8**) may contribute to the integrated current Q value. Intragranular precipitates are only of concern when the Q value is above an established acceptance criteria. In such cases, the user and supplier may have to agree to higher acceptance criteria values that reflect the contribution of intragranular precipitates to the measured Q value. Examples of correlations of Pa values to the degree of sensitization for AISI Type 304 and 304L stainless steels are offered as a general guide to interpretation of EPR results in Appendix X3.

9. Calculation

9.1 Determine the surface area by measuring all dimensions to the nearest 0.1 mm.

9.2 Calculate and record the normalized charge (P_a) in units of coulombs/cm^2, using the following equation:

$$P_a = Q/X \tag{1}$$

where:
Q = charge measured on current integration measuring instrument (coulombs). Q is normalized for both specimen size and grain size,
X = A$_s$[5.1 × 10^{-3} $e^{0.35}$G]
A_s = specimen area (cm^2), and
G = grain size at 100 × (in accordance with Test Method E 112).

NOTE 3—Often in the technical literature, the ASTM grain size number is designated as "X" and the grain boundary area is "GBA."

9.3 In the derivation of the equation in 9.2 it was assumed that the Q value is due to the attack on the specimen surface that is distributed uniformly over the entire grain boundary region of a constant width of 2 × (5 × 10^{-5}) cm. This may not represent the actual physical processes.

10. Report

10.1 Record test parameters as follows:
10.1.1 EPR test number,
10.1.2 Specimen number,
10.1.3 Material,
10.1.4 Heat,
10.1.5 Solution temperature,
10.1.6 Reactivation scan rate,
10.1.7 Passivation potential/time,
10.1.8 Rest potential, and
10.1.9 Specimen surface area.
10.2 Use the example data record sheet in Appendix X2 or an equivalent one for recording these data.

11. Precision and Bias [7]

11.1 *Statement of Precision*:

11.1.1 The precision of the single loop method has been determined by an interlaboratory test program on a set of specimens from a single heat each of Type 304 and Type 304L. Precision in this case has two components repeatability and reproducibility.

11.1.2 Interlaboratory reproducibility of the P_a values decreases with increasing degrees of sensitization. This indicates that P_a values are sensitive indicators of differences in specimens with mild degrees of sensitization, but do not readily distinguish between medium or severely sensitized specimens.

11.1.2.1 Reproducibility refers to the agreement that occurs when samples of a single material are tested by several different laboratories. The results of an interlaboratory test program (**4**) are shown in Table 1. Samples of Type 304 and 304L, compositions given in Table 2, in four different heat treatment conditions were evaluated in the round robin. Each lab value represents the average of three or more tests.

TABLE 1 EPR Test Interlaboratory rogram Reproducibility Study

Lab	As-Received		621°C for 24 h		621°C for 1 h		500°C for 24 h	
	304	304L	304	304L	304	304L	304	304L
1	0.14	0.01	13.6	4.4	0.31	1.10	0.12	0.09
2	0.01	0.00	5.6	2.7	0.20	0.38	0.12	0.16
3	0.00	0.00	5.6	3.6	0.14	0.25	0.07	0.10
4	0.06	0.06	10.3	5.0	0.19	0.11	0.08	0.05
5	0.03	0.01	8.9	2.3	0.15	0.47	0.08	0.10
6	0.00	0.00	1.7	1.8	0.07	0.32	0.05	0.15
7	0.07	0.05	17.9	3.6	0.43	0.95	0.15	0.23
8	0.04	0.02	17.6	3.7	0.47	0.32	0.14	0.08
9	0.002	0.03	17.9	6.6	0.31	0.31	0.03	0.01
10	0.07	0.01	17.6	8.2	0.26	0.50	0.23	0.09
$\bar{P}_a$	0.0422	0.0190	11.70	4.19	0.253	0.471	0.107	0.106
S_R	0.0445	0.0213	6.13	1.97	0.128	0.314	0.058	0.061
LCL[B]	8.62×10^{-4}	0.0022	1.26	1.12	0.050	0.067	0.020	0.008
UCL[B]	0.264	0.0800	3.20	7.85	0.536	1.11	0.230	0.335

[A] Each P_a value is the average of three or more tests.
[B] Lower and upper 95 % confidence limits based on Weibull analysis of data distributions.

TABLE 2 Chemical Compositions of the Alloys, in Weight Percent, Used in the Round Robin

	T-304	T-304L
C	0.049	0.021
Mn	1.30	1.38
P	0.033	0.027
S	0.015	0.009
Si	0.65	0.59
Cr	18.67	18.83
Ni	8.78	10.27
Mo	0.38	0.58
Cu	0.12	0.11
N	0.056	0.020

11.1.2.2 A linear regression (through the origin) analysis of the standard deviation values S_R shown in Table 1 showed that the standard deviations were strongly correlated to the average P_a value of all participating laboratories $\bar{P}_a$, and could be represented by:

$$s_R = 0.52 \bar{P}_a \tag{2}$$

when S_R is the standard deviation of the average values reported by the participating laboratories. However, further analysis showed that the distribution of S_R values was not normal, but could be adequately represented by Weibull two parameter functions. After fitting each of the data set for each material to a best fit Weibull distribution, a 95 % confidence interval could be calculated. These values are shown in Table 2. Linear regression through the origin for both the upper, UCL, and lower, LCL, confidence limits showed that these values were strongly correlated to the average P_a value and could be adequately represented by the following expressions:

$$UCL = 2.63 \bar{P}_a \tag{3}$$

$$LCL = 0.126 \bar{P}_a$$

11.1.3 Repeatability refers to the agreement that occurs when a single laboratory runs sequential tests under identical conditions. Repeatability results are shown in Table 3. The variation in repeatability, as measured by the standard deviation S_R is correlated to the average P_a value. The following expression was determined by a linear regression through the origin of the data listed in Table 3.

$$s_R = 0.19 \bar{P}_a \tag{4}$$

where $\bar{P}_a$ is the average of three sequential tests. The 95 % confidence interval, R, is 2.8 S_R or:

$$R = \pm (0.53) \bar{P}_a \tag{5}$$

$$UCL = 1.53 \bar{P}_a 00$$

$$LCL = 0.47 \bar{P}_a$$

where UCL and LCL are the upper and lower 95 % confidence interval limits.

11.2 *Statement of Bias*:

11.2.1 Variation in electrolyte temperature, electrolyte composition, scan rate, specimen composition, specimen thermomechanical condition, and specimen surface finish affect the measured Q value and will constitute a source of bias.

11.2.2 The EPR results are reproducible using different

TABLE 3 EPR Test Round Robin Results for Repeatability

NOTE 1—P_a [coulombs/cm²] values from three labs, (each value is an average of three or more tests).

Lab	As-Received		621°C for 24 h		621°C for 1 h		500°C for 24 h	
	304	304L	304	304L	304	304L	304	304L
(Sample)	(1)	(5)	(2)	(6)	(3)	(7)	(4)	(8)
10A	0.002	0.03	4.00	3.20	0.31	0.31	0.0016	0.0001
S	± 0.000	± 0.00	± 0.39	± 0.49	± 0.02	± 0.05	± 0.0002	± 0.000
10B	0.07	0.00	13.1	8.2	0.26	0.50	0.23	0.09
S	± 0.01	± 0.00	± 2.45	± 2.36	± 0.01	± 0.00	± 0.00	± 0.03
11	0.02	0.00	14.2	3.97	2.2	0.10	0.59	0.03
S	± 0.01	± 0.00	± 2.71	± 0.31	± 0.12	± 0.02	± 0.06	± 0.02

polarization instrumentation and correlate well to the degree of intergranular carbide precipitation observed metallographically in Practice A of Practices A 262 **(7)**.

11.2.3 As discussed in Section 5, pitting caused by the dissolution of non-metallic inclusions can increase the P_a value. In such cases, it is recommended to examine the microstructure after the test to identify the source of the elevated P_a value.

12. Keywords

12.1 carbide precipitation; electrochemical reactivation (EPR) test; electrochemical test; intergranular corrosion; sensitization; stress corrosion cracking

APPENDIXES

(Nonmandatory Information)

X1. SUGGESTED METHOD FOR PREPARING MOUNTED TEST SPECIMENS

X1.1 Center the specimen in the mold. Cap lugs (pipe end protectors) may be used. Make sure the specimen does not touch the mold wall.

X1.2 Prepare enough Marset[8] resin to fill the mold to imbed the entire sample and part of the screw. In some cases, it may take more depending on the size of the samples.

X1.2.1 Preparation of the Marset[8] mount involves mixing 70 mL of resin (Marset resin No. 655)[8] with 10 mL of hardener

(Marset hardener No. 555)[8] to a cloudy consistency, stir.

X1.2.2 Place the mixture in an 80°C (176°F) oven for 1 to 1.5 h, or until it turns clear.

X1.2.3 After about 30 min, remove mixture from oven and stir.

X1.2.4 Pour the clear mixture slowly to avoid upsetting the specimen. Place in the 80°C oven for at least 8 h, or longer.

X1.3 After the specimen has been potted, the mold is removed and the sample number engraved on the top of the mount. Chamfer the sharp edges of the mount for ease in handling and polishing. Resin may accumulate on the screw threads, to remove chase the threads with a 4-40 button die.

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[8] Marset is a product of Acme Chemical and Insulation, 166 Chapel St., New Haven, CT 06506.

X2. ELECTROCHEMICAL POTENTIOKINETIC REACTIVATION DATA RECORD SHEET

1. EPR Test Number _____

2. Specimen Number _____

3. Material and Heat Number _____

4. Material Heat Treatment   Heat Treat Lot No. _____

5. Specimen Location _____

6. Specimen Surface Area, A _____ cm^2

7. Initial Open Circuit Potential _____ mV vs SCE

8. Test Temperature _____ °C

9. Passivating Potential/Time _____ mV vs SCE/sec

10. Reactivation Rate _____ mv/s

11. Integrated Current, Q, _____ Coulombs

12. Maximum anodic current density, I_r, _____ mA/cm^2

13. ASTM Grain Size, G, _____

14. Normalized Charge, P_a, _____ coulombs/cm^2
     Calculated using the following equation: $P_a = Q/X$
where:
Q = charge in coulombs as measured by current integrating instrument
X = charge normalization factor $= A_s [5.1 \times 10^{-3} \exp (0.35\,G)]$
     where:
     A_s = Specimen Area (cm^2)
     G = ASTM Grain size at 100X (per ASTM E 112)
15. Attach:
     a. A micrograph characterizing microstructure and extent of grain boundary attack.
     b. The micrograph that was used to determine the grain size. Note: Micrograph 'a.' may be the same as 'b'.

Date _____

FIG. X2.1 Electrochemical Potentiokinetic Reactivation Data Record Sheet

X3. CORRELATION OF Pa VALUES TO DEGREE OF SENSITIZATION FOR AISI TYPES-304 AND -304L

X3.1   Due to the wide range of applications for AISI Types-304 and -304L stainless steels, the acceptance limits for an EPR test must be established by the user or by agreement between the user and supplier. The following correlations are offered as a general guide to interpretation of EPR results:

Pa Value	General Interpretation
< 0.10	Unsensitized microstructure; no pitting
0.10–0.4	Slightly sensitized microstructure; pitting and limited intergranular attack
> 0.4	Sensitized microstructure; pitting and attack of entire grain boundaries.

It is necessary to examine the etched microstructures after the EPR test to establish whether or not high Pa values are actually caused by pitting and grain boundary attack.

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ASTM G 108