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Corrosion of metals and alloys — Measurement of the electrochemical critical localized corrosion temperature (E-CLCT) for Ti alloys fabricated via the additive manufacturing method

Corrosion des métaux et alliages — Mesurage de la température critique de la corrosion localisée électrochimique pour les alliages de Ti fabriqués à l'aide d'une méthode de fabrication additive

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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

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Introduction

Ti alloys such as Ti-6Al-4V are considered the most promising engineering materials. Due to a unique combination of high strength-to-density ratio and increased mechanical and corrosion resistance, their applications are growing in a wide range of industries, e.g. aerospace, automobile, marine and biomedical fields.

Ti alloys are conventionally produced by wrought or cast processes, which are subtractive manufacturing (SM) methods. The recent emergence of a new additive manufacturing (AM) method known as “3D printing” has gained worldwide attention as a way to cut costs and improve efficiency for small quantity, batch productions.

Additively manufactured Ti alloys are extensively investigated for their usage in aerospace and medical applications. When AM is compared with conventional manufacturing, the buy-to-fly ratio is known to be around 15:1 (conventional). In terms of mechanical viewpoints, both the strength and ductility of Ti alloys such as Ti-6Al-4V fabricated via AM are comparable to or superior to those developed via conventional manufacturing methods, because of their unique microstructure based on laser or electron beam technologies. However, the characteristics of additively manufactured alloys are highly dependent upon the geometric and processing conditions (and there are over 130 variables) such as layer formation (imbedded or sprayed), size and quality of powder or wire, dimension, input energy, layer orientation and surface conditions, and tolerance in the CAD process, which converts the data into additive layers for building parts. The differences in layer orientation and the porosity generated by crossing hatches during the layer-by-layer fabrication process can result in differences in both mechanical and electrochemical properties in AM materials. Heat treatment controls the porosity or the microstructure derived from rapid melting and quenching; however, it cannot eliminate interlayers, which contribute to the differences in the mechanism of localized corrosion in AM materials. The resistance to corrosion of Ti alloys produced via AM is similar to that of conventionally manufactured Ti alloys. The mechanisms of corrosion also differ. Therefore, since the conventional testing methods have shown limited ability for evaluation of those properties, the new test method measuring electrochemical critical localized corrosion temperature (E-CLCT) has been developed to evaluate pitting and crevice corrosion in alloys generated via AM. E-CLCT is defined as the lowest temperature on the surface of the AM specimen on which localized corrosion to both pitting and crevice corrosion is initiated under specified test conditions.

This document specifies a procedure for evaluation of the resistance to localized corrosion on the AM alloys by measuring their E-CLCT, providing an efficient method for a qualitative evaluation or comparison of corrosion properties between AM materials or their heats with altered process variables. This test method demonstrates the quality of heat treatment, bonding integrities between layers, and effective control of variables for AM materials, providing a qualitative tool for long-term application. Furthermore, this document can extend its use from AM Ti-alloys to other AM alloys, such as Ni alloys by modifying the concentration of test solutions or the applied potentials. This document also provides important clues to evaluate other types of localized corrosion such as corrosion cracking and erosion-corrosion. Related documents can be developed and followed up based on the results of this test.

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Corrosion of metals and alloys — Measurement of the electrochemical critical localized corrosion temperature (E-CLCT) for Ti alloys fabricated via the additive manufacturing method

1 Scope

This document specifies procedures for testing the resistance to localized corrosion of Ti alloys fabricated via additive manufacturing (AM) method. This document regulates the electrochemical critical localized corrosion temperature (E-CLCT) of the AM Ti materials for a comparative evaluation of resistance to localized corrosion.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

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

3.1
electrochemical critical localized corrosion temperature
E-CLCT
lowest temperature on the surface of the AM Ti alloy specimen at which stable localized corrosion, including both pitting and crevice corrosion, occurs under specified test conditions

3.2
temperature ramp rate
rate at which the surface temperature of the specimen increases during the test

4 Main contents and limitations of existing standards

4.1 Application coverage of ISO 17864

The test method in ISO 17864 determines the critical pitting temperature (CPT) using a potentiostatic technique with a temperature scan. The current is monitored during the temperature scan, and CPT is defined as the temperature at which the current increases rapidly, which for practical reasons is defined as the temperature at which the current density exceeds 100 μA/cm^2 for 60 s. Pitting on the specimen is confirmed visually after the test.

4.2 Limitations of ISO 17864

ISO 17864 is useful to measure the resistance of pitting corrosion for stainless steel and related alloys. This method applies to wrought or cast products. However, this method cannot be used for Ti alloys

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fabricated via AM, which are superior to stainless steels in terms of resistance to pitting, and fabricated via SM. Therefore, it requires much higher potential and more aggressive corrosion environment.

4.3 Scope of ISO 18089

The test method in ISO 18089 determines the critical crevice temperature (CCT) via a potentiostatic technique using a temperature scan. The current is monitored during the temperature scan. CCT is defined as the temperature of a specimen beneath the crevice former at which the current increases rapidly. Crevice corrosion on the specimen is confirmed visually after the test.

4.4 Limitations of ISO 18089

This document is useful for the measurement of resistance to crevice corrosion of stainless steel and related alloys and this method applies to wrought or cast products. However, this method cannot be used for Ti alloys fabricated by AM where crevice corrosion does not occur on the specimen surface beneath the crevice former.

5 Principle

This test method generally describes laboratory tests for the measurement the E-CLCT of AM Ti alloys, based on ISO 17864 and ISO 18098. Therefore, test temperature sweep rate, basic apparatus and test procedures are similar to those standards, except for the concentrated corrosive aqueous test solution, much higher applied potentials and the Ti-alloy specimens fabricated by AM. The resistance to localized corrosion of AM Ti alloys shall be evaluated in terms of initiation of pitting or crevice corrosion or both, because of much higher pore density and varying orientation compared with those of conventional wrought alloys. In ISO 18098, CCT is determined by using a crevice former, which is the component of the crevice corrosion test tool assembly, and which allows crevice corrosion to be induced in a contacted test specimen by applying certain torques. However, in the case of AM Ti alloys, pits or crevices are induced from the pores or the edges of the specimen rather than the locations under the crevice former. Therefore, the measurement of E-CLCT does not require the crevice former, i.e. the component of the crevice corrosion test tool assembly. The flushed-port cell for CPT test is not adequate, in which the specimen is separated from the cell port and is mounted outside the cell. The whole specimen shall be immersed in the solution inside the cell, ensuring exposure of specimen parts to solution. Samples exposed to the concentrated sodium chloride (a mass fraction of 25 %) solution, are tested with a potentiostatic technique using a temperature scan. The anodic applied potential is held constant during the whole temperature scan. The current is monitored during the temperature scan. The E-CLCT shall be measured up to the temperature where the current increases rapidly as shown in Figure 1. Figure 2 shows two different shapes of crevice corrosion on Ti-6Al-4V alloys obtained via SM and AM. The different initiation sites are noted in localized corrosion between SM and AM Ti-6Al-4V alloys.

NOTE 1 Some CPT values can be found in Reference [4].

NOTE 2 Some E-CLCT values can be found in Reference [5].

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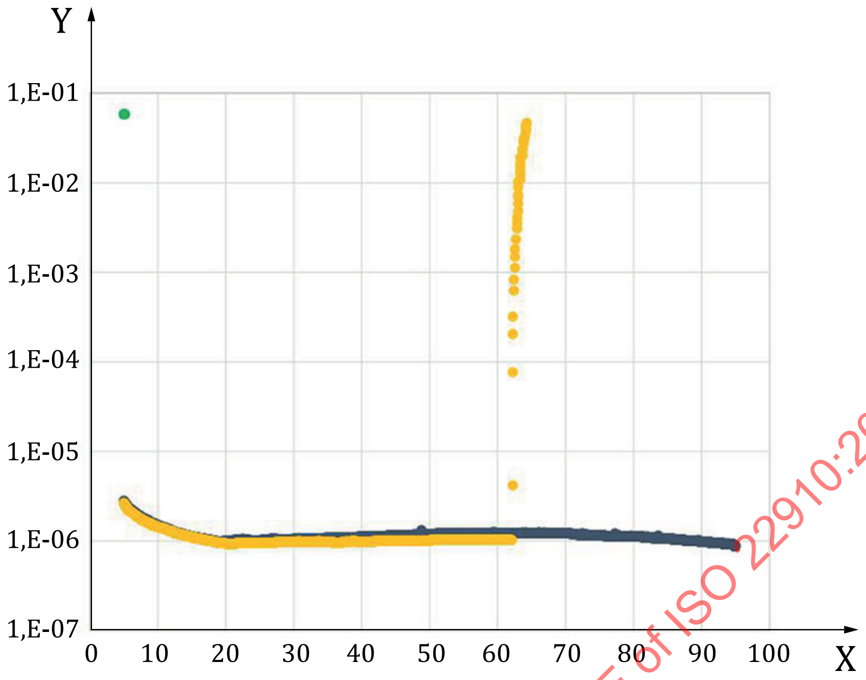

Key

X temperature (in °C)

Y current density (in A/cm^2)

● AM Cp-Ti

● AM Ti-6AI-4V

● AM Ni7 18

Figure 1 — Determination of E-CLCT values of Ti-6Al-4V, CP-Ti, and Ni718 for the side edge parallel to the AM direction produced by additive manufacturing, according to applied potentials of 2,8 V with respect to saturated calomel electrode in the concentrated sodium chloride (mass fraction of 25 %) solution

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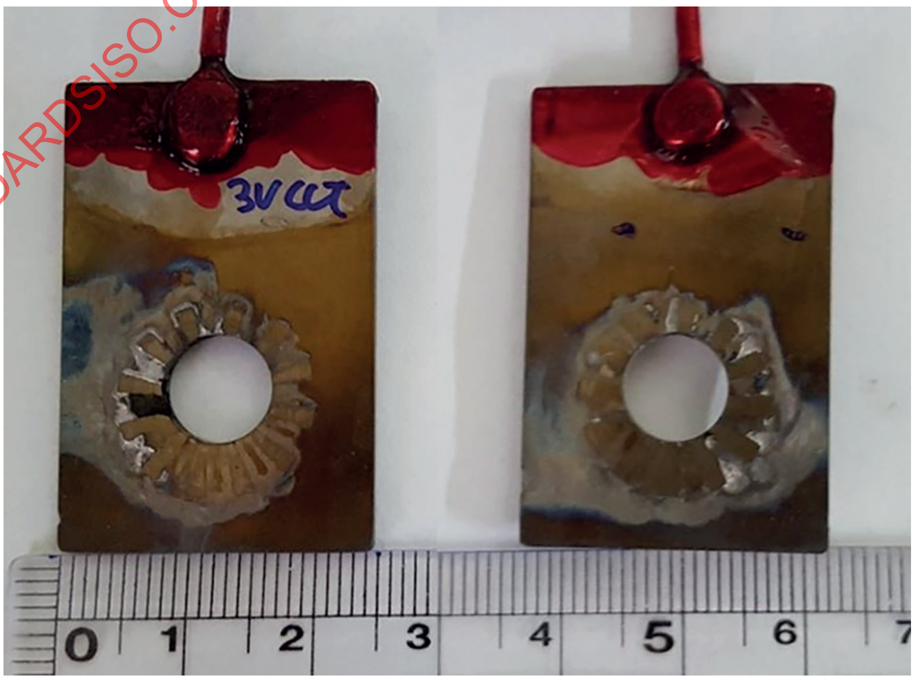
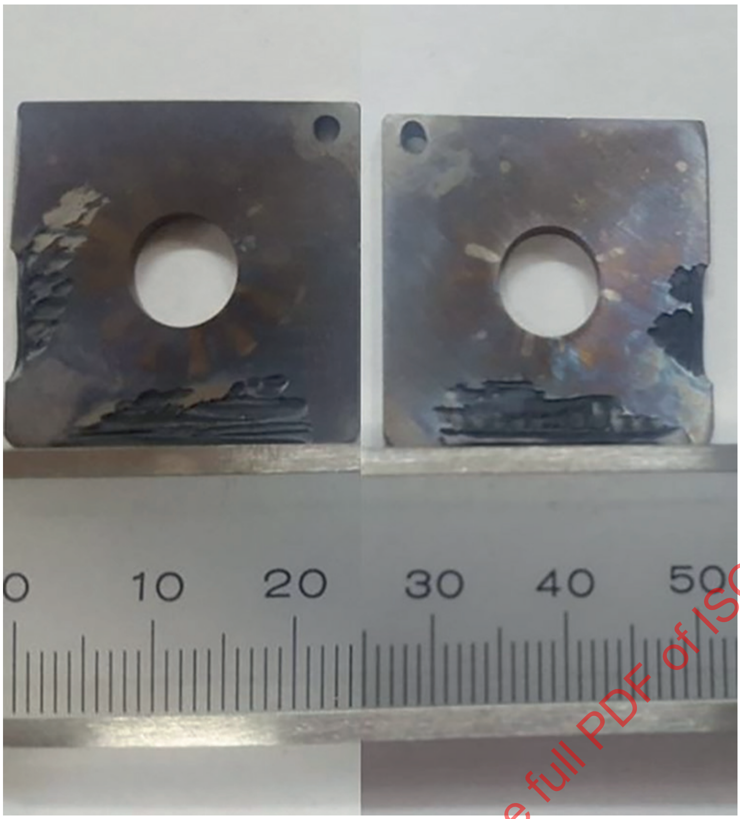


a) Subtractive-manufactured Ti-6Al-4V alloys



b) Additive-manufactured Ti-6Al-4V alloys

Figure 2 — Two different shapes of crevice corrosion on Ti-6Al-4V alloys

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6 Apparatus

6.1 Potentiostat.

The potentiostat shall be capable of controlling the electrode potential to within ±1 mV of a preset value.

This potentiostat should have the electrode potential-measuring instrument with a high impedance of the order 10^{11} Ω to 10^{14} Ω and the current-measuring instruments capable of measuring current to within 2 % of the actual value.

6.2 Recirculating heating bath with temperature controller.

The recirculating heating bath is a vessel capable of generating temperatures between 0 °C and 100 °C ± 1 °C.

The temperature controller should facilitate increase in the surface temperature of the specimen from 0 °C to 100 °C with the temperature ramp rate of 1 °C/min.

6.3 Specimen holder and connections.

Figure 3 shows the schematic design on the specimen holder and connection. Any part of the specimen holder or the connection of the electrode shall be designed to avoid corrosion in the connecting point with resin seal or a similar coating.

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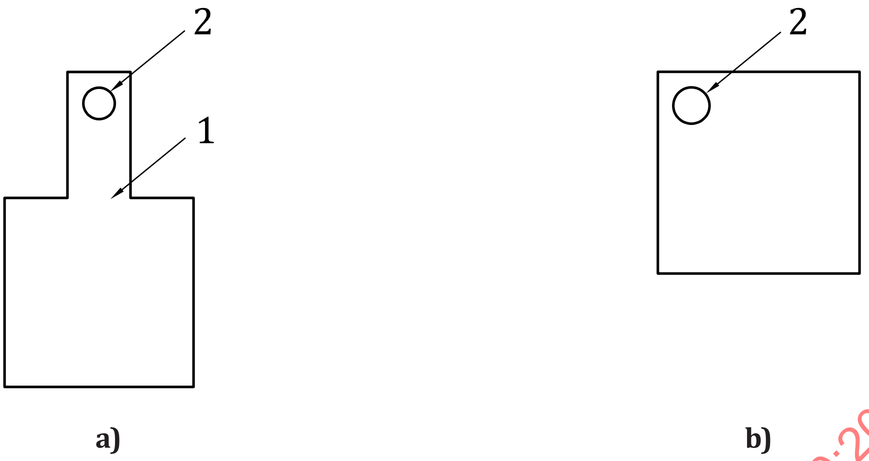


Key

1 line of solution

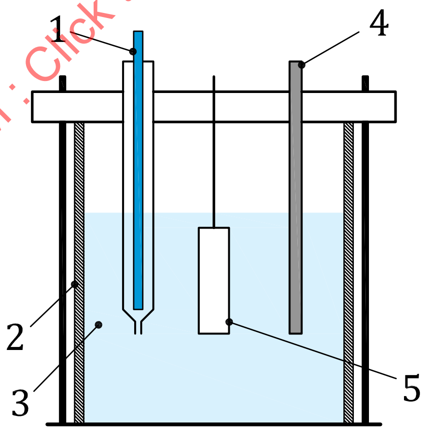
2 electric connection

The crevice former shall not be placed.

Figure 3 — Schematic diagram of a specimen

6.4 Test cell.

The test cell shall contain the test specimen, a Luggin capillary probe connected to an external reference electrode to measure the electrode potential, an auxiliary electrode, a port for insertion of a temperature-measuring device and a facility for stirring the solution by bubbling gas through the solution, as shown in Figure 4.



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Key

1	reference electrode	4	counter electrode
2	double wall	5	specimen
3	electrolyte		

Figure 4 — Schematic diagram of CLCT cell

6.5 Auxiliary electrode and reference electrode.

The auxiliary electrode is prepared from high-purity platinum. The reference electrode shall be maintained at ambient temperature external to the test cell and connected to the test cell via a Luggin capillary probe. The commonly used reference electrode is the saturated calomel electrode.

7 Test solutions

For the test, 250 g of reagent-grade sodium chloride (NaCl) is dissolved in 750 ml of reagent water, producing an aqueous solution containing 25 % NaCl by mass. The ratio of the volume of solution in the test cell to the specimen area shall be at least 100 ml/cm^2.

8 Test specimens

Any specimen geometry compatible with the specimen holder may be used. Two different test surfaces of the specimen are used, which are made by the layers perpendicular to or parallel to the stacking direction, depending on the stacking direction via AM. A minimum test volume of 10 mm × 10 mm × 1 mm or higher shall be used. A less than 20 % ratio of edge area to total area is desirable.

9 Procedure

9.1 Preparation of reference electrodes

The difference in potential between the reference electrode and two other validation electrodes shall be measured. If the potential difference is greater than 3 mV, the test electrodes shall be rejected.

9.2 Preparation of Ti alloy specimen

The specimen shall be prepared to ensure a reproducible surface finish.

The specimen shall be cleaned immediately prior to immersion in the solution by degreasing, rinsing in high-purity water, followed by ethanol or a similar solvent, and air drying. After degreasing, care shall be taken not to contaminate the test surface of the specimen.

9.3 Preparation of solution

The solution shall be prepared using reagent-grade chemicals and reagent-grade water purified to 20 µS/cm by distillation equivalent to type I grade (see ISO 3696).

9.4 Setting up the E-CLCT test

The exposed surface area of the specimen shall be measured. Two different test surfaces of the specimen are prepared, comprising layers perpendicular to or parallel to the stacking direction, depending on the stacking direction via AM.

The specimen, counter electrode and salt bridge shall be placed in the test cell. The test cell shall be filled with the solution. It is important to ensure that the specimen is immersed and exposed in the solution. The salt bridge is filled with test solution and is free of air bubbles.

The solution shall be stirred continuously throughout the test by bubbling an inert gas through the solution at a controlled rate.

The electrodes shall be connected to the potentiostat and the data-recording device. The connections for temperature measurement and control shall be made.

The open-circuit potential of the test specimen shall be recorded and the desired anodic potential shall be applied to the specimen. The recommended applied potential for Ti alloys (e.g. Ti-6Al-4V) in the concentrated sodium chloride (25 %) solution is 2,8 V with respect to the saturated calomel electrode (SCE) (25 °C). If uncertainty exists concerning whether 2,8 V_{SCE} is sufficiently high to obtain the potential independent E-CLCT, a test at 2,9 V with respect to SCE (25 °C) may be performed. A significant deviation between the E-CLCT obtained at 2,8 V_{SCE} and 2,9 V_{SCE} indicates the need for re-evaluation

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and new choice of potential (for information, refer to Annex A). Following application of potential for 60 s or longer, the specimen temperature shall be increased at a controlled rate.

The current and solution temperature shall be monitored throughout the test.

The E-CLCT is defined as the temperature at which a sharp increase in current density occurs during the temperature ramp at 1 °C/min.

9.5 Ending test

The test shall be terminated when the E-CLCT is determined.

The specimen shall be removed from the solution and rinsed in water, cleaned with ethanol, rinsed with high-purity water, cleaned with ethanol or a similar solvent, and dried in air.

The specimen shall be inspected using an optical microscope to determine pitting and crevice corrosion. The ending current density may be higher than 1 mA/cm^2 (e.g. 5 mA/cm^2) if the surface is evaluated after testing.

10 Evaluation of test results

The E-CLCT shall not be compared with values obtained using a different procedure. The E-CLCT of AM Ti alloys is specific to the test method used and should only be used as a comparative measure of performance.

Localized corrosion is generally of random nature, and thus the number of specimens required for experimental condition is preferably greater than three considering data scatter and analysis.

11 Test report

The test report shall include the following information:

a) a reference to the document used (including its year of publication);

b) the test solution and its volume;

c) a full description of the test materials from which the specimens were taken, including composition and structural condition, type of product and section thickness;

d) the orientation and size of test specimens;

e) the surface finish of specimens, including the storage time between carrying out the final surface finish and testing;

f) the test area and the minimum thickness;

g) the electrode potential;

h) the temperature ramp rate;

i) a description of the specimen surface after testing;

j) the electrochemical critical localized corrosion temperature (E-CLCT);

k) ending current density;

l) deviation of test results from the procedure;

m) unusual features observed;

n) a reference to explain the calculation of the results;

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o) the date of the test.

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