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Metallic coatings — Porosity tests — Porosity in gold or palladium coatings by sulfurous acid/sulfur dioxide vapour

Revêtements métalliques — Essais de porosité — Porosité des revêtements d'or ou de palladium par essai à l'acide sulfureux/vapeur de dioxyde de soufre

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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 15721 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 7, *Corrosion tests*.

Annex A of this International Standard is for information only.

Introduction

The test method makes use of concentrated sulfurous acid (H_2SO_3), which emits sulfur dioxide (SO_2) gas according to the equilibrium reaction:

$$H_2SO_3 = SO_2 + H_2O$$

WARNING — The sulfur dioxide gas that is released by the sulfurous acid is toxic, corrosive and irritating. The sulfurous acid shall only be handled and used in a fume cupboard. Prior to starting the test, suitable preparation shall be made to dispose of approximately 200 ml to 500 ml of sulfurous acid used in the test.

The procedure is similar to one first proposed by Lee and Ternowski [4]. Exposure periods may vary, depending upon the degree of porosity to be revealed. Reaction of the gas with a corrodible base metal at pore sites produces reaction products that appear as discrete spots on the gold or palladium surface. Individual spots are counted with the aid of a lens or low-power stereomicroscope.

This test method is suitable for coatings containing 95 % or more of gold or palladium on substrates of copper, nickel and their alloys which are commonly used in electrical contacts.

This test is considered destructive because it reveals the presence of porosity by contaminating the surface with corrosion products and it undercuts the coating at pore sites or at the boundaries of the unplated areas. Any parts exposed to this test shall not be placed in service.

The present test method is capable of detecting virtually all porosity or other defects that could participate in corrosion reactions with the substrate or underplate. The test is rapid, simple and inexpensive. In addition, it can be used on contacts with a complex geometry such as pin-sockets (it may be useful to open contact structures with deep recesses to permit reaction of the sulfur dioxide with the interior significant surfaces).

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WARNING — This International Standard does not purport to address all of the safety concerns, 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 determine the applicability of regulatory limitations prior to use. For specific hazard statements, see clause 6.

1 Scope

This test method covers equipment and methods for determining the porosity of gold or palladium coatings, particularly electrodeposits and clad metals used on electrical contacts.

It is designed to show whether the porosity level is less than or greater than a given value which by experience is considered by the user to be acceptable for the intended application.

This porosity test 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 this test cannot be recommended for the prediction of the electrical performance of contacts unless correlation is first established with service experience.

This test is intended to be used for quantitative descriptions of porosity (such as number of pores per unit area or per contact) on coatings that have a sufficiently low pore density so 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/cm^2. Above this value the tests are useful for the qualitative detection and comparisons of porosity.

A variety of other porosity testing methods are described in the literature [5],[6]. Other ISO porosity test methods are described ISO 10308, ISO 12687, ISO 14647 and ISO 15720.

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 2064, ISO 2079, ISO 2080 and ISO 10308 as well as 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

metallic coatings

includes platings, claddings or other metallic layers applied to the substrate

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

3.3

porosity

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

4 Apparatus

4.1 Test chamber, of any convenient size, made of glass or acrylic resin with a gas-tight lid, such as a glass desiccator of 9 l to 10 l, capacity. The ratio of chamber volume in cubic centimeters to the generating solution (sulfurous acid) surface area in square centimeters shall not exceed 25 ± 1.

4.2 Specimen holders, or supports, of glass, polytetrafluoroethylene, or other inert materials. It is essential that the specimens be so arranged that they do not impede gas circulation. Specimens shall not be closer than 25 mm from the wall and 75 mm from the solution surface. The measurement areas of the specimens shall be at least 12 mm apart.

NOTE 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, 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 is required. An eyepiece is recommended for the convenience in locating the contact area or other significant measurement areas.

5 Reagents

Sulfurous acid (H_2SO_3), "analytical grade reagent", or better, in individual well-sealed bottles.

6 Safety hazards

6.1 Carry out these test procedures in a clean, working fume cupboard. The SO_2 gas that is emitted is toxic, corrosive, and irritating.

6.2 Use caution however, when actually performing the tests that the drafts often found in fume cupboards do not cause significant cooling of the chamber walls which may lead to condensation of water and acceleration of the test. It is often convenient to enclose the reaction vessel in a box with a loose-fitting cover and to keep the box in a fume cupboard during the test.

6.3 Observe normal precautions in handling corrosive acids. In particular, wear eye protection completely enclosing the eyes, and make eye wash facilities readily available.

7 Procedure

7.1 Cleaning

Handle specimens as little as possible and only with tweezers, microscope-lens tissue, or clean, soft cotton gloves. Prior to the test, inspect the samples under ×10 magnification using the stereomicroscope (4.3) for evidence of particulate matter. If present, such particles shall be removed by blowing them off with clean, oil-free air. If this is not successful, discard the sample and draw a replacement sample from the batch. Then clean the samples with solvents or solutions that do not contain chlorinated hydrocarbons, CFCs or other known ozone-destroying compounds. The procedure outlined in note 1 has given satisfactory results for platings with mild to moderate surface contamination.

NOTE 1 Suggested cleaning procedure.

a) Keep individual contacts separated if there is a possibility of damaging the measurement areas during the various cleaning steps.

b) Clean samples for 5 min in an ultrasonic cleaner which contains a hot (65 °C to 85 °C) 2 % aqueous solution of a mildly alkaline (pH 7,5 to pH 10) detergent.

c) After ultrasonic cleaning, rinse samples under warm running tap water for at least 5 s.

d) Rinse samples ultrasonically for 2 min in fresh deionized water to remove the last detergent residues.

e) Immerse in fresh methanol or isopropanol, and ultrasonically "agitate" for at least 30 s in order to remove the water from the samples.

f) Remove and dry samples until the alcohol has completely evaporated. If an air blast is used as an aid for drying, the air shall be oil-free, clean and dry.

g) Do not touch measurement area of the samples with bare fingers after cleaning.

h) Re-inspect samples (under ×10 magnification) for particulate matter on the surface. If particulates are found, repeat the cleaning steps. Surface cleanliness is extremely important: contaminants, such as plating salts, organic films and metal flakes may give erroneous indications of defects and are unacceptable.

NOTE 2 Omit the cleaning steps for samples with corrosion-inhibiting, lubricant coatings, or both, if it is desired to determine the efficacy of these coatings in the SO_2 atmosphere.

7.2 Test temperature

The test temperature shall be 23 °C ± 3 °C, unless otherwise specified and the relative humidity in the immediate vicinity of the test chamber shall be no greater than 60 %. If the relative humidity is greater than 60 % do not run the test.

7.3 Sequence of operations

a) Add the sulfurous acid (H_2SO_3) carefully to the bottom of the clean and dry test chamber in a fume cupboard, insert the samples, using suitable fixtures (4.2) and replace the lid. The ambient relative humidity shall be no greater than 60 % while H_2SO_3 is added and the samples are inserted.

b) Expose the samples to the test environment for 2 h for gold coating thicknesses of 1,25 μm or greater, 90 min for gold thicknesses less than 1,25 μm and 60 min for palladium coatings.

c) Remove the samples at the end of the test period, and allow them to stabilize at low humidity in the fume cupboard for 10 min ± 5 min.

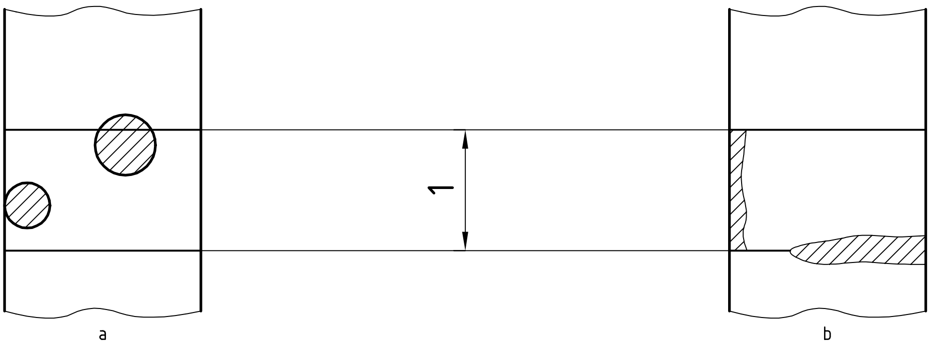
d) Place samples (preferably with their holding fixture) into an air circulating oven, maintained at 125 °C ± 5 °C, for 15 min ± 5 min, for the development of the pore corrosion products for easier examination.

e) Discard the sulfurous acid in a safe manner at the end of each day. It may be re-used for several consecutive tests on one day provided the chamber is kept closed between test runs, and no discoloration or contamination is visible to the naked eye.

8 Examination

8.1 Count individual pore corrosion products at ×10 magnification, using collimated incandescent illumination at an oblique angle below 15°. They are delineated by the corrosion products protruding from the pore sites. The solids may be transparent, especially in the case of gold or palladium on nickel; exercise great care on particularly rough or curved surfaces.

A corrosion product shall be measured and counted when at least one half of the corrosion product falls within the measurement area. Unless otherwise specified, corrosion products which initiate outside the measurement area but fall within, and are irregular in shape shall not be counted. See Figure 1. However, for small measurement areas, or where the migrating pore-corrosion product covers a significant portion of this area, the presence of such products shall be recorded.



Key

1 Measurement area

a To be counted.

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

Figure 1 — Corrosion products at boundaries of measurement area

NOTE The following hints may be useful as a counting aid.

a) Count only corrosion products that protrude above the surface. Stains are not considered porosity within the scope of this specification.

b) Loose contamination that can easily be removed by gentle air dusting is not considered corrosion products.

c) Move sample around under the light to vary the angle when unsure of a pore. Burnished gold can give the appearance of black spots.

8.2 Pore size shall be defined by the longest diameter of the corrosion product. Unless otherwise specified, corrosion products less than 0,05 mm in diameter shall not be counted. A graduated reticule in the microscope eyepiece is useful as an aid for counting and sizing.

NOTE A useful sizing technique is to tabulate the pore corrosion products in accordance with three size ranges. These are (approximately): a) 0,12 mm diameter or less, b) between 0,12 and 0,40 mm diameter, and c) greater than 0,40 mm diameter.

8.3 The acceptable number, sizes and locations of the pore corrosion products shall be as specified on the appropriate drawing or specification, or as agreed upon by the producer and user.

9 Precision

The precision of this test method is being investigated with gold-plated electrical contacts with a nickel underplate. Round-robin results from a series of four runs in each laboratory with an SO_2 test of greater severity gave coefficients of variation of less than 20 % in each of three participating laboratories. However, poorer precision was obtained when the results of the different laboratories were compared.

10 Test report

The test report shall contain the following information:

a) reference to this International Standard, i.e. ISO 15721;

b) the cleaning procedure used;

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

d) the nature of the coating;

e) the test solution used;

f) the relative humidity (see 7.2);

g) the duration of exposure to the test environment;

h) the result obtained, i.e. the number, location 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)

Significance and use

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 inlay or 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.

Palladium coatings are sometimes specified as alternatives to gold on electrical contacts and similar electrical component surfaces, both as electrodeposits and as inlay or clad metal. This test method is particularly suitable for determining porosity in palladium coatings, since the reactive atmosphere that is used does not attack the palladium if the specified test conditions are followed. In contrast, palladium coatings are attacked by nitric acid (HNO_3) and other strong oxidizing agents, so ISO 14647 cannot be used for determining the porosity in such coatings.

To have these coatings function as intended, porosity, cracks and other defects in the coating that expose substrates and underplates must 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 used. 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. When present, the location of pores on the surface is also 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 number to be determined. Contact surfaces are often curved or irregular in shape and testing methods should be suitable for them. In addition, the severity of porosity-determining tests may vary from procedures capable of detecting all porosity to procedures that detect only highly porous conditions.

The relationship of porosity levels to contact behaviour, revealed by particular tests, must 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 in the contact zone may be acceptable for others.

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