INTERNATIONAL STANDARD

ISO 19097-2

First edition
2018-02

Accelerated life test method of mixed metal oxide anodes for cathodic protection —

Part 2: Application in soils and natural waters

Méthode d'essai accéléré de durabilité des anodes à oxydes métalliques mixtes pour la protection cathodique —

Partie 2: Application dans les sols et aux naturelles

Reference number
ISO 19097-2:2018(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Published in Switzerland

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

A list of all the parts in the ISO 19097 series can be found on the ISO website.

Introduction

Impressed current cathodic protection (ICCP) is an effective method to control corrosion of metallic structures immersed in seawater, brackish water, and fresh water or buried in soil. ICCP is also widely applied to prevent steel reinforcement in concrete from corrosion in marine or other chloride contaminated environment.

Mixed metal oxide (MMO) anodes have been widely taken as impressed current anodes in the ICCP system due to their good electrocatalytic activity, low consumption rate, long service life, light weight, malleability, high ratio of performance to cost, and wide suitability for different electrolytes.

Durability is one of the most important properties of MMO anodes. ISO 19097 (all parts) examines the accelerated life test method of MMO anodes for cathodic protection to evaluate the anode's ability to achieve an expected lifetime. The accelerated lifetime can also be used to compare the stability of different MMO anodes. This document is applicable to MMO anodes intended for use in underground or underwater environments. This method is not intended to give the exact service life of the anodes in the practical applications, but to provide users and manufacturers of MMO anodes a way to evaluate whether the designed life expectancy can be achieved.

Large portions of this document were derived from NACE International Standard TM0108-2012[1].

Accelerated life test method of mixed metal oxide anodes for cathodic protection —

Part 2: Application in soils and natural waters

1 Scope

This document specifies accelerated life test method of mixed metal oxide anodes for impressed current cathodic protection used in soil or natural waters. The accelerated life test results can be used to compare the durability of the anodes and to evaluate whether the anodes can comply with required specifications of design life expectancy at rated current output.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 8044, *Corrosion of metals and alloys — Basic terms and definitions*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8044 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

3.1
mixed metal oxide anode
impressed current anode for cathodic protection consisting of conductive coating of mixed metal oxides formed on titanium substrate

Note 1 to entry: The most common mixture used for cathodic protection is iridium oxide and tantalum oxide. Exact compositions may vary.

3.2
accelerated life
lifetime of *mixed metal oxide anode* (3.1) under accelerated testing condition, usually in the specific electrolyte applied with large current density

Note 1 to entry: The total period of testing until the deactivation of the mixed metal oxide anode is taken as the accelerated life.

3.3
cell voltage
voltage between anode and cathode in a single cell

4 Test method

4.1 Principle

The accelerated life testing of mixed metal oxide anode is conducted in specific simulated environments at a much higher current density than experienced in usual working conditions. This can shorten the period of time to deactivation of the anode significantly.

4.2 Test solution

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

4.2.2 Some suitable electrolytes are as follows:

— 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.

4.2.3 Suitable electrolytes should not contain chlorides. The presence of chlorides results in chlorine gas being generated in lieu of oxygen at the anode. Chlorine gas poses handling and safety concerns during testing. Catalysed titanium anodes are much more severely tested for lifetime by oxygen generation than chlorine generation.

4.2.4 Fresh electrolyte shall be used for each test.

4.2.5 The concentration shall be maintained at ±5 % of the specified target throughout the test.

4.2.6 The temperature of the electrolyte shall be maintained at 30 °C ± 5 °C.

4.3 Test apparatus

4.3.1 The test equipment comprises 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.

4.3.2 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.

4.3.3 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 from the liquid level and the bottom of the beaker, respectively.

4.3.4 The anode-to-cathode gap should be approximately 20 mm.

4.3.5 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 mm^2. This wire shall be connected to the negative pole of the power supply.

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

4.3.7 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 mm^2 sample being run at 10 kA/m^2. One power supply can also energize several electrolytic cells connected in series at the same time. The required voltage depends on the number of test stations in series that may be used. Typically, 8 V per cell times the number of cells in series is sufficient.

4.3.8 Use data acquisition device or voltmeter to monitor the voltage of each cell continuously until the failure of the anode. The voltage measuring device shall have a high input impedance of 10 MΩ or greater, and be capable of measuring cell voltage accurate to ±1 %.

4.3.9 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.

NOTE A schematic diagram of test apparatus is shown in Annex A.

4.4 Test specimens

4.4.1 The number and type of test specimens shall be selected according to the specifications for the anode material or product being tested. To ensure the accuracy of the data, usually three, but at least two, duplicate specimens of each anode shall be tested in the separate cells under identical operating conditions.

4.4.2 The test specimens shall be carefully cleaned prior to testing so as to remove those traces (dirt, oil or other foreign matter) that could influence the result.

Care shall be taken that specimens are not contaminated after cleaning by careless handling.

4.5 Test procedure

4.5.1 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 shall be well ventilated.

4.5.2 The test cell shall be filled with electrolyte, making sure that the anode sample is completely immersed in the solution, and agitated with a magnetic stirrer or similar means of ensuring good mixing.

4.5.3 Water lost during the test shall be replaced by distilled or deionized water to maintain the electrolyte level ±5 %. At no time shall the level be less than that required to maintain the test equipment setup.

4.5.4 The power supply shall be energized, the current increased to the test setting, and the cell allowed to stabilize for 3 h. Current flow shall be confirmed by the production of gas bubbles at both the anode and the cathode.

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.

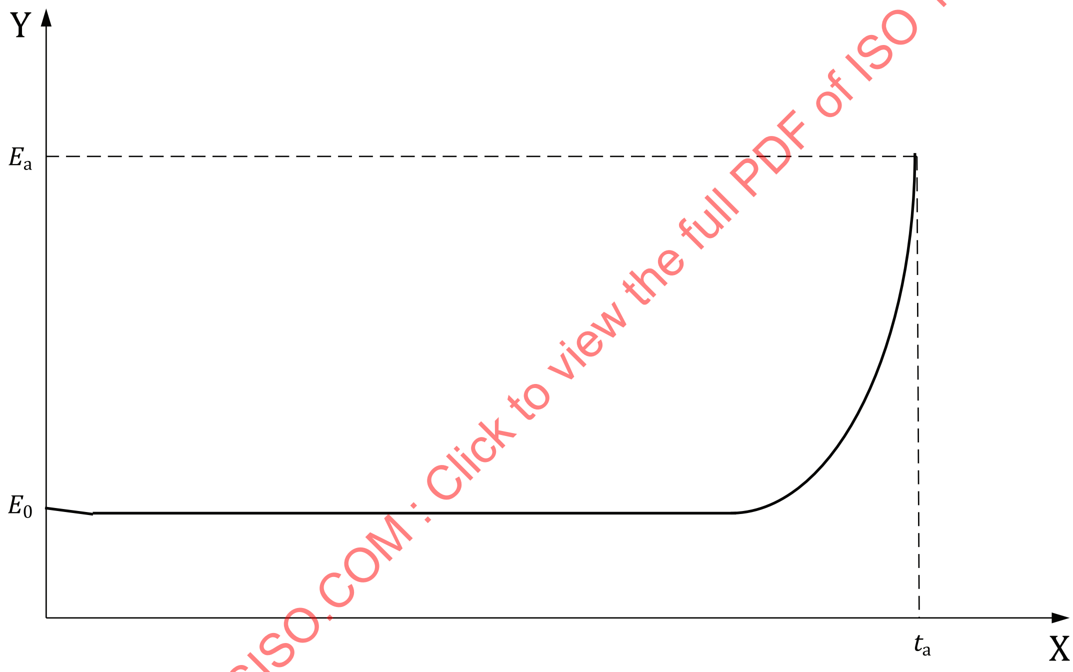
If the cell voltage exceeds the target voltage, then the test shall be paused to allow the removal of the cell from the test station. After removal of the failed cell from the circuit, the test may be started again so that the remaining samples can be tested to completion.

4.5.5 The temperature of the test solution shall be kept at 30 °C ± 5 °C through the testing period.

4.5.6 The cell current and voltage shall be monitored and recorded during the test hourly. The test duration should include the 3 h for assuring stabilization of cell operation before recording of voltage.

4.5.7 A typical curve of cell voltage with electrolysis time is shown in Figure 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. The test is stopped when the testing anode sample is failed.

4.5.8 When the cell voltage E_a is 1,5 V higher than the starting cell voltage E_0 (starting voltage is the cell voltage 3 h after test), the test anode can be taken as failed. The period from the beginning of the test to the failure of the anode is the accelerated life time t_a (see Figure 1).



Key

X electrolysis time

Y cell voltage

Figure 1 — Typical curve of cell voltage against electrolysis time

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

4.5.10 A reliable power supply shall be used to make sure that current fluctuations and power outages are avoided during the test.

4.5.11 If the test is to be compared to a correlation curve (see Annex B), then the test may be stopped after sufficient time has passed to demonstrate that the product is satisfactory for the intended design.

4.6 Continuity of tests

Tests shall preferably be continued without interruption throughout the test duration. When it is necessary to interrupt the operation for the removal of specimens for inspection, the interruption time shall be minimized.

If a longer period of interruption is unavoidable, the test specimens shall be taken out of the electrolyte and dried as soon as the test is interrupted. The test specimens should be kept in desiccator until the test restarts.

4.7 Treatment of specimens after test

At the end of the test period, the test specimens shall be removed from the electrolyte, washed with distilled or deionized water and dried.

5 Reporting test results

Date, time, applied current and cell voltage shall be reported in tabular form for each anode sample. The interruption and replacement of solution shall be remarked. The variation of cell voltage with electrolysis time can also be presented in a graph (see Figure 1).

Any other relevant information, such as change of electrolyte colour, photographic records or descriptions of the appearance of tested specimens, shall also be recorded.

The shape and dimensions of the anode specimens shall be described. The material, shape and dimensions of the cathode should also be reported. Any deviation from this standard testing method shall be documented in the report.

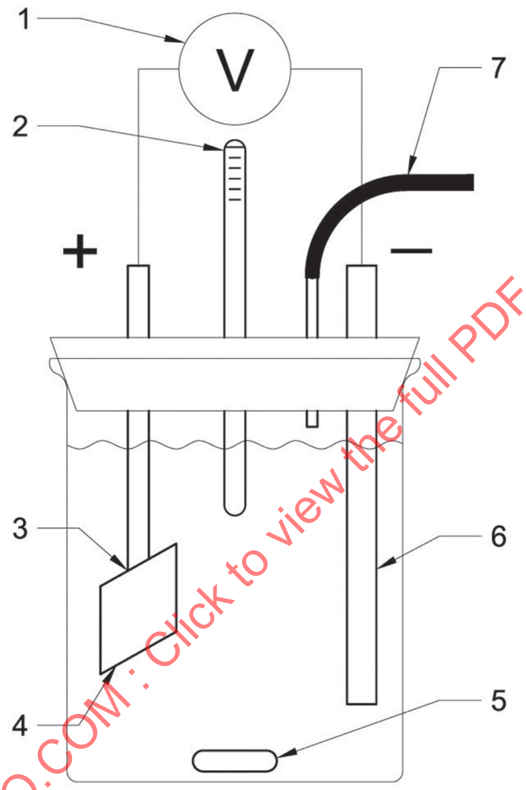
6 Application of results

The test results can be used for comparison of anode stability between different anodes under the same specified test conditions. The accelerated life test results can also be used to evaluate if the anode can meet the design requirements of life expectancy at the specified current density, as shown in Annex B. The accelerated life test produces a conservative result due to the more severe stressing on the anode than that at normal application.

Annex A
(informative)

Typical apparatus for accelerated life test

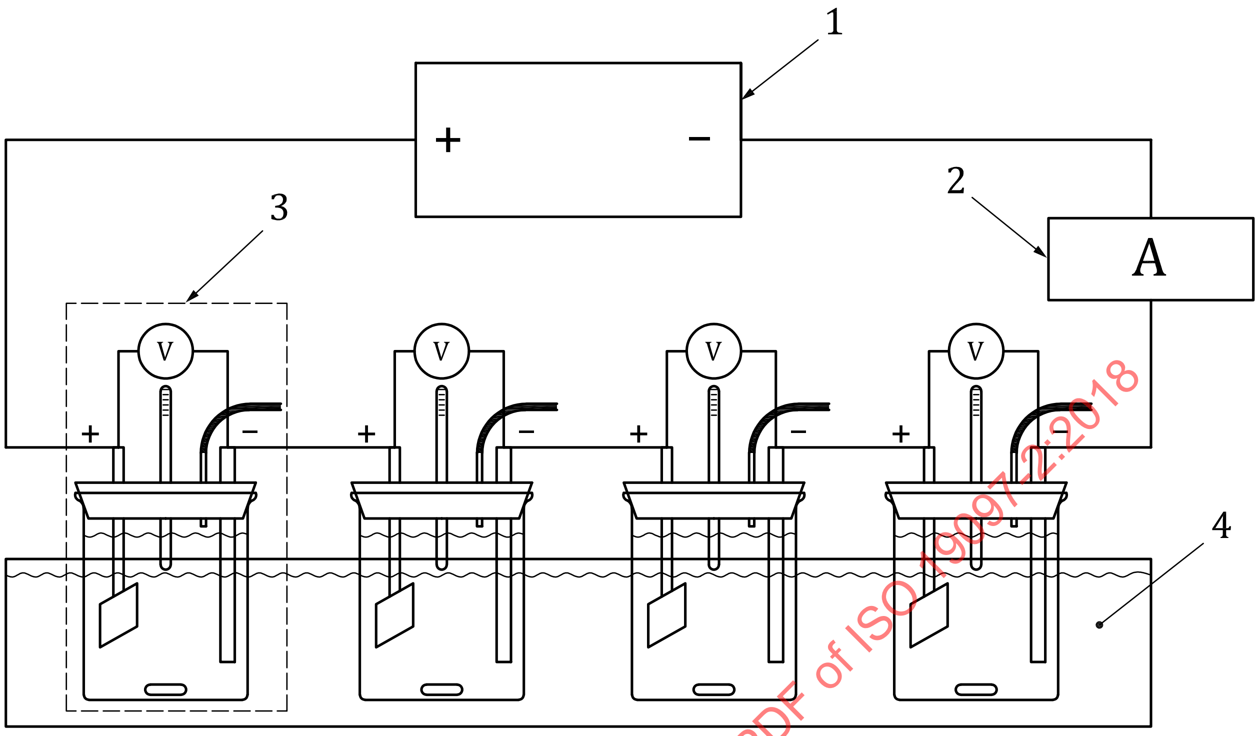
A schematic diagram of the typical test apparatus for the accelerated life testing is shown in Figures A.1 and A.2.



Key

1 data collector or voltmeter
2 thermometer
3 weld
4 test anode
5 magnetic stirrer
6 cathode
7 vent

Figure A.1 — A typical cell for the accelerated life test



Key

1 constant current power supply
2 current recorder
3 single testing cell
4 constant temperature water bath

Figure A.2 — Test cells connected in series

Annex B
(informative)

The service life evaluation of metal oxide anode for cathodic protection

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).

The relationship between anode life L and anode current density i can be established for mixed metal oxide anodes. Generally, the log L is found to be linear to log i as shown in Figure B.1, which can be expressed by Formula (B.1):

$$\log L = \mathrm{A} - \mathrm{B} \log i \tag{B.1}$$

where

L is the anode life by years;

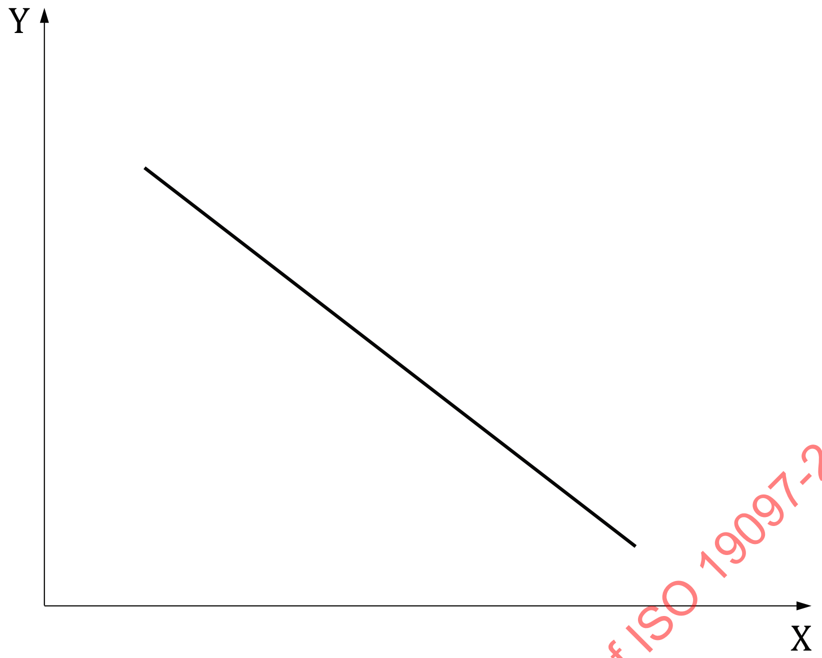
i is the current density by $\mathrm{A/m^2}$;

A and B are constants dependent on the anode materials and the test conditions.

Using the methodology outlined in this accelerated life test method, a data set including anode life and current density can be acquired, which can be used to fit the relation in Formula (B.1) and to develop a correlation curve. More data as possible should be considered to enhance the confidence of fitting result.

The fitting line can be extrapolated to the range of working current density. If the design requirements on anode current density and service life are below (to the left of the curve) or on the fitting line, then the mixed metal oxide anode can be considered to be suitable (see Figure B.2).

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



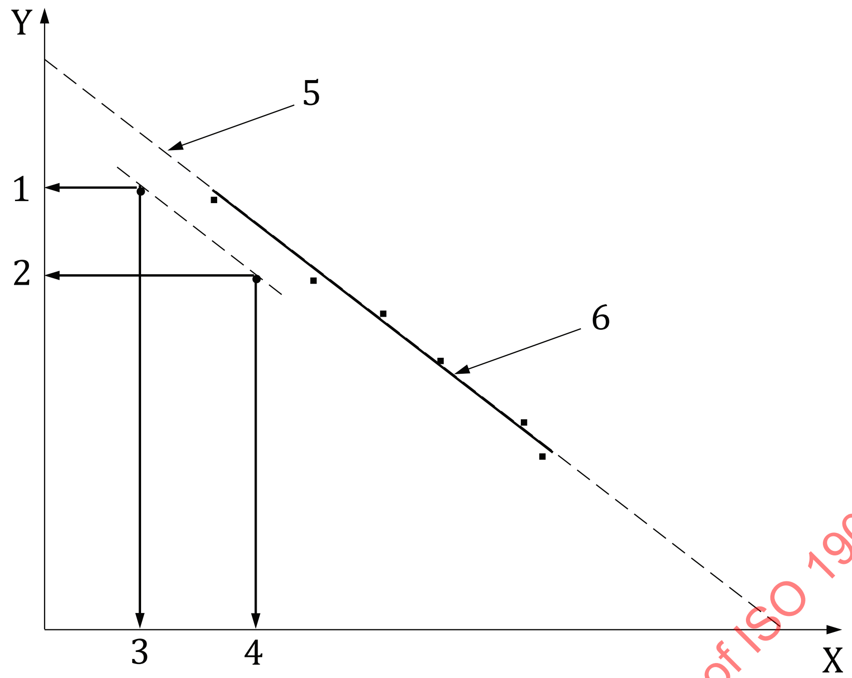
Key

X log *i* current density (A/m^2)

Y log *L* life (years)

Figure B.1 — The typical curve of anode life *L* vs. current density *i*

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.



Key

X log *i* current density (A/m^2)

Y log *L* life (years)

1 design life

2 accelerated test life target

3 design current density

4 accelerated test current density

5 extrapolation of correlation curve to range for application

6 correlation curve

Figure B.2 — Design requirements - correlation chart of test data

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 shall be made for the selected accelerated current density. Figure B.3 depicts a pass region above the application line in a diagonal pattern and the fail region below the application line as solid grey. 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.

