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Metallic coatings — Determination of porosity in gold coatings on metal substrates — Nitric acid vapour test

Revêtements métalliques — Détermination de la porosité des revêtements d'or sur les substrats de métal — Essai à la vapeur d'acide nitrique

Reference number
ISO 14647:2000(E)

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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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 14647 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 7, *Corrosion tests*.

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

Introduction

Gold coatings are often specified for the contacts of separable electrical connectors and other devices. Electrodeposits are the form of gold that is most used on contacts, although it is also employed as clad metal and as weldments on the contact surface. The intrinsic nobility of gold enables it to resist the formation of insulating oxide films that could interfere with reliable contact operation.

In order that the nobility of gold be assured, porosity, cracks, and other defects in the coating that expose base metal substrates and underplates should be minimal or absent, except in those cases where it is feasible to use the contacts in structures that shield the surface from the environment or where corrosion inhibiting surface treatments for the deposit are employed. The level of porosity in the coating that may be tolerable depends on the severity of the environment to the underplate or substrate, design factors for the contact device like the force with which it is mated, circuit parameters, and the reliability of contact operation that it is necessary to maintain. Also, when present, the location of pores on the surface is important. If the pores are few in number and are outside the zone of contact of the mating surfaces, their presence can often be tolerated.

Methods for determining pores on a contact surface are most suitable if they enable their precise location and numbers to be determined. Contact surfaces are often curved or irregular in shape, and test methods should be suitable for them. In addition, the severity of porosity-determining tests may vary. The test method described in this International Standard is regarded as severe.

The relationship of porosity levels revealed by particular tests to contact behavior should be made by the user of these tests through practical experience or by judgement. Thus, absence of porosity in the coating may be a requirement for some applications, while a few pores on the critical surfaces may be acceptable for another. Such acceptance (or pass-fail) criteria should be part of the product specification for the particular product or part requiring the porosity test.

The test method described is highly sensitive and is capable of detecting virtually all porosity or other defects in gold coatings that could participate in substrate corrosion reactions. It is rapid, simple and inexpensive. In addition, it can be used on contacts having complex geometry such as pin-socket contacts. However, it is preferred that deeply recessed sockets are opened to expose their critical surfaces prior to testing.

The test method described is considered destructive in that it reveals the presence of porosity by contaminating the surface with corrosion products and by undercutting the coating at pore sites or at the boundaries of unplated areas. Any parts exposed to these tests should not be placed in service.

The test described involves corrosion reactions in which the products delineate defect sites in coatings. Since the chemistry, and properties of these products may not resemble those found in natural or service environments, these tests are not recommended for prediction of the electrical performance of contacts unless correlation is first established with service experience.

The test method described employs nitric acid (HNO_3) vapour at low relative humidity. Reaction of the gas mixture with a corrodible base metal at pore sites produces reaction products that appear as discrete spots on the gold surface. Individual spots are counted with the aid of a lens or low-power stereomicroscope.

The test method described is intended to be used for quantitative descriptions of porosity (such as number of pores per unit area or per contact) only on coatings that have a pore density sufficiently low that the corrosion sites are well separated and can be readily resolved. As a general guideline this can be achieved for pore densities up to about 100 per square centimetre or per 100 contacts. Above this value the tests are useful for the qualitative detection and comparison of porosity.

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WARNING: This International Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use. Specific precautionary measures are given in clause 6 and in 7.4, note 2.

1 Scope

This International Standard specifies equipment and a method for using nitric acid vapour to determine porosity in gold coatings, particularly electrodeposits and clad metals used on electrical contacts.

This method is designed to show whether the porosity level is less than or greater than some value that, by experience, is considered by the user to be acceptable for the intended application.

It is suitable for inlays or claddings containing 75 % or more of gold, for electrodeposits containing 95 % or more of gold or for substrates of copper, nickel and their alloys that are commonly used in electrical contacts.

The nitric acid vapour test is too severe to be used for gold coatings less than 0,6 µm thick. It is also not suitable for coatings that are less noble than gold or platinum, such as palladium and its alloys, or gold-flashed palladium and its alloys.

Several other porosity testing methods are described in ISO 10308 and in the literature (see e.g. Bibliography, [1] and [2]).

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 2064, *Metallic and other inorganic coatings — Definitions and conventions concerning the measurement of thickness.*

ISO 2079, *Surface treatment and metallic coatings — General classification of terms.*

ISO 2080, *Electroplating and related processes — Vocabulary.*

ISO 10308, *Metallic coatings — Review of porosity tests.*

3 Terms and definitions

For the purposes of this International Standard, the terms and definitions given in ISO 2079, ISO 2080, ISO 10308 and the following apply.

3.1
corrosion products
those reaction products emanating from the pores that protrude from, or are otherwise attached to, the coating surface after a vapour test exposure

3.2
significant surface
see ISO 2064:1996, 3.1

NOTE It is essential that the significant surfaces or measuring areas of the part to be tested are indicated on the drawing of the part or by provision of suitably marked samples.

3.3
measuring area
see ISO 2064:1996, 3.2

3.4
metallic coatings
electrodeposits, claddings, or other metallic layers applied to the substrate

NOTE The coating can comprise a single metallic layer or a combination of metallic layers.

3.5
porosity
presence of any discontinuity, crack, or hole in the coating that exposes a different underlying metal

3.6
underplate
metallic coating layer between the substrate and the topmost layer or layers

NOTE The thickness of an underplate is usually greater than 0,8 µm.

4 Apparatus

4.1 Test chamber

This may be any convenient size glass vessel capable of being sealed with a glass lid, such as a glass desiccator of 9 l to 12 l capacity. The ratio of the air space in the chamber (in cubic centimetres) to the nitric acid surface area (in square centimetres) shall be no greater than 25:1.

4.2 Specimen holders or supports

Supports or hangers shall be made from glass, polytetrafluoroethylene or other inert materials. It is essential that the holders are so designed, and the specimens so arranged, that the circulation of the vapour is not impeded. Specimens shall be at least 75 mm from the liquid surface and at least 25 mm from the vessel walls. Also, the measurement areas of the specimens shall be at least 12 mm from each other.

Do not use a porcelain plate or any other structure that would cover more than 30 % of the liquid surface cross-sectional area. This is to ensure that movement of air and vapour within the vessel will not be restricted during the test.

4.3 Stereomicroscope

An instrument having a × 10 magnification, shall be used for pore counting. In addition a movable source of illumination capable of giving oblique lighting on the specimen surface may be useful.

4.4 Oven, capable of operating at a temperature of 125 °C.

4.5 Desiccator, for cooling the samples.

5 Reagent

5.1 Nitric acid, reagent grade, concentrated (69 ± 2) % HNO_3, relative density, (20 °C) 1,39 to 1,42.

6 Safety

Carry out this test method in a chemical fume cupboard, since the gases that are released, mainly when the reaction vessel is opened at the end of each test, are very corrosive and toxic.

Use caution however, to ensure that draughts that are often found in fume cupboards do not cause significant cooling of the chamber walls that could lead to a rise in the relative humidity and acceleration of the test (see 9.3). It is often convenient to enclose the reaction vessel in a box with a loose-fitting cover.

Observe normal precautions in handling corrosive acids. In particular, wear goggles completely enclosing the eyes when handling nitric acid and make eye wash facilities readily available.

7 Procedure

7.1 Avoid any unnecessary handling of the specimens, and then handle only with tweezers, microscope lens tissue or clean, soft cotton or nylon gloves. Prior to the test, using the stereomicroscope (4.3) inspect the samples under ×10 magnification for any evidence of particulate matter. If present, remove the particles by dusting with clean, oil-free air then thoroughly clean the particle-free samples by gently rinsing with solvents or solutions that do not contain chlorofluorocarbons (CFCs), chlorinated hydrocarbons or other known ozone-destroying compounds and air dry with clean air. A hot, analytical reagent grade methanol, ethanol, denatured ethanol or isopropanol dip is often used to speed up the drying.

NOTE If large areas of exposed non-noble metal are present, masking of these areas may be necessary. However, when masking is done with plater's tape, take care not to inhibit the flow of acid vapour to the measurement area.

7.2 The ambient temperature and the temperature of the specimens and solution are 23 °C ± 3 °C at the beginning of the test and maintained throughout the test period.

7.3 The relative humidity (RH) in the test chamber (4.1) shall be within 40 % to 55 % and shall not be allowed to drop below 40 % or to exceed 60 %. If the RH is greater than 60 %, do not run this test.

7.4 Add 500 ml fresh HNO_3 (5.1) to the bottom of the clean and dry test chamber and immediately close the cover. After 30 min ± 5 min, load the samples, using suitable fixtures (4.2), and replace the cover. The ambient RH shall be no greater than 60 %, and preferably between 40 % and 55 %, during both the addition of the HNO_3 and the insertion of the samples (see e.g. Bibliography, [3]).

NOTE 1 At RH values above 60 % metal surfaces tend to adsorb a microscopic layer of water. The thickness of this invisible, but finite (up to 1 µm) water layer will increase with the RH of the air in the vicinity of the sample (see Bibliography, [4] to [7]).

NOTE 2 If a desiccator is used do not grease the rim of the desiccator nor its cover. If desired, press a minimum of three strips of pressure sensitive polytetrafluoroethylene tape (adhesive side down) at equal intervals around the desiccator rim.

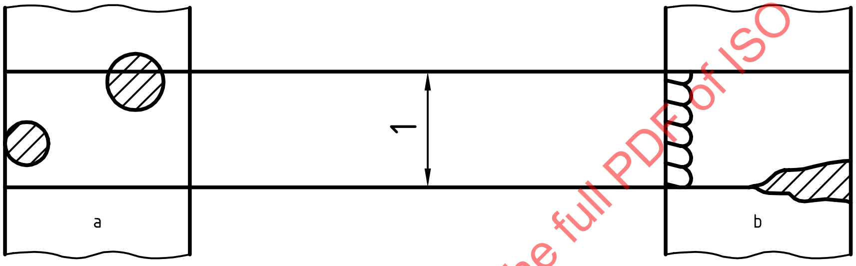
7.5 Unless otherwise specified, the exposure time to nitric acid vapour shall be 60 min ± 5 min. An exposure time of 75 min ± 5 min is also commonly used for gold thicknesses in the 2 μm to 2,5 μm range. A table of convenient exposure times is given in annex A.

NOTE Variations in exposure time with thickness are often recommended because pores in thicker coatings are deeper and their average sizes are smaller than those in thinner coatings. The nitric acid medium would therefore take longer to penetrate an average pore in thicker coatings compared with thinner ones. On the other hand when exposure times are too long, the corrosion products will overlap and impair pore delineation. A detailed discussion of these effects is given in [3].

7.6 Remove the samples at the end of the test and dry in an oven (4.4) at 125 °C ± 5 °C for 30 min to 60 min. Then remove from the oven, and place directly into a desiccator (4.5) containing active desiccant, and allow to cool to room temperature.

NOTE The HNO_3 should be disposed of in a safe, legally acceptable manner.

7.7 Leave the samples in the desiccator until the examination begins. Then open the desiccator slowly, since cooling specimens can create a partial vacuum.



[a] To be counted

[b] Not to be counted if corrosion products exist on edges or are continuous outside the measurement area

Key

1 Measurement area

Figure 1 — Corrosion product counting

7.8 The examination shall take place within 1,5 h of removal from the oven.

7.9 Count individual pores at × 10 magnification using collimated incandescent illumination at an oblique angle below 15°. The pore sites will be delineated by the corrosion products protruding from these sites. As these solids can be transparent in the case of gold-plated nickel or nickel underplate, take great care in counting, particularly with rough or curved surfaces.

7.10 Measure and count a corrosion product when at least three quarters of the corrosion product falls within the measurement area. Do not count corrosion products that initiate outside the measurement area but fall within it, and that are irregular in shape (see Figure 1).

7.11 Occasionally blisters may form with gold electroplated on nickel or a nickel underplate. This may occur at pore sites and may be due to entrapment of corrosion products beneath the plating. Count the blisters as pores.

7.12 It is also good practice to make duplicate readings of each area, and then average the two readings.

7.13 Define pore size by the longest diameter of the corrosion product. Corrosion products smaller than 0,05 mm are generally not resolvable at × 10. Do not count them as pores, even if they are observed at higher magnification.

NOTE If the corrosion products are being tabulated according to size, a useful sizing technique is to tabulate the pores in accordance with three size ranges. These are (approximately): 0,12 mm diameter or less, between 0,12 mm and 0,40 mm diameter, and greater than 0,40 mm diameter. A graduated reticule in the microscope eyepiece is useful as an aid to counting and sizing.

8 Precision

The precision of this test method is being investigated with gold-plated electrical contacts having a nickel underplate. Inter-laboratory results from a series of four runs in each laboratory with a test of similar severity gave coefficients of variation of less than 20 % in each of three participating laboratories [4]. However, poorer precision was obtained when the results of the different laboratories were compared.

9 Test report

The test report shall include the following information:

a) reference to this International Standard, i.e. ISO 14647;

b) the nature of the substrate and any intermediate underlayers;

c) the nature of the coatings, i.e., electrodeposit or claddings;

d) composition of the test solution;

e) the relative humidity of the test chamber (see 7.3);

f) the ambient relative humidity (see 7.4);

g) the duration of exposure in the test environment (see 7.5);

h) the results obtained, i.e., the number, location, colour and size of pore corrosion;

i) the acceptable number, location, colour and size of pore-corrosion products specified in the relevant product standard.

Annex A
(informative)

Time of exposure to nitric acid vapour

The exposure times recommended in this International Standard are those that are sufficiently long to produce corrosion products greater than 0,05 m in diameter, but short enough to prevent significant overlap of these products. If the exposure time is too short, the corrosion products at many pore sites can be too small to be easily seen at × 10 magnification. However, if the exposure time is too long, the corrosion products can overlap or coalesce, thereby impairing the ability to delineate the individual pore sites [2].

Table A.1 may be helpful in specifying exposure times for those gold coatings that are normally applied to electrical contacts.

Table A.1 — Exposure time for gold coatings

Gold thickness µm	**Exposure time to nitric acid vapour** min
< 0,6	
0,6 to 2,0	60 ± 5
2,0 to 2,5	75 ± 5