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Corrosion of metals and alloys - Method for metallographic examination of samples after exposure to high-temperature corrosive environments

金属和合金的腐蚀 在高温腐蚀环境下暴露后试样的金相检验方法

(ISO 26146:2012, IDT)

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Corrosion of metals and alloys - Method for metallographic examination of samples after exposure to high-temperature corrosive environments

1 Scope

This document applies to the metallographic examination of samples after exposure to high-temperature corrosive environments.

This document specifies methods for the classification, identification and thickness measurement of the corrosion scale formed on samples after exposure to high-temperature corrosive environments.

2 Normative references

The following referenced documents are indispensable for the application 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 3611, *Geometrical product specifications (GPS) - Dimensional measuring equipment: Micrometers for external measurements - Design and metrological characteristics*

ISO 13385-1, *Geometrical product specifications (GPS) - Dimensional measuring equipment - Part 1: Design and metrological characteristics of calipers*

3 Terms and definitions

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

NOTE: Figure 1 is a schematic representation of the scales in the exposed sample.

deposit scale in the normal direction perpendicular to the surface of the sample.

3.6 coating

A protective scale of material applied to the surface of metallic materials.

3.7 coating thickness

The distance between the original metal surface and the coating surface in the direction perpendicular to the normal to the surface of the sample.

3.8 outward growing corrosion scale

A scale of corrosion that grows outward from the original surface of a metal or coating.

3.9 inward growing corrosion scale

A scale of corrosion that grows inward from the original surface of a metal or coating.

3.10 external scale

Generic term for inward growing and outward growing successive scales of corrosion.

3.11 internal corrosion

Corrosion products formed below the external scale.

NOTE: Generally, appear as discrete particles.

3.12 grain boundary corrosion

Corrosion products grow along metal grain boundaries, which is a special form of internal corrosion.

3.13 de-alloyed zone

Below the corrosion scale, the area where the concentration of the alloying elements that form the corrosion scale is reduced.

NOTE: It may manifest as dissolution of pre-existing precipitates in the microstructure.

3.14 metal loss

The distance between the original surface of the sample and the boundary of the alloy-unaffected area of the sample.

3.15 remaining sound metal

That part of a metal that is not affected by corrosion.

4 Symbols and abbreviations

4.1 Symbols

The following symbols apply to this document:

x - the thickness of each scale;

t - the thickness of metal material.

4.2 Abbreviations used in footnotes

The identification of each scale adopts the following footnotes:

o - original;

og - the outward growing part of the external scale;

ig - the inward growing part of the external scale;

tot - total thickness of the external scale ($x_{tot}=x_{og}+x_{ig}$);

in - internal corrosion;

gb - grain boundary corrosion;

d - deposit;

c - coating;

da - de-alloying;

ml - metal loss compared with the original size;

rm - remaining sound metal.

5 Requirements

5.1 The minimum requirement for test accuracy is that when the 95% confidence level is reached, the loss of the measured material does not exceed ±5μm or 5%. The uncertainty is the minimum value required for calibration, misalignment (in both vertical and horizontal directions) and measurement errors.

5.2 The optical microscope used in the test shall have a stage that can move in the X-Y axis direction or have counting lines and cross lines. The accuracy of the test system shall reach ±1μm.

6.2 Test process

6.2.1 Inspection before exposure test

Before the exposure test, it shall be in accordance with the provisions of ISO 3611 and ISO 13385-1. Use a measuring instrument with a measuring accuracy of ±0.02 mm to measure the dimensions of the uncorroded sample. In the area to be measured after the exposure test, it is advisable to select no less than 8 equidistant positions for size measurement.

Dimensional measurements are the basis for the characterization of the original surface characteristics of metals.

6.2.2 Preparation of cross-sectional metallographic samples

Cross-sectional metallographic samples shall be prepared to determine the dimensional change of the samples after the exposure test. Great care shall be taken when preparing cross-sectional metallographic samples. Ensure mounting and polishing are carried out parallel to the cross-section to be measured. Retain all corrosion products and deposits. The recommended method for preparing cross-sectional metallographic samples is as follows.

The samples shall be protected with a suitable coating, so as to ensure the retention of corrosion products and deposits on the samples. Depending on the sample system tested, metallic coatings or thermoplastic resins prepared by deposition methods can be used.

Sample supports may be required in the mounting mold to ensure that the section remains flat.

Samples can be cut before and after mounting. Rod and tubular samples shall be cut into circular cross-sections along the direction perpendicular to the main axis. Disc-shaped samples shall be cut parallel to the main axis. Block samples shall be cut along the direction parallel to the longest face. See Figure 3 for details of cutting requirements.

If it is required to expose a cross-section of a particular plane relative to the surface of the reference mark, it shall be cut at a plane far enough from that surface to allow sufficient removal for grinding and polishing (see Figure 3).

One or more artificial samples of known dimensions shall be used as references for mounting to demonstrate that the sample is mounted and polished in a direction parallel to the intended cross-section. Flat samples (disc and square structures) can be mounted together. Use a metal sheet with a constant thickness and known (±1μm) to fix it tightly along the direction parallel to the sample. A bar sample may be a similar sheet metal of known thickness bent to a known angle (90° is recommended). Place the sample on the bent corner of the sheet metal. The alternative of placing multiple spheres around the sample during mounting can also be used. A schematic diagram of checking alignment during polishing using a reference artificial sample is shown in Figure 4.

- internal corrosion scale;

- grain boundary erosion zone;

- de-alloyed zone;

- residual coating;

- interdiffusion zone;

- unaffected alloy (remaining sound metal).

The definitions of these scales are detailed in Chapter 3.

6.2.4 Identification of corrosion scale

6.2.4.1 It is recommended to take optical pictures of corrosion products and deposits at a range of standard magnifications such as 100X, 400X and 1000X. The standard magnification shall be determined according to the damage degree of the sample. The magnification is chosen to clearly show the total extent of damage in one picture, so that the entire thickness of the external corrosion scale could be seen. An optical microscope shall be equipped with a micrometer ruler.

6.2.4.2 Various techniques such as chemical etching or interference films can be used to enhance the contrast between the different scales.

6.2.4.3 According to specific inspection requirements, more characterizations can also be carried out by scanning electron microscopy (SEM), X-ray diffraction (XRD).

6.2.5 Corrosion scale thickness test for evaluation of metal material loss

6.2.5.1 The sample shall be divided into 8 parts of approximately the same size.

6.2.5.2 Each part shall be viewed under low magnification. Find the zone with the smallest remaining metal thickness. Testing will be conducted in this selected zone.

6.2.5.3 Test the selected zone at high magnification. A 100μm long surface is displayed within a single field of view.

6.2.5.4 Positions c and d as shown and defined in Figure 5 shall be tested. In addition, positions a, f, b, and e may also be tested in order to obtain supplementary information on the different corrosion scales.

6.2.5.5 The minimum remaining metal thickness (t_{rm}) is the distance between c and d. Metal loss thickness (t_{ml}) is calculated by $t_{ml}= (t_0-t_{rm})/2$. t_{rm} and t_{ml} shall be written into the test report.

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