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Metallic and other inorganic coatings — Electrodeposited silver and silver alloy coatings for engineering purposes — Specification and test methods

Revêtements métalliques et autres revêtements inorganiques — Dépôts électrolytiques d'argent et d'alliages d'argent pour applications industrielles — Spécifications et méthodes d'essai

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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 4521 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 3, *Electrodeposited coatings and related finishes*.

This second edition cancels and replaces the first edition (ISO 4521:1985), and also ISO 4522-1:1985, ISO 4522-2:1985 and ISO 4522-3:1988, which have been technically revised.

Introduction

Electrodeposited silver and silver alloy coatings are often specified for their extremely good electrical conductivity, but corrosion protection is often an additional requirement for electrical, electronic and other applications. In many conditions of service, sulfide films may form on the coatings, increasing the contact resistance at the silver electroplated mating surface and making them unsuitable for use in low-voltage electronic circuits. Sulfide films are not especially detrimental to other electronic applications where higher voltage and higher contact pressures are used, because the films are not completely insulating.

Because the appearance and serviceability of electroplated silver coatings depend on the condition of the basis material, agreement should be reached between interested parties that the surface finish and roughness of the basis material are satisfactory for electroplating.

Electroplated silver coatings have been used as bearing surfaces for many decades and are particularly useful where the load-bearing surfaces are not well lubricated.

Electroplated silver coatings have largely replaced electroplated gold coatings on metallic lead frames, the devices that support the majority of silicon chips.

Metallic and other inorganic coatings — Electrodeposited silver and silver alloy coatings for engineering purposes — Specification and test methods

WARNING — This International Standard may not be compliant with some countries' health and safety legislations and calls for the use of substances and/or procedures that may be injurious to health if adequate safety measures are not taken. This International Standard does not address any health hazards, safety or environmental matters and legislations associated with its use. It is the responsibility of the user of this International Standard to establish appropriate health, safety and environmentally acceptable practices, and take suitable actions to comply with any national and international regulations. Compliance with this International Standard does not, in itself, confer immunity from legal obligations.

1 Scope

This International Standard specifies requirements for electroplated silver and silver alloy coatings for electrical, electronic and other engineering applications, including test methods. Engineering applications are defined as those in which the coating essentially serves a non-decorative purpose.

Although this International Standard does not specify the condition, finish or surface roughness of the basis material prior to electroplating, the appearance and serviceability of electroplated silver and silver alloy coatings depend on the condition of the basis material. It is essential that the purchaser specifies the surface finish and roughness of the basis material in order to conform to the product requirements.

This International Standard does not apply to coatings on screw threads or to coatings on sheet, strip or wire in the non-fabricated form.

2 Normative references

The following referenced documents are indispensable for the application 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 1463, *Metallic and oxide coatings — Measurement of coating thickness — Microscopical method*

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 2177, *Metallic coatings — Measurement of coating thickness — Coulometric method by anodic dissolution*

ISO 2178, *Non-magnetic coatings on magnetic substrates — Measurement of coating thickness — Magnetic method*

ISO 3497, *Metallic coatings — Measurement of coating thickness — X-ray spectrometric methods*

ISO 3543, *Metallic and non-metallic coatings — Measurement of thickness — Beta backscatter method*

ISO 3868, *Metallic and other non-organic coatings — Measurement of coating thicknesses — Fizeau multiple-beam interferometry method*

ISO 4516, *Metallic and other inorganic coatings — Vickers and Knoop microhardness tests*

ISO 4518, *Metallic coatings — Measurement of coating thickness — Profilometric method*

ISO 4519:1980, *Electrodeposited metallic coatings and related finishes — Sampling procedures for inspection by attributes*

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

ISO 9587, *Metallic and other inorganic coatings — Pretreatment of iron or steel to reduce the risk of hydrogen embrittlement*

ISO 9588, *Metallic and other inorganic coatings — Post-coating treatments of iron and steel to reduce the risk of hydrogen embrittlement*

ISO 10111, *Metallic and other inorganic coatings — Measurement of mass per unit area — Review of gravimetric and chemical analysis methods*

ISO 10289, *Methods for corrosion testing of metallic and other inorganic coatings on metallic substrates — Rating of test specimens and manufactured articles subjected to corrosion tests*

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

ISO 10587, *Metallic and other inorganic coatings — Test for residual embrittlement in both metallic-coated and uncoated externally-threaded articles and rods — Inclined wedge method*

ISO 12687, *Metallic coatings — Porosity tests — Humid sulfur (flowers of sulfur) test*

ISO 14647, *Metallic coatings — Determination of porosity in gold coatings on metal substrates — Nitric acid vapour test*

ISO 15724, *Metallic and other inorganic coatings — Electrochemical measurement of diffusible hydrogen in steels — Barnacle electrode method*

IEC 60068-2-20, *Basic environmental testing procedures — Part 2: Tests. Test T: Soldering*

3 Terms and definitions

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

4 Information to be supplied by the purchaser to the electroplater

4.1 Essential information

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

a) the number of this International Standard (ISO 4521) and the designation (see Clause 5);

b) the significant surface indicated, for example, on drawings or by the provision of suitably marked samples;

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

d) the position on the surface of unavoidable defects, such as rack marks (see 6.2);

e) the finish required, for example, bright, dull or another type, preferably accompanied by approved samples of the finish (see 6.2);

f) the requirements for thickness, accelerated corrosion and adhesion testing (see 6.3, 6.5 and 6.9);

g) the tensile strength of parts and the requirements for heat treatment before and/or after electrodeposition (see 6.7 and 6.8);

h) sampling methods, acceptance levels or any other inspection requirements if different from those specified in ISO 4519:1980, Clause 7.

4.2 Additional information

If the following additional information is required, it shall be specified by the purchaser in writing, for example, in the contract, purchase order or on the drawing with the definition.

a) the minimum silver content of the coating, details of any alloying elements, the methods of determining the minimum silver content (see 6.6) and presence of residual salts (see 6.16);

b) any cleaning precautions to be followed;

c) any special requirements for undercoats (see 6.15 and Annex A);

d) any requirements for an anti-tarnish treatment, the type of treatment and the test method to be applied (see 6.17);

e) the method of porosity testing to be applied, and the number and location of acceptable pores (see 6.4);

f) the electrical properties of the coating and the methods of test to be used (see 6.10);

g) the microhardness of the coating and the test method to be used (see 6.11);

h) requirements for solderability and the test method to be used (see 6.12);

i) any requirements for wear resistance and the test method to be used (see 6.13);

j) the ductility of the coating and the method of test to be used (see 6.14);

k) any requirements for freedom from contamination of the finished articles (see 6.16);

l) the density of the silver coating, if the thickness method requires a density correction (see Annex B).

5 Designation

5.1 General

The designation shall appear on engineering drawings, in the purchase order, the contract or in the detailed product specification. The designation specifies, in the following order, the basis material, stress-relief requirements, the type and thickness of undercoats, if present; the thickness of the silver coating, heat treatment requirements after electroplating, and the type of conversion coating and/or supplementary treatment.

5.2 Designation specifications

The designation shall comprise the following:

a) the term, Electrodeposited coating;

b) the number of this International Standard, ISO 4521;

c) a hyphen;

d) the chemical symbol of the basis material followed by its standard designation (see 5.3);

e) a solidus (/);

f) if appropriate, the chemical symbol for an undercoat metal followed by a solidus and by a number giving the thickness of the undercoat, in micrometres (see 6.15 and Annex A);

g) a solidus(/);

h) the chemical symbol for silver, Ag, followed by a number in parentheses giving the minimum mass fraction of silver in the coating, expressed as a percent to one decimal place;

i) a number indicating the minimum local thickness, in micrometres, of the silver coating.

5.3 Designation of the basis material

The basis material shall be designated by its chemical symbol, or its principal constituent if it is an alloy. For example:

a) Fe for iron or steel;

b) Zn for zinc alloys;

c) Cu for copper and copper alloys;

d) Al for aluminium and aluminium alloys.

For plateable plastics materials, the letters PL shall be used. and for non-metallic materials, the letters NM shall be used.

It is recommended that the specific material be designated by its standard designation; for example, by its UNS number or the local national equivalent, in parentheses, following the chemical symbol for the basis material. For example, Fe(G43400) is the UNS designation of a high-strength steel (see References. [2] to [8] in the Bibliography).

5.4 Designation of heat treatment requirements

The heat-treatment requirements shall be designated as follows:

a) the letters SR for stress-relief heat treatment prior to electroplating, and/or the letters ER for hydrogen-embrittlement-relief heat treatment after electroplating;

b) in parentheses, the minimum temperature, in degrees Celsius (°C);

c) the duration of the heat treatment, in hours (h).

For example, SR(210)1 designates stress-relief heat treatment at 210 °C for 1 h.

When heat treatment prior to or after electrodeposition is specified, the requirements shall be included in the designation as shown in the examples (see 5.5).

5.5 Examples of designations

A silver coating (Ag) with a minimum thickness of 20 µm on brass (Cu) shall have the following designation:

Electrodeposited coating ISO 4521 - Cu/Ag(98,8)20

A silver alloy coating containing 98,9 % silver, Ag (98,9), with a minimum thickness of 10 µm on steel (Fe) with a nickel undercoat (Ni) of unspecified thickness shall have the following designation:

Electrodeposited coating ISO 4521 – Fe/Ni/Ag(98,9)10

A silver alloy coating on steel containing 99,8 % silver [Ag(99,8)] with a minimum thickness of 10 µm with a nickel undercoat (Ni) that is 5 µm thick shall have the following designation:

Electrodeposited coating ISO 4521 – Fe/Ni5/Ag(99,8)10

A silver coating with a minimum thickness of 15 µm (Ag15) deposited over a copper undercoat that is 5 µm thick (Cu5) on steel that has an ultimate tensile strength of 1 200 MPa heat treated prior to electroplating for stress relief at 200 °C for 3 h, SR(200)3, and after electroplating to reduce the risk of hydrogen embrittlement at 190 °C for at least 12 h, ER(190)12, shall have the following designation:

Electrodeposited coating ISO 4521 – Fe/SR(200)3/Cu5/Ag(98,8)15/ER(190)12

The designation describes the heat treatment and electroplating steps in the order that they are performed. The standard designation of the basis material shall be placed in parentheses immediately after the chemical symbol for steel, Fe, in the above example. It is especially important to know the standard designation of a metal or alloy that is difficult to prepare for electroplating and that is susceptible to hydrogen embrittlement.

6 Requirements

6.1 General

The test procedures specified in 6.2, 6.3, 6.7, 6.8, 6.9 and 6.11 for electroplated silver and silver alloy coatings are to be performed in the absence of any anti-tarnish treatment. The tests specified in 6.6 and 6.10 shall be carried out after the anti-tarnish treatment.

Mercury compounds shall not be used in the pretreatment of basis materials.

6.2 Appearance

Over the significant surface, the electroplated article shall be free from clearly visible defects, such as blisters, pits, roughness, cracks or uncoated areas other than those that arise from defects in the basis material. The electroplated article shall be free from extraneous soil and mechanical damage. On articles where a contact mark is unavoidable, its position shall be specified by the purchaser [see 4.1 d)].

In the case of selectively electroplated articles, the amount of stain at the boundary between the areas of the article that have been electroplated with silver and those that are not electroplated with silver shall be agreed between the parties concerned [see 4.1 e)].

Silver and silver alloy coatings that are to be subsequently machined shall be free from excessive nodulation and treeing at edges, and from other imperfections detrimental to subsequent fabrication.

If required, a preliminary sample with the required finish shall be supplied or approved by the purchaser [see 4.1 e)].

6.3 Thickness

The thickness of the coating specified in the designation shall be the minimum local thickness. The minimum local thickness of the coating shall be measured at any point on the significant surface that can be touched with a ball 20 mm in diameter, unless otherwise specified. The minimum thickness of the silver or silver alloy coating shall be that specified by the purchaser [see 4.1 f)].

When silver and silver alloy coatings are to be machined, the purchaser shall specify the minimum thickness required after machining. If machining is not to be carried out by the electroplater, the purchaser shall specify the minimum thickness required before machining.

The thickness of the coatings shall be measured by an appropriate method selected by the purchaser from those contained in Annex B.

6.4 Porosity

When specified by the purchaser, the parts shall be subjected to one or more of the porosity tests, such as ISO 12687 or ISO 14647, given in ISO 10308 [see 4.2 e)]. The acceptable number and location of pores shall also be specified by the purchaser.

6.5 Accelerated corrosion testing

When the corrosion resistance of the coatings is important, the accelerated corrosion test to be used and the acceptable corrosion rating after testing shall be specified by the purchaser [see 4.1 f)]. The method of rating specimens and manufactured articles shall be in accordance with ISO 10289.

The duration and results of artificial accelerated corrosion tests can bear little relationship to the service life of the coated article and, therefore, the results obtained are not to be regarded as a direct guide to the corrosion resistance of the tested coatings in all environments where these coatings may be used.

6.6 Composition

When the silver content is specified by the purchaser in the designation [see 4.1 a) and 5.2 h)], the purchaser shall also specify the methods to be used for determining the silver content of the coating and presence of residual salts [see 4.2 a)].

6.7 Stress-relief heat treatment prior to electroplating

When specified by the purchaser, steel parts that have an ultimate tensile strength equal to or greater than 1 000 MPa and that contain tensile stresses caused by machining, grinding, straightening or cold-forming operations shall be given a stress-relief heat treatment prior to cleaning and metal deposition. The procedures and classes for stress-relief heat treatment shall be as specified by the purchaser, or the purchaser shall specify appropriate procedures and classes from ISO 9587 [see 4.1 g)].

Steels with oxide or scale shall be cleaned before application of the coatings. For high-strength steels (equal to or greater than 1 000 MPa), non-electrolytic alkaline and anodic alkaline cleaners, as well as mechanical cleaning procedures, are preferred to avoid the risk of producing hydrogen embrittlement during cleaning procedures.

6.8 Hydrogen-embrittlement-relief heat treatment after electroplating

Steel parts having an ultimate tensile strength equal to or greater than 1 000 MPa, as well as surface-hardened parts, shall receive hydrogen-embrittlement heat treatment in accordance with the procedures and classes of ISO 9588, or as specified by the purchaser [see 4.1 g)].

The effectiveness of the hydrogen-embrittlement-relief treatment shall be determined in accordance with ISO 10587 for testing threaded articles for residual hydrogen-relief treatment, and with ISO 15724 for measuring relative, diffusable hydrocarbon concentration in steels, unless otherwise specified by the purchaser.

6.9 Adhesion

Coatings shall pass one or more of the adhesion tests given in Annex C, as specified by the purchaser [see 4.1 f)]. Coatings greater than 125 µm shall pass the shear test included in Annex C.

The preparation of metallographic specimens for the microscopical determination of thickness may give an indication of poor adhesion, because grinding and polishing can cause separation of the coating from the substrate which can be observed in the microscope.

To avoid poor coating adhesion, it is common practice to apply thin silver coatings (strikes) from electroplating solutions especially formulated to prevent chemical deposition of silver onto the basis material. For electroplating certain alloys, a gold strike may be required.

Aluminium alloys are given a post-plating heat treatment at 130 °C to improve the adhesion of the coating. This treatment is not recommended for alloys which could suffer deterioration at or above this temperature.

6.10 Electrical properties

If the electrical properties are important to the function of the coating, those properties shall be specified by the purchaser, along with the methods of assessing the properties [see 4.2 f)].

6.11 Microhardness

If the microhardness of the coating is specified [see 4.2 g)], it shall be measured by one of the methods given in ISO 4516.

The microhardness of silver and silver alloy coatings prepared from many types of bright silver electroplating processes decreases during the first 24 h following electrodeposition. In such cases, the hardness determination is not to be made until that time elapses, or alternatively, until an accelerated ageing treatment, for example, heating at 100 °C for 1 h, is carried out.

6.12 Solderability

If specified, the solderability of the silver or silver alloy coating shall be measured by the test described in IEC 60068-2-20, or by an alternative test selected by the purchaser [see 4.2 h)]. The form of test and any artificial ageing treatment carried out before testing shall be related to the intended service of the electroplated product, the details of which shall be agreed between the interested parties.

6.13 Wear resistance

If the wear resistance is important to the function of the coating, the purchaser shall specify the wear resistance and its method of measurement [see 4.2 i)].

6.14 Ductility

When ductility is important, the ductility required and its method of test shall be specified by the purchaser [see 4.2 j)].

6.15 Undercoats

Undercoats applied prior to electroplating with silver or silver alloy coatings may be specified to improve corrosion resistance, adhesion and solderability; to prevent interdiffusion between the basis material and the coating; to prevent contamination of electroplating solutions; or to reduce surface roughness and porosity. When undercoats are specified, they shall meet the requirements given in Annex A [see 4.2 c)].

6.16 Freedom from contamination

The articles electroplated with silver or silver alloy coatings shall be thoroughly rinsed and dried after electroplating.

If specified by the purchaser, the articles shall be subjected to the residual-salt test specified in Annex D [see 4.2 a)]. An increase in conductivity of not more 150 µS/m shall be acceptable, as measured by that test.

6.17 Anti-tarnish treatments

There are a number of anti-tarnish treatments that retard the onset of tarnishing of silver and silver alloys. If such a treatment is required, it shall be specified by the purchaser, together with any associated test procedure. A suitable test is given in ISO 4538 and, if applied, the duration of the test shall be specified by the purchaser.

Packaging materials, such as paper and cardboard, frequently contain small amounts of sulfur compounds that can cause excessive formation of sulfide films on silver and silver alloy electroplated articles. This should be taken into account when articles electroplated with these coatings are packed, stored and transported. Packaging materials shall, therefore, be as free as possible from contamination with sulfur compounds. Specially treated paper strips, that can be included in packages of silver and silver alloy electroplated items, react with sulfides and oxides of sulfur to inhibit tarnish formation during shipment and storage, without affecting the surface properties of the coating adversely.

NOTE Many anti-tarnish treatments increase the electrical resistivity of silver and silver alloy coatings, and also impair solderability.

7 Sampling

A random sample of the size specified in ISO 4519 shall be selected from the inspection lot. The articles in the sample shall be inspected for conformance to the requirements of the International Standard. The lot shall be considered as conforming or non-conforming to each requirement, in accordance with the criteria of the sampling plans in ISO 4519 [see 4.1 h)].

Annex A
(normative)

Requirements for undercoats

A.1 Thickness requirements and measurement

The minimum thickness requirements for electrodeposited undercoats applied prior to silver and silver alloy electroplating are given in Table A.1 for different basis materials. When undercoats are required, the minimum coating thickness shall be that given in the table. The thickness of any undercoat shall be determined by the microscopical method (ISO 1463) or the coulometric method (ISO 2177), when specified.

Table A.1 — Thickness requirements for undercoats for various basis materials

Basis material	Undercoat(s)	Minimum thickness µm
Copper	None	—
Copper alloys (notably free-cutting brass containing lead)	Copper (Cu) or nickel (Ni) may be required.	Specified by the purchaser
Ferrous materials (other than austenitic “stainless” steel)	Nickel (Ni)	10
	Copper plus nickel (Cu/Ni)	10 of Cu plus 5 of Ni (i.e. Cu10/Ni5)
Austenitic “stainless” steel	An acid nickel strike (Wood's bath) will normally be required.	Thin coating to promote adhesion of the silver coating.
Zinc and its alloys	Copper plus nickel (Cu/Ni)	8 of Cu plus 10 of Ni (i.e. Cu8/Ni10)
Aluminium and its alloys	Nickel (Ni)	20
Other basis materials, such as copper alloys, with soldered joints	Nickel and/or copper may be required to meet functional requirements.	Cu10 or Cu2Ni8 or as specified by the purchaser

In Table A.1 above, low-stress nickel undercoats are essential. A Wood's bath consists of 240 g/l of nickel chloride hexahydrate and 85 ml/l of 36 % (mass fraction) hydrochloric acid. The parts are made anodic for not more than 2 min and then cathodic for 6 min, at room temperature with a current density of 300 A/m^2. If current reversal is not feasible, the anodic treatment can be replaced by immersion in the solution without current flowing for 15 min.

An initial copper coating can be used under the nickel coating but the thickness of the nickel shall not be reduced.

Silver-plated items made of copper-base materials on which a nickel undercoat is not used, and silver-plated items made from other materials having a copper undercoat without a subsequent nickel undercoat, are not to be used for continuous service at temperatures higher than 150 °C.

A.2 Chemical symbols for common undercoats

Chemical symbols for some common undercoats are given in Table A.2 and shall be used in the coating designation when required.

Table A.2 — Chemical symbols of common undercoats applied prior to silver electroplating

Chemical symbol	Undercoat
Ni	Nickel
Cu	Copper
Cu/Ni	Nickel applied over copper
SnNi	Tin-nickel alloy

Annex B
(normative)

Methods of measuring the thickness of electroplated silver and silver alloy coatings

B.1 Uncertainty of thickness measurements

The methods cited in B.2 to B.6 have adequate precision; that is, the uncertainty in the measurement is less than 10 %, when properly used with standard-thickness reference materials. If a referee method is required, it shall be specified by the purchaser and shall be selected from the test methods given in B.3, B.4 or B.5. The most reliable method shall be selected taking into consideration the expected coating thickness, the shape and size of the components, the coating material and the basis material.

B.2 Density and thickness calculations

For those thickness test methods where a value of the density of the coating is required, the true density of the silver or silver alloy coating should be used. If the true density is not known, use a suitable arithmetically calculated value. For example, an alloy coating containing 60 % gold (mass fraction) and 40 % silver (mass fraction), would have the following calculated density, in grams per cubic centimetre:

$$\rho = 100/(60/19{,}3 + 40/10{,}5) = 14{,}5$$

where

density of pure gold = 19,3 g/cm^3;

density of pure silver = 10,5 g/cm^3;

ρ is the calculated density of the alloy coating, in grams per cubic centimetre.

A value of the density is required for the beta backscatter, X-ray spectrometric, coulometric, gravimetric and chemical analytical methods described in B.3, B.4 and B.5. If a density is calculated or assumed, the uncertainty in the measurement is likely to be greater than 10 %.

The suppliers of proprietary silver electroplating solutions may be able to estimate the density of the silver coating electrodeposited from their particular solutions, but variations can arise, in practice, due to different operating conditions, ageing of the solution, poor bath maintenance or inclusions of organic matter.

The true values of density for silver coatings electrodeposited from different solutions are compared to values calculated from deposit purity in Table B.1. Table B.1 illustrates that calculated density values may introduce large errors.

Table B.1 — True and calculated densities of silver coatings electroplated from different solutions

Type of solution	Deposit purity % (mass fraction)	True density[a] g/cm^3	Calculated density g/cm^3
Alkaline cyanide (matt)	99,999	10,5	10,5
Alkaline non-cyanide	99,955	9,7	10,499
Alkaline nitrate (bright)	99,020	8,7	10,397

[a] The values mentioned above are for illustrative purposes only and should not be used as density factors when converting from mass per unit area to thickness.

B.3 Non-destructive methods

B.3.1 Beta backscatter, ISO 3543

This method has a measurement uncertainty of less than 10 % for silver thicknesses corresponding to mass per unit area equal to or greater than 1 mg/cm^2 on substrates with an atomic number of less than 35. A value for the density of the silver or silver alloy coating is required for precise measurements.

B.3.2 Magnetic method, ISO 2178

This method is considered to have a measurement uncertainty of less than 10 % for coatings that are from 0,5 µm to 7,5 µm thick.

B.3.3 X-ray spectrometric method, ISO 3497

This method has a measurement uncertainty of less than 10 % over a thickness range of 0,5 µm to 7,5 µm. A value of the density of the coating is required for precise measurements.

B.3.4 Micrometer

Coatings greater than 50 µm thick can be measured directly with a micrometer by measuring the article or part before and after electroplating.

The method is not necessarily valid if two layers of the coating are to be measured and both are included between the platens of the micrometer. Use the microscopical method, ISO 1463, paying particular attention to the requirements for overplating and its limitations when measuring thin coatings.

B.4 Semi-destructive methods

For the purposes of this annex, the term "semi-destructive methods" refers to thickness tests where a minute area, usually less than a few square millimetres, is removed during testing. The area of the coating removed is considered insignificant and can be repaired by replating, or coating with an organic coating before being returned to service.

B.4.1 Coulometric method, ISO 2177

The instrument manufacturer should be asked to recommend the solution to be used to remove the silver or silver alloy coatings by anodic dissolution for the specific substrate material.

NOTE Certain addition agents in electroplating solutions can affect the results of coulometric thickness measurements.

B.4.2 Profilometric method, ISO 4518

This method is considered to have a measurement uncertainty of less than 10 %.

B.4.3 Interferometric method, ISO 3868

This method is considered to have a measurement uncertainty of less than 10 %.

B.5 Destructive methods

B.5.1 Microscopical method, ISO 1463

This method is considered to have a measurement uncertainty of less than 10 % or ± 0,8 µm, whichever is the greater value. With high-resolution microscopes and careful specimen preparation, measurement uncertainties of less than 0,5 µm can be achieved.

B.5.2 Gravimetric method, ISO 10111

B.5.2.1 General principle

Chemical or electrochemical dissolution of the silver or silver alloy coating without attacking the substrate, and determination of the mass of the coating. Calculation of the average thickness of the coating from its area, mass and density.

B.5.2.2 Stripping solutions

Use a solution capable of stripping the silver or silver alloy coating either chemically or electrochemically without attacking the substrate.

Silver can be stripped electrolytically from nickel and steel substrates in a solution containing 90 g/l of sodium cyanide and 15 g/l of sodium hydroxide, at room temperature using steel cathodes and a voltage of 2 V to 6 V.

Silver can be stripped from copper and copper alloys by immersion at 65 °C in a solution containing 19 parts by volume of concentrated sulfuric acid ($\rho = 1{,}84$ g/ml) and 1 part by volume of concentrated nitric acid ($\rho = 1{,}42$ g/ml). The samples to be stripped should be thoroughly dry and water should be kept out of the solution.

Silver can be stripped electrolytically from tin alloys in a solution containing 30 g/l sodium cyanide at room temperature using steel cathodes and a voltage of 4 V.

B.5.2.3 Procedure

Thoroughly degrease a sample of known area, rinsing and drying if necessary. Weigh the sample. Using the recommended stripping solution appropriate to the basis material, completely remove the silver of the silver alloy coating. Thoroughly rinse in running water. Dry and weigh the sample.

B.5.2.4 Thickness calculation

The average coating thickness is calculated from the following equation:

$$d = 10\Delta m/A\rho$$

where

d is the average coating thickness, in micrometres;

Δm is the loss in mass, in milligrams, of the sample;

A is the surface area, in square centimetres, of the coating;

ρ is the density, in grams per cubic centimetre, of the coating (unless the true value is known, a value of 10,6 g/cm^3 shall be used to make the calculation).

B.5.3 Chemical analysis

B.5.3.1 Procedure

Dissolve the silver or silver alloy coating from a sample of known area in a suitable reagent (see B.5.2.2) and determine the mass of silver in the solution by a suitable analytical method.

B.5.3.2 Thickness calculation

The average coating thickness is calculated from the following equation:

$$d = 10^3 m/A\rho w_{Ag}$$

where:

d is the average coating thickness, in micrometres;

m is the mass, in milligrams, of the silver coating;

A is the surface area, in square centimetres, of the coating;

ρ is the density, in grams per cubic centimetre, of the coating; and

w_{Ag} is the silver content, expressed as a percentage of the mass of the coating.

B.6 General test report concerning thickness

The test report shall contain at least the following information:

a) a reference to this annex including an identification of the specific test 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 determinations;

d) any operation not included in this annex or in the International Standards to which reference has been made;

e) any other relevant information requested by the purchaser.

Annex C
(normative)

Adhesion tests

C.1 Burnishing

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 test will only detect extremely poor adhesion and is not recommended for the evaluation of electroplated items for severe engineering service. It is not applicable to coatings thicker than 40 µm.

C.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.

C.3 Peel

This method is recommended for coatings that are greater than 10 µm thick.

Solder a strip of tinned steel or brass to the silver electroplated surface as flat as possible, so that a length of about 15 mm is included in the joint. The strip should be about 10 mm × 75 mm × 0,5 mm. The normal soldering temperature should be used. The solder shall contain about 60 % tin, 38 % lead and 2 % silver. A non-corrosive rosin-based flux should be used. Soldering shall not cause blistering of the coating. Then apply a force to the soldered strip, at right angles to the test piece, that is sufficient to detach the strip. Examine the specimen for signs of detachment of the coating using a microscope of low magnification. The specimens shall not show signs of detachment of the coating and failure shall occur in the solder layer only.

C.4 Bend

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.

C.5 Shear

Cut through the sample with a hacksaw (1 ¼ tooth/mm) with the blade set to cut on the outward stroke. Position the sample so that the cutting stroke pushes the coating away from the basis material. File the cut

edge smooth using a second cut-mill file, moving the file from the basis material towards the coating. Examine the coating for separation from the basis material and for signs of blistering, flaking and peeling using a microscope with low magnification. Tested specimens shall show no signs of separation from the basis material at the interface, nor shall there be any sign of blistering, peeling or flaking.

C.6 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.

C.7 General test report concerning adhesion

The test report shall contain at least the following information:

a) a reference to this annex including identification of the specific test 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 determinations;

d) any operation not included in this annex or in the International Standards to which reference has been made;

e) any other relevant information requested by the purchaser.

Annex D
(normative)

Determination of the presence of residual salts

D.1 Field of application

This test method specifies a process for assessing the freedom from contamination by residual salts of silver and silver alloy coatings for engineering, decorative and protective purposes. It is applicable to parts made entirely of metal and excludes composite parts, for example, those containing both plastics and coated metal.

D.2 General principle

Boiling of the parts in water of known electrical conductivity for a specified time and measurement of any increase in conductivity arising from extraction of residual salts and other conducting impurities.

D.3 Reagent

Water, having a conductivity not greater than 100 µS/m at 20 °C ± 1 °C.

D.4 Apparatus

All glassware used shall be made of borosilicate glass and shall meet the requirements for cleanliness of D.5.2.

D.4.1 Round-bottomed flask, of capacity 250 ml, fitted with a reflux water condenser.

D.4.2 Beaker, of suitable dimensions for the parts being tested, marked at a volume of 100 ml and fitted with a suitable means of reducing undue evaporative loss of water, for example, a water-cooled lid.

D.4.3 Conductivity meter.

D.5 Procedure

D.5.1 Test piece

Take a part or parts, consisting entirely of coated metal and having a total surface area of about 30 cm^2. Depending on their dimensions (see D.5.3), carry out the determination in accordance with D.5.3.1 or D.5.3.2, as appropriate.

Care shall be taken to avoid accidental contamination of the test portion. Clean gloves shall be used to handle the parts under examination.

D.5.2 Check for cleanliness of apparatus

Before carrying out any determination, transfer 100 ml of water (see D.3) to the extraction vessel (see D.4.1 or D.4.2) and boil it gently for 10 min using the appropriate test conditions (see D.5.3.1 and D.5.3.2). Allow the water to cool to 20 °C ± 1 °C and measure its conductivity with the conductivity meter (see D.4.3).

If the value exceeds 100 µS/m, repeat the procedure with a further 100 ml of the water. If the value again exceeds 100 µS/m, discard the extraction vessel and repeat the test using a new vessel.

Reserve satisfactory glassware solely for this determination.

D.5.3 Determination

D.5.3.1 Parts with a cross-sectional width or diameter not greater than 15 mm and not longer than 40 mm

Having checked the apparatus for cleanliness (see D.5.2), transfer the test piece (see D.5.1) to the round-bottomed flask (D.4.1) and add 100 ml of water (see D.3), the conductivity of which has been measured immediately prior to the determination, ensuring that the water completely covers the test piece. Fit the flask with its reflux condenser, bring the water in the flask to the boil and allow it to boil gently for 10 min. Allow the water to cool to 20 °C ± 1 °C and measure its conductivity with the conductivity meter (D.4.3). Calculate an increase in conductivity incurred as a result of the determination.

D.5.3.2 Parts with a cross-sectional width or diameter greater than 15 mm and longer than 40 mm

Carry out the procedure described in D.5.3.1 but placing the test piece (see D.5.1) in the beaker (see D.4.2) to which water (see D.3) has been previously added to the 100 ml mark. Prevent undue evaporative loss during the determination using, for example, the water-cooled lid (see D.4.2), adding more water (see D.3), if necessary, to replace that lost due to boiling.

D.6 Test report

The test report shall include at least the following information:

a) a reference to this annex;

b) the results and the method of expression used;

c) any unusual features noticed during the determination;

d) any operation not included in this International Standard;

e) any other relevant information requested by the purchaser.

Bibliography

[1] ASTM B 117, *Practice for Operating Salt Spray (fog) apparatus*

[2] ASTM E527-07, *Standard Practice for Numbering Metals and Alloys in the Unified Numbering System (UNS)*

[3] EN 573-3, *Aluminium and aluminium alloy — Chemical composition and form of wrought products — Part 3: Chemical composition*: 1994:08

[4] EN 1706, *Aluminium and aluminium alloys — Castings — Chemical composition and mechanical properties*: 1998-03

[5] CR 13388, *Copper and copper alloys — Compendium of compositions and products*: 1998-11

[6] EN 1412:1995, *Copper and copper alloys — European numbering system*

[7] Stahlschlüssel (steel code), 10th edition 2001, published by Verlag Stahlschlüssel Wegst GmbH, Theodor-Heuss-Strasse 36, 71672 Marbach, Germany, ISBN 3-922599-17-6

[8] EN 10088-1, *Stainless steels — Part 1, List of stainless steels*

[9] ISO 2819, *Metallic coatings on metallic substrates — Electrodeposited and chemically deposited coatings — Review of methods available for testing adhesion*

[10] ISO 3882, *Metallic and other inorganic coatings — Review of methods of measurement of thickness*

[11] ISO 9227, *Corrosion tests in artificial atmosphere — Salt spray tests*

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