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Metallic coatings — Test methods for electrodeposited gold and gold alloy coatings — Part 2 : Environmental tests

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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Metallic coatings — Test methods for electrodeposited gold and gold alloy coatings —

Part 2 : Environmental tests

1 Scope and field of application

This part of ISO 4524 specifies methods for simulated environmental testing of electrodeposited gold and gold alloy coatings for engineering, and decorative and protective purposes.

2 References

ISO 4524/3, *Metallic coatings — Test methods for electrodeposited gold and gold alloy coatings — Part 3 : Electrographic tests for porosity*.

ISO 4538, *Metallic coatings — Thioacetamide corrosion test (TAA test)*.

IEC Publication 68-2-42, *Basic environmental testing procedures — Test Kc : Sulphur dioxide test for contacts and connections*.

IEC Publication 362-2, *Printed boards — Part 2 : Test methods*.

3 General

Four tests are described, two using atmospheres containing sulfur dioxide, one using an atmosphere containing nitric acid vapour, and one using an atmosphere containing thioacetamide vapour. The effect produced will depend on the nature of the metal exposed at discontinuities in the gold coating. Sulfur dioxide is a general purpose reagent producing visible effects for exposure of any basis metal or undercoat, although the effects are not always well marked for substrates or undercoats of silver. Thioacetamide, a producer of sulfide, will have most effect on exposed silver, some effect on copper and no effect on nickel or tin. Nitric acid vapour gives indications for copper and nickel substrates or undercoats, but is not useful for silver. Any effect on the coating itself will depend on the nature and amount of alloying elements. The test chosen should be capable of showing the exposure of the basis metal or any of the undercoats. For general purposes it has not been considered necessary to include a test during which a correctly applied undercoat metal is likely to be penetrated. The thickness of undercoats specified should be an adequate

safeguard against penetration of corrosion to the basis metal in normal service, but it is suggested that when exceptionally severe conditions have to be met, special tests should be agreed upon.

It is possible to obtain direct indications of porosity by use of one of the electrographic tests described in ISO 4524/3. However, it is often more convenient, especially for articles of complex shape, to reveal the pore sites and to develop corrosion products more likely to resemble those produced in service by exposing test specimens to a humid atmosphere polluted with a gas or vapour able to react with basis metal or undercoat exposed at pores in the gold coating.

The tests are applicable to articles of any size smaller than about 100 mm × 100 mm. Specimens shall be degreased in 1,1,1-trichloroethane vapour or other suitable solvent before the test. Any cut edges or other uncoated substrate shall be protected during the test.

3.1 Engineering purposes

Electroplated gold and gold alloy coatings for engineering purposes are required to retain over long periods of storage or use the ability to perform the functions they serve. Among these functions are low contact resistance, easy solderability and the provision of a stable surface suitable for carrying high frequency currents. Impairment of performance may result from

- corrosion of basis metal or of undercoat metal exposed at discontinuities in the gold coating, with the formation of corrosion products as spots or as a thin general layer;
- for some alloys, the formation of corrosion products of the alloying elements as a layer over the surface.

The properties of the corrosion products produced during the tests differ from the products formed in actual environments, and the tests are not recommended for evaluation of the electrical performance of contacts unless correlation is first obtained with service experience. There is some evidence that the industrial atmosphere test (see clause 5) may permit such a correlation to be obtained rather more readily than the other tests. However, this test requires much more elaborate apparatus for its correct operation.

3.2 Decorative and protective purposes

The main requirement of decorative gold coatings is that they shall remain unchanged in appearance when exposed to indoor atmospheres for long periods.

Changes in appearance may result from

- a) corrosion at discontinuities in the coating, with formation of corrosion products as spots or as a thin dull film;
- b) for some alloys of low gold content, general loss of brightness or change of colour.

4 Sulfur dioxide exposure test

WARNING — Attention is drawn to the toxic hazards of sulfur dioxide; the test vessels, at least during filling and opening shall be placed under a suitable extraction hood.

This test is suitable for application with undercoats of copper and/or nickel. The generation of sulfur dioxide from known reagents inside the test vessel permits an easy control of the atmosphere composition. A suitable test vessel is a container made of glass or acrylic resin of capacity about 10 l, capable of being closed by a gastight cover or door. A conventional desiccator of capacity 10 l is recommended. Provision shall be made for the support of test specimens within the vessel so that they are about 100 mm above its base. The specimens may be suspended from rods by means of suitable material, for example acrylic thread or glass hooks. All materials used for chamber construction or specimen support shall be non-metallic and inert to the action of sulfur dioxide. Glass and acrylic resin are suitable materials.

The test temperature shall be maintained within a temperature range agreed as being suitable for the materials under test and for a selected duration of test.

During the test, the test vessels shall be kept screened from draughts or from any source of local radiant heat so that sudden temperature fluctuations and local heating of the test chamber are avoided.

Introduce 200 ml of a 200 g/l sodium thiosulfate pentahydrate ($\text{Na}_2\text{S}_2\text{O}_4 \cdot 5 \text{H}_2\text{O}$) solution into the test chamber. Add to it 50 ml of sulfuric acid ($\rho \approx 1,84 \text{ g/ml}$), previously diluted to 100 ml with distilled water, taking appropriate precautions. Place the test specimens immediately in the vessel so that they are not less than 75 mm above the surface of the liquid and not less than 25 mm from the walls. Close the vessel immediately and move it gently to ensure complete mixing of the two solutions.

After the end of the test period, open the test chamber, remove and inspect the test specimens. Two recommended test periods are 24 h at $25 \pm 5^\circ\text{C}$ and 2 h at $60 \pm 3^\circ\text{C}$.

Alternatively, the method specified in IEC Publication 326-2 can be used.

5 Industrial atmosphere test

Details of the equipment required and of the operation of the test are provided in IEC Publication 68-2-42. The test was devised to assess the action on materials of atmospheres containing the major combustion products of sulfur-containing carbon and hydrocarbon fuels. It has been found suitable to provide information on a comparative basis about the behaviour of materials exposed to polluted atmospheres. An outline of the test procedure is as follows :

A test cabinet with a capacity of about 200 l is provided with a means of moving test specimens through the test atmosphere at $11 \pm 3 \text{ mm/s}$. The test atmosphere, maintained at $25 \pm 2^\circ\text{C}$, contains $25 \pm 5 \text{ cm}^3/\text{m}^3$ of sulfur dioxide and has a relative humidity as close as possible to 75 % and within a range of 70 % to 80 %.

The test atmosphere shall flow through the chamber at a rate sufficient to produce 3 to 5 complete changes of atmosphere per hour. It may be generated either by mixing directly the necessary constituents, or by burning propane, butane or natural gas with a controlled addition of carbon disulfide and adjusting the resulting combustion product to the required composition by addition of air and water vapour.

If the object of testing is to determine any increase in contact resistance, the test pieces will usually be samples taken from finished products and the contact resistance will be measured before and after test.

6 Sulfide test using thioacetamide

WARNING — Thioacetamide is classified as a carcinogenic agent in some countries.

Carry out the thioacetamide test described in ISO 4538.

7 Nitric acid test

WARNING — A partial vacuum may be created in the test chamber during this test. This can be avoided by inserting a piece of polytetrafluoroethylene (PTFE) tape under the chamber lid or by using an apparatus fitted with a stopcock. Do not attempt to equalize the pressure by heating the nitric acid solution in the chamber as this can result in violent spattering.

Tests shall be carried out under an extraction hood since the gases released when the test vessel is opened at the end of the test may be harmful to health.

The test chamber requirements are similar to those described for the sulfur dioxide test in clause 4 except that only glass may be used as the material of construction and as specimen supports. The chamber volume, in cubic centimetres, shall be not more than 25 times the nitric acid surface, in square centimetres. Accurate temperature control is not essential, but a test temperature of $23 \pm 3^\circ\text{C}$ is recommended.

The solution used ($\rho 1,40$ to $1,42 \text{ g/ml}$) contains between 65 and 71 % (*m/m*) of nitric acid (HNO_3).

The required quantity of nitric acid is added to the test chamber, which is then closed. After 30 min, the cover is carefully lifted, the test samples are introduced and the cover is replaced. Unless otherwise specified, the test duration is 60 ± 10 min for gold coatings on copper or copper alloys and 120 ± 10 min for nickel substrates and undercoats.

At the end of the test, the samples are removed and dried in an oven at 125 ± 3 °C for 30 min. The surface is then examined at X 10 magnification. The corrosion products formed may be transparent in the case of gold-plated nickel and it is recommended that the illumination used for the examination of the samples should make it possible to see the corrosion products as protrusions from the pore sites; for example a collimated beam at an oblique angle below 15° may be used.

The nitric acid is discarded after each test.

The test is not recommended for gold coatings on silver or with substrates for which a silver undercoating is used. Caution is necessary in the application of the nitric acid test to some gold

alloy coatings, with a high alloy content, particularly those containing over 10 % copper, since stress corrosion cracking may produce an apparent increase in defects.

8 Test report

The test report shall include at least the following information :

- a) a reference to this part of ISO 4524, including an identification of the specific method used;
- b) the result(s) of the test(s) carried out and the form in which these are expressed;
- c) any unusual features noticed during the determination;
- d) any operation not included in this part of ISO 4524 or in the International Standards to which reference is made;
- e) any other relevant information requested by the purchaser.

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