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Metallic and other inorganic coatings — DC magnetron sputtered silver coatings for engineering purposes — Measurement of coating adhesion

Revêtements métalliques et autres revêtements inorganiques — Revêtements d'argent pulvérisés par magnétron à courant continu à des fins techniques — Mesurage de l'adhérence des revêtements

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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Contents

Page

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Foreword

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This document was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*.

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Introduction

Silver coatings are often specified for their extremely good electrical conductivity for optical, electronic and electrical engineering applications. Silver coatings for optical and electronic applications have the minimal thickness of 0,1 µm to 1,0 µm depending on the applications type and demand from customers.

The adhesion test methods of the Ag coatings deposited by DC magnetron sputtering method, given in this document, are considered to have an adequate accuracy when properly used with test specimens suitable for the particular method.

The method chosen shall be one which is expected to yield the most reliable results considering the coating thickness, shape and size of the coating material, the basis material and its roughness, etc.

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Metallic and other inorganic coatings — DC magnetron sputtered silver coatings for engineering purposes — Measurement of coating adhesion

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1 Scope

This document specifies the test methods for coating adhesion of sputtered silver layer coating for electrical, electronic, optical and other engineering applications. Engineering applications are defined as those in which the coating essentially serves a non-decorative purpose.

Although the appearance and serviceability of the sputtered silver coating depends on the condition of the basis material, this document does not specify the condition, finish or surface roughness of the basis material prior to the deposition. Therefore, it is the responsibility of the purchaser to specify the surface finish and roughness of the basis material in order to conform to the product requirements.

This document specifies the test methods of coatings on sheets and the flat objects. However, it does not apply to coatings on screw threads, strip or wire in the non-fabricated form.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 2064, *Metallic and other inorganic coatings — Definitions and conventions concerning the measurement of thickness*

ISO 2080, *Metallic and other inorganic coatings — Surface treatment, metallic and other inorganic coatings — Vocabulary*

ISO 4521, *Metallic and other inorganic coatings — Electrodeposited silver and silver alloy coatings for engineering purposes — Specification and test methods*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 2064, ISO 2080 and ISO 4521 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
sputtering
deposition of material (metal) on a surface by using fast ions to eject particles of it from a target

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3.2
adhesion
state in which coatings and the underlying substrate are held together by interfacial forces which may consist of valence forces or interlocking forces or both

4 Abbreviated terms

DC magnetron direct current magnetron

5 Information to be supplied by the purchaser to the manufacturer

The following information shall be supplied by the purchaser to the manufacturer, for example, in the purchase order or contract, or on engineering drawings:

a) the number of this document, i.e. ISO 21164;

b) the nature, condition and finish of the basis metal, if they are likely to affect the serviceability and/or the appearance of the coatings;

c) the finish required, preferably accompanied by approved samples of the finish;

d) the requirements for thickness and adhesion testing;

e) any special requirements for undercoats;

f) the electrical properties of the coating and the methods of test to be used;

f) the density of the silver coating, if the thickness method requires a density correction (see ISO 4521).

6 Test methods for adhesion

6.1 Burnishing

Burnishing method shall be used in accordance with the method specified in ISO 4521.

Select an area of not more than 6 cm^2 of the significant surface and rub rapidly and firmly for 15 s with a suitable burnishing tool. An agate dental spatula with a handle 60 mm to 100 mm long is suitable. Apply a pressure sufficient to burnish the coating at every stroke, but not so great as to cut through the coating. Examine the specimen for signs of blistering of the coating with a microscope of low magnification.

This method is not applicable to the sputtered silver coatings thicker than 10 um.

6.2 Barrel burnishing

Unless dry burnishing is specified, wet burnish the sample for 40 min in a suitable burnishing machine, for example, using a hexagonal rubber-lined barrel about 250 mm across at 25 r/min. Examine the sample for signs of blistering or peeling using a microscope of low magnification. The parts tested shall show no signs of blistering or peeling of the coating.

Complete batches of electroplated articles may be tested by this procedure, provided a burnished finish is acceptable. Only those parts that fail need to be rejected.

6.3 Peel test

Use the test method given in ISO 4521.

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6.4 Bending test

Place the sample in a bend-testing device with a bending radius of 4 mm (or in the jaws of a vice). Bend the sample through 90° and then bend it back to its original position. Repeat three times. Examine the specimen for signs of detachment of the coating using a microscope with low magnification. Tested specimens shall withstand three bends without detachment of the coating. Failure of the substrate due to micro- or macro-racking shall not be a cause for rejection, provided the coating does not exfoliate.

6.5 Thermal shock

Heat the sample in an oven at a temperature between 200 °C and 300 °C for about 30 min, and quench it by immersing in water at ambient temperature. Examine the coating for signs of blistering or detachment using a microscope of low magnification. The sample shall show no sign of blistering or detachment of the coating.

7 Nano scratch test

7.1 General

This test method is used to evaluate the adhesion of sputtered silver coatings of thickness 0,5 µm to 1,5 µm on steel or copper substrates. The results refer to qualitative tests only. This test method may destroy the parts on which the adhesion is carried out.

7.2 Scratch test equipment

Nano scratch tester with sphero-conical diamond indenter with radius 2 um shall be used.

7.3 Specimens

The scratch test shall be performed on the surface of the coated specimen. The specimen shall be silver sputtered flat object with roughness no more than 5,0 µm. The specimen size for scratch testing varies as the sample holders of scratch testing machines differ. Generally, a specimen size of 20 mm^2 × 20 mm^2 is recommended.

Embedding in a cold or hot mounting medium, such as acrylic resin, is not recommended as specimens might be sensitive to heat or pressure.

Prior to scratch testing, the test specimen should be freed from surface contaminants, such as oil, grease and moisture. Finger prints on the test area should be avoided.

7.4 Scratch test parameters

The linear scratch test with progressive mode shall be used. The scratch load can be varied depending upon the thickness of the silver coating. For example, the silver coating with thickness of 0,1 µm to 2,0 µm, the starting load and final load shall be made of 1 mN and 100 mN, respectively with the loading rate of maximum 100 mN/min.

7.5 Evaluation

Acoustic emission, coefficient of friction and frictional force shall be recorded to evaluate the change in their pattern. Acoustic emission may not be recorded if the silver coating is very thin. The penetration depth shall be used to determine the hardness of the coating qualitatively. Examine the silver plated surface carefully with the optical microscope. Where the adhesion has been poor, several micro-cracks will originate at the point where critical load exceeds or the coating itself will be detached from the substrate.

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