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Copper alloys — Ammonia test for stress corrosion resistance

Alliages de cuivre — Essai à l'ammoniaque pour la résistance à la corrosion sous contrainte

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Foreword

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International Standard ISO 6957 was prepared by Technical Committee ISO/TC 26, *Copper and copper alloys*.

Annexes A and B of this International Standard are for information only.

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Copper alloys — Ammonia test for stress corrosion resistance

1 Scope

This International Standard specifies a test, using an ammoniacal atmosphere, for the detection of applied or residual stresses in copper alloy products which can cause failure of the material in service or storage through stress corrosion cracking. The method can also be used for testing assemblies and partial assemblies (of limited size).

This method is relatively simple to carry out and its severity can be regulated by changing the pH value of the solution producing the ammoniacal atmosphere.

The appropriate pH-value for the test shall be specified in the product specification or agreed upon with respect to the alloy and its application.

In annex A, recommendations are given for the choice of pH-values to be used for some frequently occurring applications.

2 Principle

Exposure of test pieces to an ammoniacal atmosphere for 24 h, followed by examination for cracks at a magnification of $\times 10$ to $\times 15$.

3 Reagents and materials

Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

3.1 Ammonium chloride, 4 mol/l solution, for the preparation of the test solution.

Dissolve $107 \text{ g} \pm 0,1 \text{ g}$ of ammonium chloride (NH_4Cl) in water, make up the volume to 500 ml and keep the solution in a closed vessel.

3.2 Sodium hydroxide, 30 to 50 % (m/m) solution, for adjusting the pH-value.

3.3 Clean organic solvent (for example, trichloroethylene) or **hot alkaline solution**, for degreasing the test pieces.

3.4 Sulfuric acid, 5 % (m/m) solution, for cleaning the test pieces.

3.5 Hydrogen peroxide, 30 % (m/m) solution, for addition to the pickling solution.

4 Apparatus

Ordinary laboratory apparatus and

4.1 pH-meter.

4.2 Closed vessel (such as a desiccator).

4.3 Equipment for examining test pieces at $\times 10$ to $\times 15$ magnification.

5 Definitions

For the purpose of this International Standard, the following definitions apply.

5.1 stress corrosion: A process involving conjoint corrosion and straining of the metal due to residual or applied stresses.

5.2 applied stress: Stress in a body due to application of an external load.

5.3 residual stress: Stress remaining within a body as the result of plastic deformation.

6 Test media

6.1 Slowly add sodium hydroxide solution (3.2) to ammonium chloride solution (3.1) to give a test solution with the specified pH-value $\pm 0,05$ (see annex A). Maintain the solution at ambient temperature and dilute with water up to a volume of 1 000 ml. Check the pH-value with the pH-meter (4.1) after dilution. Prepare the solution preferably in a fume cupboard in a laboratory and store it in a closed vessel. Before use, check the pH-value again, and adjust it if necessary.

6.2 The pickling solution for cleaning test pieces before and after testing shall be 5 % (m/m) sulfuric acid (3.4). If necessary, for cleaning test pieces after testing, a small amount of hydrogen peroxide solution (3.5) may be added to the pickling solution (for example, 20 to 50 ml of hydrogen peroxide solution per litre of pickling solution).

7 Test piece

7.1 The length of the test piece shall be at least 150 mm for wrought products having a maximum dimension in their cross-section of 75 mm. For other products, the test piece size shall be subject to agreement.

7.2 The number of replicate test pieces shall be agreed upon, if not specified in the product specification.

7.3 Each test piece shall be marked for identification (if necessary) in a way so as to avoid introduction of additional stresses in the piece.

8 Procedure

8.1 Degrease the test piece using the clean organic solvent or the hot alkaline solution (3.3).

8.2 After degreasing, clean the test piece in the pickling solution (see 6.2) and immediately thereafter thoroughly rinse it, first in cold running water, then in hot water, and finally completely dry it in a stream of warm air.

8.3 Allow the dry test piece to reach the exposure temperature specified below, and transfer it to the closed vessel (4.2) at the same temperature, and containing the freshly prepared test solution at the specified pH-value (see 6.1). Place the test piece in the vessel in such way that the ammonia vapour has free access to all surfaces.

The volume of test solution shall be at least 200 ml per litre of total vessel volume and at least 100 ml per square decimetre of test piece surface. The exposure temperature shall be between 20 °C and 30 °C and shall be kept constant to within ± 1 °C during the test; in case of dispute, the temperature shall be kept at 25 °C ± 1 °C. The exposure time shall be 24 h.

8.4 After exposure, remove the test piece from the closed vessel and immediately clean it in pickling solution for a few minutes at ambient temperature (below 40 °C), i.e. until the surfaces of the test piece are sufficiently clean from corrosion products to allow observation of possible cracks. After rinsing in water and drying in warm air, as specified in 8.2, examine the surfaces of the test piece for cracks at a magnification of $\times 10$ to $\times 15$. Before inspection, it may be necessary to deform the test piece slightly so that fine cracks are opened up and more readily seen. For heavy sections, a slice may be cut to facilitate bending. Cracks within a 5 mm wide zone along cut or sawn edges, or around punched markings, originating from the test piece preparation shall be disregarded. For thin sections (below 0,2 mm), check by metallographic examination whether observed cracks represent stress corrosion cracking or intergranular corrosion.

9 Test report

The test report shall include the following information:

- a) an identification of the sample, and the location of the test piece in the sample if the test was carried out on a test piece taken from the sample, rather than on the sample itself;
- b) the reference of the method used;
- c) the pH-value of the solution producing the ammoniacal atmosphere;
- d) the exposure temperature;
- e) the number of replicate test pieces tested;
- f) the test results: cracks or no cracks (as required in the appropriate product specification);
- g) any unusual features noted during the determination; any operation not included in this International Standard or regarded as optional;
- h) the date of the test.

Annex A (informative)

Guidance for choice of the pH-value of the test solution

On the basis of the known correlation between the behaviour of test pieces in the ammonia test and the behaviour of copper alloy products under service conditions, the following pH-values are recommended as being representative of atmospheres of different corrosiveness and corresponding to different safety requirements.

| Corrosiveness of atmosphere | pH-value | |
|--|--------------------|--------------|
| | Safety requirement | |
| | Low | High |
| Low Indoor atmosphere under dry conditions | 9,3 | 9,5 |
| Moderate Indoor atmosphere with risk of formation of condensation Outdoor atmosphere, temperate climate | 9,5 9,5 | 10,0 10,0 |
| High Atmosphere with ammoniacal pollution, for example in stables | 10,0 | 10,5 |

Annex B (informative)

Bibliography

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