

Standard Test Method

Testing of Catalyzed Titanium Anodes for Use in Soils or Natural Waters

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Foreword

This NACE International standard has been prepared to provide users and manufacturers of impressed current cathodic protection (CP) anodes with a test method for evaluating the anode's ability to achieve an expected lifetime. It is applicable to catalyzed titanium anodes intended for use in underground or underwater environments.

This test method is intended to evaluate whether such anode complies with required specifications of design life expectancy at rated current output. This is not a quality assurance (QA) test, but rather a test method that may be used to develop a QA test.

The appendixes of this test method are provided for guidance and as a supplement to the test method. They outline the logic for developing a QA test based on this test method.

This standard was originally prepared in 2008 by NACE Task Group (TG) 017, "Anodes, Catalyzed Titanium: Testing for Use in Soils and in Waters," and it was revised by TG 017 in 2012. TG 017 is administered by Specific Technology Group (STG) 05 on Cathodic/Anodic Protection. This standard is issued by NACE under the auspices of STG 05.

In NACE standards, the terms *shall*, *must*, *should*, and *may* are used in accordance with the definitions of these terms in the *NACE Publications Style Manual*. The terms *shall* and *must* are used to state a requirement, and are considered mandatory. The term *should* is used to state something good and is recommended, but is not considered mandatory. The term *may* is used to state something considered optional.

NACE International Standard Test Method

Testing of Catalyzed Titanium Anodes for Use in Soils or Natural Waters

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Section 1: General

1.1 Test Method for Catalyzed Titanium

This test method is for anodes comprised of a titanium substrate to which a mixed metal oxide (MMO) catalytic coating has been applied. This test method accelerates the time-to-failure by operating the anode at higher current than the application's design requirements.

1.2 The user should not imply that this test method is mandatory for any application of these anode materials.

1.3 Alternatively, the user may elect to use the manufacturer's rating instead of developing the test methodology independently.

1.4 Limitations

1.4.1 Accelerated testing should provide conservative results by stressing the anode more severely than normal applications. See Appendix A (Nonmandatory) for further discussion.

1.4.2 All manufacturers' products may not perform identically. Comparing test samples to a correlation curve established for a different manufacturer's product may lead to erroneous results.

1.4.3 The result for a sample run in the accelerated life test may be used to validate its performance at the design current density only if the correlation curve has been established for that material. See Appendix B (Nonmandatory) for development of the correlation curve using this test method.

1.4.3 This test method is not an acceptance criterion. It may be used to develop a test criterion as shown in Appendix C (Nonmandatory).

1.4.4 This test method outlines the test conditions that must be maintained for effective comparisons.

1.4.5 Varying the test conditions may have a significant effect on the sample lifetime, leading to erroneous results.

1.4.6 The test method as described in this standard should not be interpreted as being the only correct test method that may be used.

Section 2: Definitions

Mixed Metal Oxide (MMO): A mixture of a platinum group metal (Pt, Ir, Ru) oxide and a valve metal (Ti, Ta, Nb, Zr) oxide. The most common mixture used for cathodic protection is iridium oxide and tantalum oxide. Exact compositions may vary.

Section 3: Test Apparatus

3.1 The test equipment is comprised of an anode (test sample), a cathode, a thermometer, a glass vessel, electrolyte agitation device (e.g., magnetic stirrer), rubber stopper for tall glass beaker, a funnel for deionized water addition during the test, vent tubing, and power supply. See Figure 1.

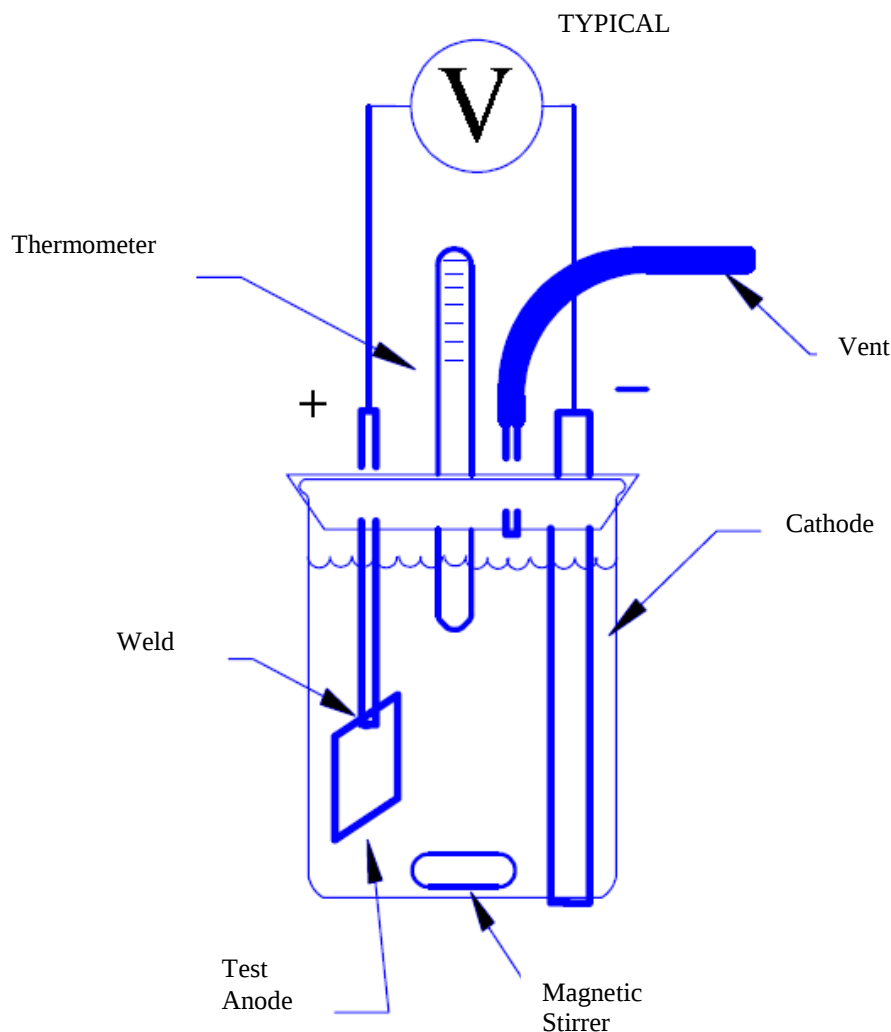


Figure 1: Test cell with anode measurement setup.

3.2 The glass vessel shall be sized to minimize the fluctuation in level resulting from the electrolysis of water during the test and evaporative losses. A 1 L beaker may be used.

3.3 A means of securing the relative positions of the anode sample, the cathode, and the thermometer should be used. This apparatus can be a rubber stopper with holes for the various pieces of equipment and vents or a series of stands and clamps. The gap between the anode and cathode shall be fixed. The anode top and bottom edges shall be at least 10 mm (0.4 in) from the liquid level and the bottom of the beaker, respectively.

3.4 The anode-to-cathode gap should be approximately 20 mm (0.8 in).

3.5 The cathode shall be zirconium, titanium, niobium, or platinum. The size of the cathode shall be sufficient to extend from the bottom of the beaker to well above the top of the beaker. External to the cell, the cathode current conductor shall be securely connected to an insulated copper wire no smaller than 1 mm² (#16 AWG⁽¹⁾). This wire shall be connected to the negative pole of the power supply.

3.6 A thermometer shall be inserted in the cell. If more than one cell is used, each cell shall have a thermometer installed. The exact type of thermometer used shall be in accordance with the general safety practices of the laboratory and shall not have any materials exposed to the electrolyte that could corrode and contaminate the solution. Alternately, a thermocouple may be used.

⁽¹⁾ American Wire Gauge.

The temperature range of the thermometer shall be 20 to 100 °C (68 to 212 °F). Stainless steel or other metallic thermocouples shall not be exposed to the electrolyte to avoid corrosion and contamination.

3.7 The power supply shall be a constant current control, typical for laboratory use. The required amperage shall be determined from the sample size and the current density for the particular accelerated test point. For example, 5 A is appropriate for a 500 mm² sample being run at 10 kA/m². The required voltage depends on the number of test stations in series that may be used. Typically, 8 volts per cell times the number of cells in series is sufficient.

3.8 Voltmeter. Most power supplies have a digital voltmeter. If using a dedicated power supply, then no additional voltmeter is necessary. If using a series of cells arrangement, then each cell shall have a digital voltmeter.

3.9 The simplest operation is to use a single cell with a dedicated power supply.

3.10 Figure 2 illustrates a typical electrical series setup for duplicate evaluations. A separate coulometer or current measuring device (accurate to ± 1%) can be included if not incorporated within the power supply. Alternatively, the product of the current times the test duration may be used.

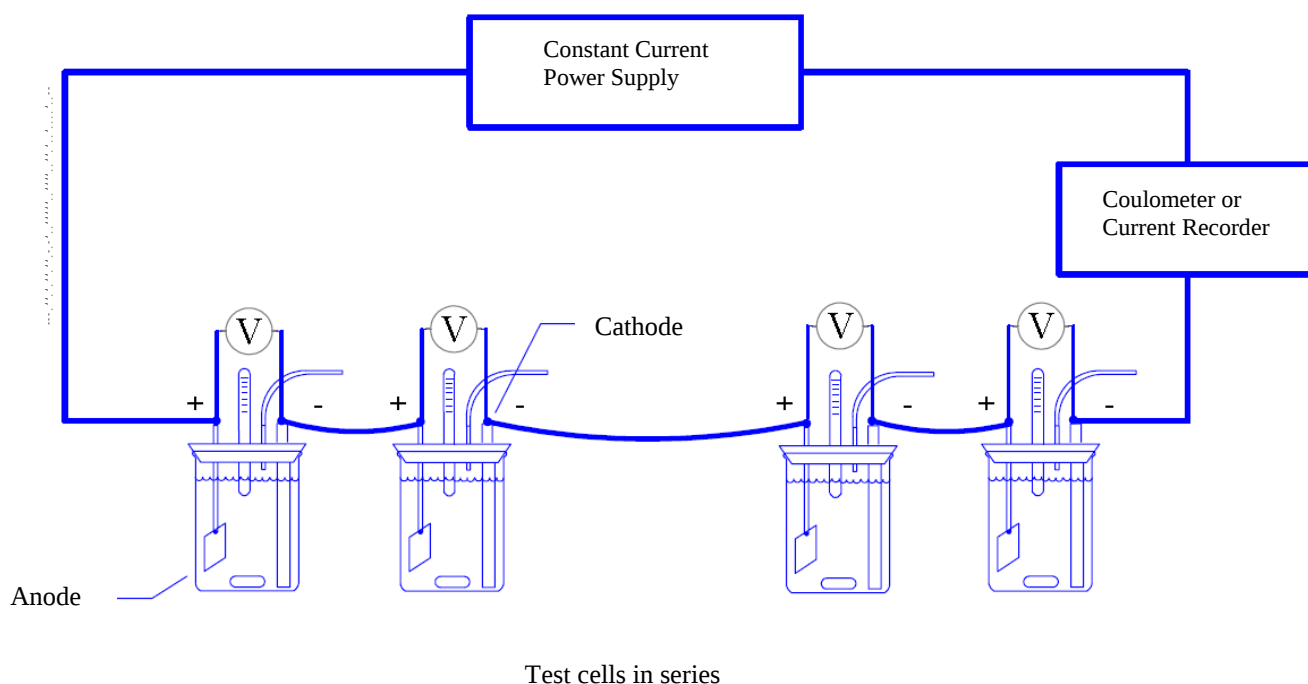


Figure 2: Test cells connected in series.

3.11 Temperature control equipment may be necessary to maintain the electrolyte within the required range depending on laboratory conditions. Essentially, a fluid may be used to input or extract heat from the electrolyte indirectly. Examples of such equipment are jacketed beakers and water bath for beakers.

Section 4: Test Electrolyte

4.1 The composition of the electrolyte used for the test shall be suitable to force the reaction to be the electrolysis of water to produce oxygen at the anode and hydrogen at the cathode.

4.1.1 The ionic concentrations shall be suitable to provide sufficient conductivity in the solution to avoid excessive voltage requirements for the power supply.

4.1.2 Some suitable electrolytes are:

- 1 M sulfuric acid (H_2SO_4)
- 1 M sodium sulfate (Na_2SO_4)
- 180 g/L sodium sulfate with 0.1 N sulfuric acid to maintain pH at 1.

4.1.3 Suitable electrolytes should not contain chlorides. The presence of chlorides results in chlorine gas being generated in lieu of oxygen at the anode.

4.1.3.1 Chlorine gas poses handling and safety concerns during testing.

4.1.3.2 Catalyzed titanium anodes are much more severely tested for lifetime by oxygen generation than chlorine generation.

4.1.4 Fresh electrolyte shall be used for each test.

4.1.5 The concentration shall be maintained at $\pm 5\%$ of the specified target throughout the test.

4.1.6 The temperature of the electrolyte shall be maintained at $30 \pm 5^\circ\text{C}$ ($86 \pm 9^\circ\text{F}$).

Section 5: Test Procedure

5.1 The test shall be conducted in a well-ventilated laboratory fume hood. The gas released from the cell is a potentially explosive mixture of hydrogen and oxygen and must be well ventilated.

5.2 The test cell shall be filled with electrolyte and agitated with a magnetic stirrer or similar means of ensuring good mixing.

5.3 Water lost during the test shall be replaced by distilled or deionized water to maintain the electrolyte level $\pm 5\%$. At no time shall the level be less than that required to maintain the test equipment setup described in Section 3.

5.4 The power supply shall be energized, the current increased to the test setting, and the cell allowed to stabilize for three hours. Current flow shall be confirmed by the production of gas bubbles at both the anode and the cathode.

5.4.1 If the current changes more than 1% from the set point, then the test shall be stopped and the cause investigated. The test may be continued after appropriate corrective action.

5.4.2 If the cell voltage exceeds the target voltage (see Section 6, Test Completion), then the test shall be paused to allow the removal of the cell from the test station. After removal of the failed cell from the circuit, the test may be started again so that the remaining samples can be tested to completion.

5.4.3 Appropriate adjustments shall be made to the recorded time to ensure the time to failure is accurate for each sample.

5.4.3.1 The temperature of the electrolyte shall be controlled to $30 \pm 5^\circ\text{C}$ ($86 \pm 9^\circ\text{F}$). A bath or jacketed beaker may be necessary to maintain the temperature depending on ambient conditions and the operating voltage of the cell.

5.4.3.2 The cell current and voltage shall be monitored and recorded during the test hourly.

5.4.3.3 The test duration should include the three hours for assuring stabilization of cell operation before recording of voltage.

Section 6: Test Completion

6.1 Anode failure is marked by a rapid escalation in cell voltage. The specific cell voltage is dependent on the selected current density and test conditions. However, the voltage rise from the starting voltage (starting voltage is the cell voltage three hours after the start) should not exceed 1.5 V. When the cell voltage rise exceeds this, then the test is complete and the time to failure should be recorded.

6.2 It should be verified that the voltage rise is not the result of any other factor than anode failure, e.g., loose electrical connection.

Section 7: Reporting Test Results

7.1 The test method procedure should be described.

7.2 The information recorded shall include as a minimum the sample size, cell current, cell voltage, and temperature on an hourly basis.

7.2.1 If the test is to failure, the time to failure should be recorded.

7.2.2 If the test is to be compared to a correlation curve, then the test may be stopped after sufficient time has been accumulated to demonstrate that the product is satisfactory for the intended design.

7.2.3 Any visible change in anode appearance such as color, pitting, or flaking of material shall be reported.

Appendix A Discussion (Nonmandatory)

This appendix is intended to provide supplementary information only, although it may contain mandatory or recommending language in specifications or procedures that are included as examples of those that have been used successfully. Nothing in this appendix shall be construed as a requirement or recommendation with regard to any future application of this technology.

Accelerated testing is intended to provide a measure of the anode's ability to perform satisfactorily for a specific design requirement in a relatively short period of time. Normally, the design requirements for an anode of a given surface area are expressed as a current rating (amperes) for a lifetime (years).

Common design requirements for an anode in a CP system are 100 ~ 150 A/m² for 20 years. A practical test method must be able to be run in far less time. If the purpose is for testing product quality, it is preferable to have a test that could be run in minutes or hours, but this is usually not possible.

The test method shortens the time required for the test by increasing the current on the anode during the test. The simplest approach would increase the current so that the product of the current and time remained constant. This assumes a linear relationship between current and time.

Example:

If an anode rated for 10 A for 20 years were to be tested in 24 hours, then the current would need to be 73,000 A.

There are two problems with this approach.

First, the rectifier is far larger than practical for a test station. An alternative is to either sacrifice an anode by cutting a smaller sample from it or to produce a test coupon. In both cases, the surface area is much smaller than the anode and the test currents are much smaller. This test coupon can be related to the specific anode in question by using the current density or current rating divided by the anode surface area.

Second, the high current density on the anode imparts a higher stress and causes a different failure mechanism than in normal operation, resulting in a lifetime that is shorter than would be predicted from a linear relationship. Typically, these correlations between current density and lifetime are best fitted by a log-log relationship for catalyzed titanium anodes.

Typically, the accelerated test duration used in this test method are from 50 to 4,500 hours, depending on many factors including test purpose and the particular product being tested.

Appendix B Development of a Correlation Curve (Nonmandatory)

This appendix is intended to provide supplementary information only, although it may contain mandatory or recommending language in specifications or procedures that are included as examples of those that have been used successfully. Nothing in this appendix shall be construed as a requirement or recommendation with regard to any future application of this technology.

This test method may be used to construct a correlation curve by generating a log-log plot of lifetime vs. current density. The data should be developed using a test methodology outlined in this test method. The data set is comprised of the time to failure of the test and the associated current density. The data set should be comprised of as many data points as can be economically justified to enhance the confidence of the results. The data set can be plotted in software and a trend line generated. The y-axis should be log life and the x-axis should be log current density. A power relationship should be assumed. A sample chart is provided below for clarity. This chart is artificial and represents no manufacturer's data.

Some software is capable of charting a least squares fit of the data and generates an equation relating the life and the current density. See Figure B1.

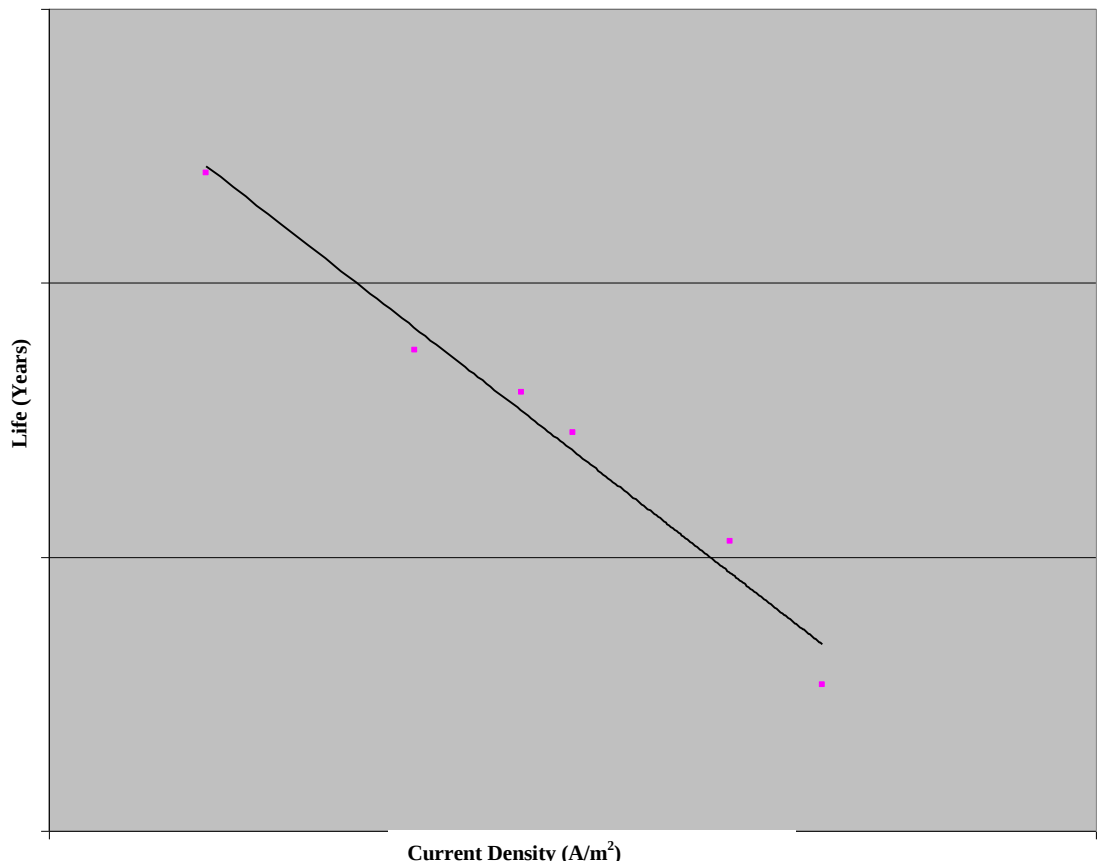


Figure B1: Correlation chart of test data.

The formula for Equation (B1) is:

$$\text{Life} = \text{Constant1} * (\text{Current Density})^{\text{constant2}} \quad (\text{B1})$$

Where:

Life = years

Current density = A/m²

Appendix C Development of Acceptance Criterion (Nonmandatory)

This appendix is intended to provide supplementary information only, although it may contain mandatory or recommending language in specifications or procedures that are included as examples of those that have been used successfully. Nothing in this appendix shall be construed as a requirement or recommendation with regard to any future application of this technology.

Note that this is not the only method that may be used, but it does describe a logical use of the information.

The measured result of this test method may be used to project the anode performance with the use of an appropriate correlation curve. To develop a correlation curve, refer to Appendix B.

The design requirements of the anode shall be converted to the units used on the correlation chart. Ideally, the selected correlation curve has the current density and lifetime of the project design requirements as one point on the curve. However, if the design requirements are reasonably close to the correlation curve, the chart can still be used to approximate the test criterion. See Figure C1.

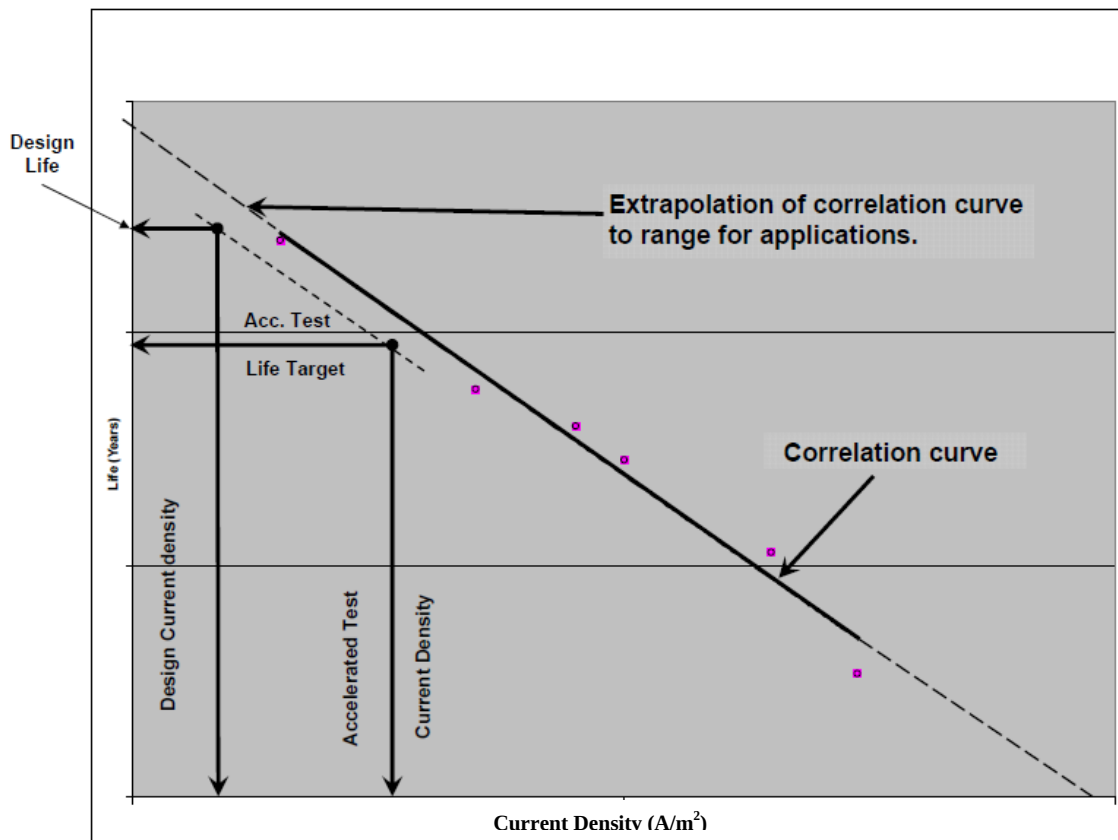


Figure C1: Design requirements—correlation chart of test data.

Sometimes, instructive comments are made regarding the work completed at this point.

First, the line generated from the data set used to generate the correlation curve is depicted as a solid line. The application of the test method assumes that this line is essentially straight when plotted on log-log charts. The dashed line extending the line represents the extrapolation of the data that are used to reach the range more typical for CP. This is to the left of the solid correlation line. Practically, the extrapolation is from a time period of less than a year of testing to as long as the design requirements are. Often, the design requirement is 20 years or longer.

Secondly, the design requirements are located just below and to the left of the correlation curve. This typically indicates that the correlation curve for the product is slightly better than the project requirements. If the design requirement is above or to the right of the curve, then the product under consideration is not considered suitable.

If the design requirements point does not fall exactly on the correlation curve line, then a line parallel to the correlation curve and containing the design requirements point is drawn. This is sometimes called the “application line.” Using this application line, the accelerated test current density shall be selected and the corresponding lifetime noted. The user moves along the application line to the right and down to the selected point on the application line that corresponds to the desired life for the test. The associated current density is the test current density.

The time-to-failure of the test sample shall be compared to the required lifetime at the accelerated test conditions. Time-to-failure of the sample shall be equal to or longer than the lifetime predicted by the application line (that represents satisfactory design requirements). In other words, if the time-to-failure falls into the region on the chart that is to the right or above the application line, the sample can be considered as passing. This comparison must be made for the selected accelerated current density. The chart below depicts a “pass” region above the application line in a diagonal pattern and the “fail” region below the application line as solid gray. The entire region is shown because the user may select any point on the line. The test sample would pass as long as the life in the test was equal to or greater than the predicted life determined by the intersection of the line and the vertical line representing the accelerated test.

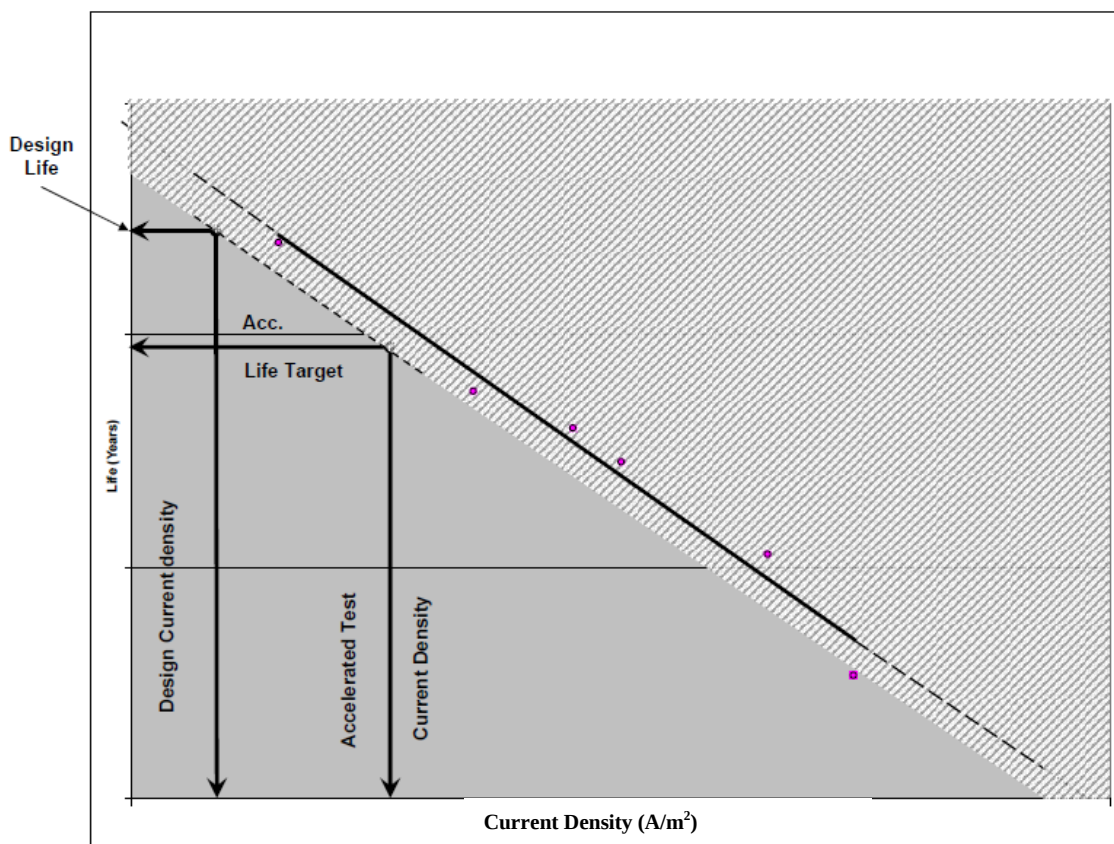


Figure C2: Sample test result evaluation using correlation chart.