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Corrosion of metals and alloys — Requirements for localised corrosion and environmentally assisted cracking testing of additively manufactured metals and alloys

Corrosion des métaux et alliages — Exigences pour les essais de corrosion localisée et de fissuration assistée par l'environnement sur les métaux et alliages de fabrication additive

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Foreword

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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).

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For an explanation of 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 www.iso.org/iso/foreword.html.

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Additive manufacturing (AM) offers a route to rapid and delocalized end product generation, often with complex shapes, without the need for extensive machining, with the expectation of reducing cost, time and waste. For metals, AM products can be built using non-fusion based or fusion based technologies, the former being less common. The more established fusion based additive manufacturing methods are powder bed fusion, powder-feed fusion or wire-feed fusion depending on required size, speed and complexity. Powder-bed fusion tends to be used for relatively small products of complex shape and involves spreading the powder over the powder bed and melting the powder to the shape required using a programmed laser beam (under inert gas such as argon or nitrogen) or electron beam (under vacuum). The next powder layer is spread uniformly across the powder bed and the process is repeated to build up the full part. In powder-feed directed energy deposition, the powder is fed through a nozzle onto the build surface under an inert gas. The beam creates a meltpool into which powder is fed and the process is repeated layer by layer to create the desired shape. In wire-feed systems, the feedstock is wire but the energy source can be electron beam, laser beam or plasma arc under inert gas or vacuum as appropriate. Initially, a single bead of material is deposited and this is built upon in subsequent passes. Wire-feed systems are used when large build volumes are desirable.

It is important to recognize the possible challenges in deploying these products, including the inhomogeneous and graded microstructure, microstructural grain/dendrite size distribution, phase distribution, strong crystallographic texture, elemental segregation, residual stress, surface properties, shrinkage fissures, pores and anisotropic mechanical properties. Some of these factors impinge on corrosion and environmentally assisted cracking resistance (see Reference [1]) and how it is measured. Post-processing thermal treatments are commonly applied to homogenize the material and these can reduce the impact in some cases. Also, the degree to which the factors listed affect properties is highly dependent on the specific AM process and process parameters adopted, and the manufacturer's expertise and experience. The difference in the quality of the end product is expected to diminish as the technology further matures.

INTERNATIONAL STANDARD **ISO 23669:2022(E)**

Corrosion of metals and alloys — Requirements for localised corrosion and environmentally assisted cracking testing of additively manufactured metals and alloys

1 Scope

This document establishes requirements for designing tests and test specimens and conducting tests to assess susceptibility of additively manufactured metals and alloys to localized corrosion and environmentally assisted cracking in aqueous solutions.

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 6892-1, *Metallic materials — Tensile testing — Part 1: Method of test at room temperature*

ISO 6892-2, *Metallic materials — Tensile testing — Part 2: Method of test at elevated temperature*

ISO 7539-1, *Corrosion of metals and alloys — Stress corrosion testing — Part 1: General guidance on testing procedures*

ISO 7539-6, *Corrosion of metals and alloys — Stress corrosion testing — Part 6: Preparation and use of precracked specimens for tests under constant load or constant displacement*

ISO 8044, *Corrosion of metals and alloys — Vocabulary*

ISO 15158, *Corrosion of metals and alloys — Method of measuring the pitting potential for stainless steels by potentiodynamic control in sodium chloride solution*

ISO/ASTM 52900, *Additive manufacturing — General principles — Fundamentals and vocabulary*

ISO/ASTM 52921, *Standard terminology for additive manufacturing — Coordinate systems and test methodologies*

3 Terms and definitions

For the purposes of this document the terms and definitions given in ISO 7539-1, ISO 7539-6, ISO 8044, ISO/ASTM 52900 and the following apply.

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

- IEC Electropedia: available at https://www.electropedia.org/
- ISO Online browsing platform: available at https://www.iso.org/obp

3.1
additive manufacturing
AM
process of joining materials to make parts from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing and formative manufacturing methodologies

3.2
end product

product after being subject to all manufacturing steps, according to the nominal specification

3.3
coupon

part produced according to the same nominal specification as the product/component, intended to have the same characteristics and properties of the product/component, and to be used as the basis for testing either directly or after machining to the desired specimen configuration as specified in the appropriate test standard

4 Material characterization

4.1 The material characterization shall be undertaken using material removed from the processed part (coupon or end product) after post-build treatment, including any thermal treatment, machining or surface modification, in such a way that the sections examined are representative of the material that would be exposed to the environment and would be potentially susceptible to localized corrosion or environmentally assisted cracking. Specific attention should be paid to the orientation of the section removed for characterization with respect to the AM build direction and the specific location of the section in the build space, in accordance with ISO/ASTM 52921, shall be reported.

NOTE Variation in microstructure and material properties can occur within AM parts. This is associated with different thermal gradients that can be present throughout manufacture; examples are: the first layer melted compared with the last layer or a thin section compared with a large section. Another variable is the post fabrication thermal treatments which can fail to give incomplete homogenization.

4.2 Prior to specimen testing, the material microstructure shall be characterized, in accordance with the applicable metal or alloy standard where appropriate, together with the following: orientation of the microstructure with respect to the build direction; grain size and indication of any non-uniformity of grain dimensions, phases present, the presence of pores or other physical defects such as shrinkage fissures, with particular attention given to near surface and surface breaking pores and defects.

NOTE Coupons built in the X/Y orientation, according to ISO/ASTM 52921, can show a different level of porosity from those built in the Z orientation.

4.3 Measurement of the pore size distribution and volume fraction should be undertaken.

NOTE Metallographical techniques can be used for pore size distribution; for example, as described in ISO 4499-4.[2] X-ray computed tomography can be used for 3D imaging of pores and unconverted powder and volume fraction of pores can be estimated by methods such as that due to Archimedes (see Reference [3]).

4.4 The near surface residual stress shall be determined, for example, using X-ray diffraction,[4] in different orientations with respect to the build direction, before undertaking any environmentally assisted cracking test programme and compared with measurements on the related end product, where data are available. The measurements shall be made on a test specimen that represents the final material condition.

NOTE While high temperature solution anneal will tend to remove residual stress effectively if applied, lower temperature treatments can be ineffective and can form detrimental phases. Low temperature thermal treatments while reducing residual stresses can have no effect on the as-built microstructure, retaining an as-processed microstructure and chemical segregation.

4.5 The surface roughness of the test specimen shall be characterized (e.g. by profilometry, or infinite focus microscopes enabling 3D mapping) and shall be compared with the surface roughness of the product. Any post-build machining or surface treatment of the end product shall be replicated in the test specimen.

5 Tensile properties

5.1 The tensile stress-strain response of the alloy shall be determined following ISO 6892-1 or ISO 6892-2, prior to any environmentally assisted cracking test; the proof stress, ultimate tensile stress and elongation to fracture shall be quantified. Testing shall be undertaken with the orientation of the microstructure relative to the stress axis consistent with that adopted for the environmentally assisted cracking testing.

NOTE 1 Tensile specimens can be prepared directly by additive manufacturing or by machining and grinding (in accordance with the specific tensile test standard adopted) from coupons prepared by additive manufacturing (see also 4.2).

NOTE 2 Residual stress can be present in the coupon. The magnitude of the residual stress will be dependent on processing parameters and post-build treatment, which can include stress relief treatment, full solution anneal and ageing treatments.

NOTE 3 Macro tensile and compressive residual stress are balanced in the specimen and cannot affect the average mechanical properties but differential yielding within the material can result in a serrated response in the stress-strain curve.

6 Localized corrosion testing

6.1 For susceptible alloys, such as austenitic stainless steels, a preliminary assessment of sensitization due to elemental segregation during processing should be undertaken using standardised methods such as the double-loop electrochemical potentiokinetic reactivation test (see ISO 12732[5]) or methods based on etching such as ISO 3651-1[6].

6.2 Tests for sensitization should be carried out with a surface finishing procedure corresponding to the end product. However, to examine variations between the bulk and surface, additional testing should be conducted with a ground finish, compatible with that specified in the adopted test standard.

6.3 Tests for pitting corrosion, such as measurement of the critical pitting temperature, CPT, (e.g. see ISO 17864[7]) or determination of the pitting potential by potentiodynamic polarization (e.g. see ISO 15158) shall be undertaken with a surface finish that replicates that of the end product, as localized attack is very sensitive to surface condition, including physical defects and surface oxides.

NOTE 1 It is common in testing of pitting resistance to use a well-defined, usually fine-ground or polished finish as this gives repeatable and reproducible results. However, such testing provides only a ranking of materials and can possibly not have a bearing on the likelihood of pitting in service. It can also lead to an overemphasis on the influence on pitting of microchemical features of the alloy, such as manganese sulphide, MnS, inclusions in stainless steel, where in service physical defects on the surface, or the specific oxides formed in processing, can have a more significant impact. This can be more significant for AM alloys where additional surface finishing can possibly not always be undertaken or feasible for all surfaces.

NOTE 2 The first stage in assessing AM produced metals and alloys is to use the same test conditions as for conventional alloys.

6.4 In conducting pitting tests, the precision in the measured parameters shall be determined by repeat tests as given in ISO 15158, with the number of such tests dependent on the specimen size (e.g. see Reference [8]).

NOTE In view of the distribution of pore size in AM materials and other possible defects, there is a greater possibility of more statistical variability in test results compared to wrought alloys. In that case, determination of parameters such as the pitting potential or CPT, can necessitate more tests than currently specified or require the use of specimens of greater surface area.

6.5 Tests shall be undertaken with the surface in the Z and X/Y orientation (in accordance with ISO/ASTM 52921), as the extent of pitting attack in some alloy systems can be sensitive to the microstructural orientation. For tests in the direction parallel to the build direction, tests should be conducted primarily with the exposed material in the plane of the build direction (XY plane) as being closest to the exposed surface in service, although consideration should be given also to testing in the ZY plane.

6.6 Crevice corrosion testing shall be undertaken with the surface in the same finish as in service and follow the appropriate test standard (such as ISO 18089[9]).

NOTE Crevice corrosion is sensitive to many of the same issues as pitting corrosion with the additional factor that the surface roughness impacts markedly on the effective crevice gap in most testing methods.

7 Environmentally assisted cracking testing

7.1 The specimen type and method of preparation from AM coupons should be configured to reflect the key objective of the testing, whether crack initiation resistance or crack propagation determination.

7.2 A testing methodology that uses test specimens that retain the original surface of the end product shall be adopted to evaluate the resistance to crack initiation and to undertake pass/fail tests.

NOTE 1 Testing standards that enable a testing configuration with one surface of the specimen in the original finish include bend tests (e.g. see ISO 16540[10]) and flat dog-bone specimens (e.g. see ISO 7539-4[11]).

NOTE 2 Testing with specimens of AM alloys prepared to a defined finish can be undertaken to compare with the extensive data for conventionally processed alloys. Ground specimens have traditionally been the basis of qualification for service in many applications despite the surface finish often not relating to that in service. However, there is extensive service history to benchmark performance and that does not exist for AM alloys. Also, the residual stress following specimen preparation will differ markedly from that of the as-built product and can distort the results.

7.3 The number of repeat tests to determine the fatigue limit in fatigue and corrosion fatigue testing (see ISO 11782-1[12]) should reflect the variability associated with the material which is potentially greater with AM products.

NOTE 1 Measurement of the fatigue limit often shows significant variability due to sensitivity to the surface finish as well as microstructural characteristics of the alloy. The presence of pores and any other physical defect present will accentuate that variability because of the impact of flaws on stress localization. In corrosion fatigue testing, the likely increased variability in pitting likelihood (see Clause 6) will also impact on the dispersion of fatigue strength data.

NOTE 2 Hot isostatic pressing (HIP) does not close up pores at the surface as well as in the bulk because of the 2D nature of the stresses at the surface. Pores can be retained at the surface on the product if tested with the product surface retained. Hence, associated variability in test results can be retained for HIP AM materials. If the test specimen surface of HIP AM materials are ground, thereby removing the outer surface with the higher density of pores, the results will be unrepresentative unless a similar surface preparation is adopted in service. It can also be the case that pores formed by entrainment of the inert gas, usually argon, in some processing routes can remain open during HIP because of low solubility of the inert gas in the matrix.

7.4 Testing shall be undertaken with the stress axis both parallel and perpendicular to the build direction and associated microstructural orientation, but other orientations of the stress axis should be considered when pertinent to the application.

Specimens cut from a single large coupon with the long axis of the test specimen parallel or perpendicular to the build direction may not give the same response as individual coupons printed in the Z and X/Y-direction.