# INTERNATIONAL STANDARD

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# Corrosion of metals and alloys — Guidelines for the evaluation of pitting corrosion

*Corrosion des métaux et alliages — Lignes directrices pour l'évaluation de la corrosion par piqûres* 

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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 <a href="https://www.iso.org/directives">www.iso.org/directives</a>).

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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 <a href="https://www.iso.org/iso/foreword.html">www.iso.org/iso/foreword.html</a>.

This document was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/ TC 262, *Metallic and other inorganic coatings, including for corrosion protection and corrosion testing of metals and alloys*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 11463:1995), which has been technically revised. The main changes compared with the previous edition are as follows:

 modern surface analysis and characterization techniques for ex situ examination have been included.

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 <u>www.iso.org/members.html</u>.

## Introduction

It is important to be able to determine the extent of pitting and its characteristics, either in a service application, where it is necessary to estimate the remaining life in a metal structure, or in laboratory test programmes that are used to select pitting-resistant materials for a particular service. Corrosion pits can also act as the precursor to other damage modes such as stress corrosion cracking and corrosion fatigue.

The application of the materials to be tested will determine the minimum pit size to be evaluated and whether total area covered, average pit depth, maximum pit depth or another criterion is the most important to measure.

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# **Corrosion of metals and alloys — Guidelines for the evaluation of pitting corrosion**

## 1 Scope

This document gives guidelines for the selection of procedures that can be used in the identification and examination of corrosion pits and in the evaluation of pitting corrosion and pit growth rate.

#### 2 Normative references

There are no normative references in this document.

## 3 Terms and definitions

No terms and definitions are listed in this document.

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

- ISO Online browsing platform: available at <a href="https://www.iso.org/obp7">https://www.iso.org/obp7</a>
- IEC Electropedia: available at <a href="http://www.electropedia.org/">http://www.electropedia.org/</a>
  - (standards.iteh.ai)

## 4 Identification and examination of pits<sub>020</sub>

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#### 4.1 Preliminary low magnification visual inspection

**4.1.1** A visual examination of the corroded metal surface with or without the use of a low-power magnifying glass may be used to determine the extent of corrosion and the apparent location of pits. It is often advisable to photograph the corroded surface so that it can be compared with the clean surface after the removal of corrosion products or with a fresh unused piece of material.

**4.1.2** If the metal specimen has been exposed to an unknown environment, the composition of the corrosion products may be of value in determining the cause of corrosion. Recommended procedures for the removal of particulate corrosion products should be followed and the material removed should be preserved for future identification.

**4.1.3** To expose the pits fully, it is recommended that cleaning procedures should be used to remove the corrosion products. Rinsing with water followed by light mechanical cleaning can be sufficient for lightly adhered corrosion products. Chemical cleaning is required for more adherent products. ISO 8407<sup>[1]</sup> provides a range of chemical cleaning processes. Preliminary testing should be undertaken to ensure that attack of the base metal is avoided.

#### 4.2 Optical microscopic examination of pit size and shape

**4.2.1** Examine the cleaned metal surface to determine the approximate size and distribution of pits. Follow this procedure by a more detailed examination through a microscope using a low magnification (approximately × 20). Pits can have various sizes and shapes. A visual examination of the metal surface can show a round, elongated or irregular opening, but it seldom provides an accurate indication of the

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extent of corrosion beneath the surface. Thus, it is often necessary to cross-section the pit to determine its actual shape. Several common variations in the cross-sectioned shape of pits are shown in <u>Figure 1</u>.



**4.2.2** It is difficult to determine pit density by counting pits through a microscope eyepiece, but the task can be made easier by the use of a plastic grid. Place the grid, containing 3 mm to 6 mm squares, on the metal surface. Count and record the number of pits in each square and move across the grid in a systematic manner until all the surface has been covered. This approach minimizes eyestrain because the eyes can be taken from the field of view without fear of losing the area of interest. Enlarged photographs of the area of interest may also be used to reduce eyestrain. An alternative approach is to mount the specimen on an x-y stage and measure both the number and spatial distribution of pits. When coupled with optical depth measurement, where applicable, the number, depth and spatial distribution of pits can be determined.

**4.2.3** Advanced optical microscopy techniques, such as infinite focus microscopy and confocal laser microscopy may be used to obtain three-dimensional images of the pit surface, within the constraints of optical observations [most relevant to Figure 1 a) to c) but not applicable to undercut]. Such measurements can be used to view the surface features and quantify surface roughness, pit depth, surface profile, etc.

**4.2.4** To carry out a metallographic examination, select and cut out a representative portion of the metal surface containing the pits and prepare a metallographic specimen. If corrosion products are to be examined in cross-section, it may be necessary to fix the surface in a mounting compound before cutting. Examine microscopically to determine whether there is a relation between pits and inclusions or microstructure, or whether the cavities are true pits or might have resulted from metal loss caused by intergranular corrosion, dealloying, etc.

#### 4.3 In situ non-destructive inspection

#### 4.3.1 General

Several techniques have been developed to assist in the detection of cracks or cavities in a metal surface without destroying the material (see Reference [2]). These methods are less effective for locating and defining the shape of pits than some of those described previously, but they merit consideration because they are often used in situ, and thus they are more applicable to field applications.

#### 4.3.2 Radiographic

Radiation, such as X-rays, passes through the object. The intensity of the emergent rays decreases with increasing thickness of the material. Imperfections can be detected if they cause a change in the absorption of X-rays. Detectors or films are used to provide an image of interior imperfections. The metal thickness that can be inspected is dependent on the available energy output. Pits must be as large as 0,5 % of the metal thickness to be detected and care should be taken to ensure that pits are not confused with pre-existing pores.

#### 4.3.3 Electromagnetic

**4.3.3.1** Eddy currents may be used to detect defects or irregularities in the structure of electrically conductive materials. When a specimen is exposed to a varying magnetic field, produced by connecting an alternating current to a coil, eddy currents are induced in the specimen and they in turn produce a magnetic field of their own. Materials with defects will produce/a magnetic field that is different from that of a reference material without defects, and an appropriate detection instrument is required to determine these differences. (standards.iteh.ai)

**4.3.3.2** The induction of a magnetic field in ferromagnetic materials is another approach that is used. Discontinuities that are transverse to the direction of the magnetic field cause a leakage field to form above the surface of the part. Ferromagnetic particles are placed on the surface to detect the leakage field and to outline the size and shape of the discontinuities. Rather small imperfections can be detected by this method. However, the method is limited by the required directionality of defects to the magnetic field, by the possible need for demagnetization of the material, and by the limited shapes of parts that can be examined.

#### 4.3.4 Ultrasonics

In the use of ultrasonics, pulses of sound energy are transmitted through a couplant, such as oil or water, on to the metal surface where waves are generated. The reflected echoes are converted to electrical signals that can be interpreted to show the location of flaws or pits. Both contact and immersion methods are used and various techniques can be applied. The test should be carried out from the non-pitted face. The test is affected by the morphology of the pits, the ultrasonic technique selected and the performance of the probe and flaw detector. Information about the size and location of flaws can be established. However, the capability of the technique for the pitting expected should be assessed and reference standards produced for comparison. Operators should be trained in the application of the technique and the interpretation of the results.

#### 4.3.5 Penetrants

Defects opening to the surface can be detected by the application of a penetrating liquid that subsequently exudes from the surface after the excess penetrant has been removed. Defects are located by spraying the surface with a developer that reacts with a dye in the penetrant, or the penetrant may contain a fluorescent material that is viewed under ultraviolet light. The size of the defect is shown by the intensity of the colour and the rate of bleed-out. This technique provides only an approximation of the depth and size of pits.

#### 4.3.6 Replication

Images of a pitted surface can be created by applying a material to the surface that conforms to the shape of the pits and can be removed without damaging its shape. This method will not work, however, for pits of subsurface or undercut type. The removed material contains a replica of the original surface that, in some cases, is easier to analyse than the original. Replication is particularly useful for the analysis of very small pits.

#### 4.4 Ex situ examination techniques

#### 4.4.1 General

Several sophisticated ex-situ techniques are available for examining the size, shape and distribution of pits in metallic samples. Their application would involve transport of the specimens to a laboratory or dedicated analytical facility. Some of these techniques are described in 4.4.2 to 4.4.5.

#### 4.4.2 Scanning electron microscopy

Scanning electron microscopy (SEM) can be used to obtain images containing topographic and phase contrast information. It is a very useful technique for obtaining images of pits in surfaces and the technique can be used to determine the dimensions of the pit and any relationships with different phases within the microstructure of the metal. By combining electron-dispersive X-ray spectroscopy (EDS) or wavelength-dispersive X-ray spectroscopy (WDS), elemental composition and distribution of any corrosion products in pits can be determined. However, in deeper pits and where subsurface undercutting of the pit mouth has occurred, electron emission is shielded from the detector and this can limit the effectiveness of the technique for imaging the pit morphology.

#### 4.4.3 X-ray computed tomography

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X-ray computed tomography (**CT**) is a non-destructive technique that, coupled with reconstruction software, can enable 3D imaging of pits. The images are constructed by taking slices through the sample using high intensity X-ray sources, which may be X-ray tubes in conventional laboratories or derived from synchrotron X-ray sources. The thickness of specimen can be limited due to X-ray attenuation. Sectioning parallel to the surface can be required to reduce the thickness. Nevertheless, the technique is a powerful tool for 3D imaging of pits of complex shape.

#### 4.4.4 Image analysis

Image analysis is the technique whereby images that have been taken using a measurement technique such as optical microscopy or X-ray computed tomography are post-processed to extract quantitative information. The technique can be used to automate the analysis or post-processing of images to reduce time and cost. It also permits the analysis of a greater number of images, thereby improving the statistical reliability of the measurements. Image analysis allows micrographs to be processed rapidly and can produce data that are more accurate and statistically robust than manual methods.

#### 4.4.5 Profilometry

Profilometry measures the physical surface geometry or topography of a sample. It may be classed as "contact" or "non-contact". Contact profilometry involves a stylus, with known tip dimensions, being brought into contact with the sample surface, and then rastered over the surface. The displacement of the stylus tip as it comes into contact with high and low features on the surface is monitored and recorded as a function of its position. From this data, the physical characteristics of the surface, such as roughness, can be measured, and any features of interest, such as pitting, can be identified and quantified.

Non-contact methods record the same type of information, although they usually employ laser-based optical methods, such as an infinite focus microscope, and they do not require direct physical contact with the sample surface. Such techniques develop a 3D surface profile through the accumulation of

images at different optical focal planes, and white-light interferometry, where the phase difference between light reflected from the sample surface and light from a reference mirror are compared, and differences in the path length due to the surface morphology may be recorded. Confocal laser microscopes can give similar information.

The disadvantage of these techniques is that they characterize only what they can detect optically and are applicable mainly to pit types such as Figure 1 a) to c) (see also 4.2.3).

## 5 Extent of pitting

#### 5.1 Mass loss

Metal mass loss is not ordinarily recommended for use as a measure of the extent of pitting unless general corrosion is slight and pitting is fairly severe. If uniform corrosion is significant, the contribution of pitting to total metal loss is small, and pitting damage cannot be determined accurately from mass loss. In any case, mass loss can only provide information about total metal loss due to pitting but nothing about density of pits and depth of penetration. However, mass loss should not be neglected in every case because it may be of value. For example, mass loss along with a visual comparison of pitted surfaces can be adequate to evaluate the pitting resistance of alloys in laboratory tests. Mass loss may also be useful to detect the existence of subsurface metal loss.

#### 5.2 Pit depth measurement

# 5.2.1 Metallography Teh STANDARD PREVIEW

Pit depth may be determined by sectioning vertically through a preselected pit, mounting the crosssectioned pit metallographically and polishing the surface. A better or alternative way is to section slightly away from the pit and slowly grind until the pit is in the cross-section. Sectioning through a pit can be difficult and the deepest portion can be missed. The depth of the pit is measured on the flat, polished surface using a microscope with a calibrated eyepiece. The method is very accurate, but it requires good operator skill, good judgement in the selection of the pit and good technique in cutting through the pit. Its limitations are that it is time-consuming, the deepest pit may not have been selected and the pit may not have been sectioned at the deepest point of penetration. This technique will result in the destruction of the specimen.

#### 5.2.2 Machining

NOTE See References [3] and [4].

**5.2.2.1** This method requires a sample that is fairly regular in shape. It usually involves the destruction of the specimen. Measure the thickness of the specimen between two areas that have not been affected by general corrosion. Select a portion of the surface on one side of the specimen that is relatively unaffected, then machine the opposite surface where the pits are located on a precision lathe, grinder or mill until all signs of corrosion have disappeared. Some difficulty from galling and smearing can be encountered with soft metals and pits can be obliterated. Conversely, inclusions can be removed from the metal thus confusing the examination. Measure the thickness of the specimen between the unaffected surface and subtract from the original thickness to give the maximum depth of pitting. Repeat this procedure on the unmachined surface unless the thickness has been reduced by 50 % or more during the machining of the first side.

**5.2.2.2** This method is equally suitable for determining the distribution of pit depths in a sample. Count the visible pits then machine away the surface of the metal in measured stages (the amount of material removed in each step will determine the uncertainty in pit depth). Continue this process, noting for each pit the depth of material removed at which it is no longer visible. The depth of the pit will lie between the depth of last observation and the depth of material at which it is no longer visible.