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Electroplated coatings of tin — Specification and test methods

Dépôts électrolytiques d'étain — Spécifications et méthodes d'essai

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Foreword

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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.

International Standard ISO 2093 was prepared by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings*.

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This second edition cancels and replaces the first edition (ISO 2093-1973), of which it constitutes a technical revision.

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Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Electroplated coatings of tin — Specification and test methods

0 Introduction

This International Standard specifies requirements for electroplated coatings of tin on fabricated metal articles to protect them from corrosion and to facilitate soldering.

Attention is drawn to legislative requirements that exist in many countries for tin coatings used in the food industry.

Annex C gives additional information as guidance to the user.

It is essential that the purchaser should state the information itemized in 4.1 and, if appropriate, 4.2. Specifying ISO 2093 without this information is insufficient.

1 Scope and field of application

This International Standard specifies requirements for electroplated coatings of nominally pure tin on fabricated metal articles. The coatings may be dull or bright as electroplated or may be flow-melted by fusion after electroplating.

It does not apply to

- a) threaded components;
- b) tin-coated copper wire;
- c) coatings on sheet, strip or wire in unfabricated form, or on articles made from them;
- d) coatings on coil springs;
- e) coatings applied by chemical means (immersion, autocatalytic or "electroless");
- f) electroplating of steels with tensile strength greater than 1 000 MPa¹⁾ (or of corresponding hardness), because such steels are subject to hydrogen embrittlement (see 8.2).

2 References

ISO 1463, *Metallic and oxide coatings — Measurement of coating thickness — Microscopical method.*

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

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

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

ISO 2859, *Sampling procedures and tables for inspection by attributes.*²⁾

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

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

ISO 3768, *Metallic coatings — Neutral salt spray test (NSS test).*

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

ISO 6988, *Metallic and other non-organic coatings — Sulfur dioxide test with general condensation of moisture.*

IEC Publication 68-2-20, *Basic environmental testing procedures — Test T: Soldering.*

3 Definitions

For the purpose of this International Standard, the following definitions apply.

3.1 significant surface: The part of the article covered or to be covered by the coating and for which the coating is essential for serviceability and/or appearance.

(Definition taken from ISO 2064.)

3.2 flow-melting; fusing; flow-brightening; reflowing: A process by which a coating is melted in order to impart desirable properties such as brightness or improved solderability (see clause C.4).

1) 1 MPa = 1 N/mm²

2) At present at the stage of draft. (Revision of ISO 2859-1974.)

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:

- a) the number of this International Standard;
- b) the nature of the basis material (see clause 5);
- c) the service condition number (see 7.1) or the classification code of the coating required (see 7.2);
- d) the significant surface of the article to be electroplated indicated, for example, by drawings or by the provision of suitably marked samples;
- e) the sampling procedure to be adopted (see clause 6);
- f) the positions where unavoidable contact marks and other defects are acceptable (see 10.1);
- g) the method of adhesion testing to be used (see 10.3).

4.2 Additional information

The following additional information may be required and, if so, shall be specified by the purchaser:

- a) any heat treatment required (see clause 8);
- b) any requirements for porosity testing (see 10.4);
- c) any requirement for solderability testing and the test methods and conditions to be applied (see 10.5);
- d) any special requirements for undercoats (see clause 9);
- e) a sample showing the required finish (see 10.1);
- f) any special pretreatment required;
- g) any specific requirements for the purity of the coating (see clauses 0 and C.5);
- h) any special packaging requirements for plated components;
- i) any special post-plating treatment.

5 Basis material

This International Standard specifies no requirements for the condition, finish or surface roughness of the basis material prior to electroplating (see C.2.1).

6 Sampling

Sampling procedures are specified in ISO 2859 and ISO 4519.

The method of sampling and acceptance levels shall be agreed between purchaser and supplier.

7 Classification

7.1 Service condition number

The service condition number indicates the severity of the service conditions in accordance with the following scale:

- 4: exceptionally severe — for example service outdoors in severe corrosive conditions or contact with food or drink where a complete cover of tin has to be maintained against corrosion and abrasion (see C.1.1).
- 3: severe — for example service outdoors in typical temperate conditions
- 2: moderate — for example service indoors with some condensation
- 1: mild — for example service indoors in dry atmospheres or applications where solderability is the primary requirement

NOTE — See 10.2, which gives guidance on the relation between service condition number and minimum thickness.

When specifying the service condition number or coating classification code, it should be noted that tin is susceptible to damage in abrasive environments or in those containing certain organic vapours. See also C.1.1.

7.2 Coating classification code

The coating classification code shall consist of four parts, the first two of which shall be separated by an oblique stroke, as follows:

a/b c d

where

- a indicates the chemical symbol for the basis metal (or for the main constituent if an alloy);
- b indicates the chemical symbol for the undercoat metal (or for the main constituent if an alloy) followed by a figure for its minimum coating thickness, in micrometres, and is omitted if no undercoat is required [see 4.2 d)];
- c indicates the chemical symbol for tin, Sn, followed by a figure for its minimum thickness, in micrometres;
- d indicates the surface finish, by the symbol **m** if the coating is matt, or **b** if it is bright electroplated or **f** if it is flow-melted.

An example is

Fe/Ni 2.5 Sn 5 f

which represents an iron or steel basis metal, with a 2,5 µm nickel undercoat, tin electroplated to a coating thickness of 5 µm and flow-melted.

8 Heat treatment of steel

8.1 Stress relief before electroplating

Severely cold-worked steel parts shall be stress relieved before electroplating by heating for 1 h at 190 to 220 °C.

The properties of some steels which have been carburized, flame-hardened or induction-hardened and subsequently ground would be impaired by this treatment and shall instead be stress relieved at a lower temperature, for example at 130 to 150 °C for not less than 5 h.

8.2 Hydrogen embrittlement relief after electroplating

Because diffusion of hydrogen through tin is very slow, heat treatment for hydrogen embrittlement relief after electroplating is impractical.

9 Requirements for undercoats

Undercoats may be necessary on certain basis materials for any of the following reasons:

- to prevent diffusion (see C.2.2 and C.2.3);
- to retain solderability (see C.2.2, C.2.3 and C.2.4);
- to ensure adhesion (see C.2.4 and C.2.5);
- to improve protection against corrosion.

Care should be taken to select an undercoat or undercoat system that will not confer undesirable properties such as embrittlement of the basis material or finished article. For example the use of highly stressed nickel should be avoided.

If the basis material is a copper alloy containing zinc as an alloying constituent, and solderable properties are required, a nickel or copper undercoat of minimum local thickness 2,5 µm is essential in addition to the specified coating thickness of tin (see C.2.3); such coatings may also be necessary to retain good appearance and adhesion.

If an undercoat is specified, its nature (see annex C) and minimum local thickness (see 10.2) shall be specified by the purchaser.

The thickness of the undercoat or undercoats shall be measured by the appropriate method specified in annex A.

10 Requirements for coatings

10.1 Appearance

When examined by the unaided eye or corrected vision the significant surfaces of the electroplated article shall be free from any visible defects such as blisters, pits, roughness, cracks or unelectroplated areas, and shall not be stained or discoloured.

The acceptability and positions of unavoidable contact marks and defects on non-significant surfaces shall be specified by the purchaser.

The finished article shall be clean and free from damage. The surface shall be of a smooth texture, free from nodules and, where fused, shall be free from dewetted areas.

If necessary, a sample showing the required finish shall be supplied or approved by the purchaser.

10.2 Thickness

Tin coatings are classified by thickness and for each service condition (see 7.1), minimum values are specified in the table (see also C.3.2).

The thickness of the coating shall be measured over a reference area (see ISO 2064) by the appropriate method given in annex A on any part of the significant surface that can be touched with a 20 mm diameter ball. In the case of articles having a significant surface area of 100 mm² or greater, the minimum thickness shall be regarded as the minimum value of local thickness. In the case of articles having a significant surface area of less than 100 mm², the minimum thickness shall be regarded as the minimum value of average thickness.

In the case of printed circuit boards with electroplated-through holes, the requirements shall apply to the surfaces within the holes, as well as to the areas that can be touched with a 20 mm diameter ball.

In the case of flow-melted coatings, the thickness requirements apply to the as-electroplated condition, prior to flow-melting. (See C.3.2., clause C.4 and annex A.)

Table — Coating thicknesses

Service condition number	Copper basis materials ¹⁾		Other basis materials ²⁾	
	(Partial) classification code	Minimum thickness	(Partial) classification code	Minimum thickness
		µm		µm
4	Sn 30	30	Sn 30	30
3	Sn 15	15	Sn 20	20
2	Sn 8	10	Sn 12	12
1	Sn 5	5	Sn 5	5

1) Attention is drawn to the essential requirement in clause 9 for undercoats on copper alloy basis materials that contain zinc as an alloying constituent.

2) See C.2.4 and C.2.5 regarding the need to undercoat certain basis materials.

In case of dispute, the referee methods are as given in A.0.2.

10.3 Adhesion

When tested by one of the methods described in annex B, as specified by the purchaser, the coating shall not show signs of detachment.

10.4 Porosity

If specified by the purchaser, coatings having a minimum thickness of 10 µm or greater shall be subjected to a test as follows:

- a) for ferrous basis materials, the test given in ISO 3768;
- b) for non-ferrous basis material, the test given in ISO 6988.

In either case, there shall be no evidence of corrosion of the substrate when viewed with a magnification of X3 (see C.1.1).

10.5 Solderability (see clause A.2)

10.5.1 General materials and piece parts

If specified by the purchaser, solderability shall be tested in accordance with method 1 of test Ta of IEC Publication 68-2-20, using non-activated flux.

If accelerated ageing before the test is required, the procedure shall be specified by the purchaser.

10.5.2 Printed circuit boards

If specified by the purchaser, a coating complying with this International Standard on printed circuit boards shall be tested in accordance with test Tc of IEC Publication 68-2-20.

If accelerated ageing before the test is required, the procedure shall be specified by the purchaser.

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Annex A

Determination of coating thickness

(This annex forms an integral part of the Standard.)

A.0 Introductory notes

A.0.1 Routine methods

All the methods given in this annex are those which are considered to have an adequate accuracy when properly used with samples suitable for the particular method. The method chosen for routine test purposes shall be one which is expected to yield the most reliable results considering such factors as coating thickness, shape of component, size of component, coating composition, basis material, etc.

Other test methods may be used if it can be demonstrated that they are as good as or better than the methods given in this annex for the particular application.

A.0.2 Referee methods

A.0.2.1 General

In cases of dispute, the methods designated for referee purposes shall be in accordance with A.0.2.2. to A.0.2.6. For the coulometric and analytical methods, the density of the tin shall be taken as 7,30 g/cm³ even though this may result in a thickness value less than the true thickness.

A.0.2.2 Local thickness greater than 9 µm

Use the microscopical method specified in A.1.1.

A.0.2.3 Local thickness less than 9 µm

Use the coulometric method specified in A.1.2 if the coating surface is sufficiently smooth and flat so that there is no leakage of the electrolyte at the cell-probe; otherwise use the microscopical method specified in A.1.1.

NOTE — For the coulometric measurement of undercoats, it is essential to remove the tin first. This can be accomplished by the coulometric dissolution of the tin coating or by stripping as described for the analytical method specified in clause A.2.

A.0.2.4 Average thickness of tin on copper, nickel or steel

Use the analytical method specified in A.2.

A.0.2.5 Average thickness of undercoat and of tin on undercoats on basis material such as aluminium

Use the coulometric method specified in A.1.2 if the coating surface is sufficiently smooth and flat so that there is no leakage of the electrolyte at the cell-probe; otherwise use the microscopical method specified in A.1.1. The microsection shall traverse the centre of the test specimen, and at least five evenly spaced measurements shall be made along the microsection.

A.0.2.6 Thickness of tin in electroplated-through holes of circuit boards

Use the microscopical method specified in A.1.1. The microsection shall be parallel to the axis of the hole and perpendicular to the surface where the coating or layer is to be measured (see ISO 1463).

A.1 Measurement of local thickness

A.1.1 Microscopical method

Use the method specified in ISO 1463, with the overplating procedure, overplating with not less than 10 µm of copper.

This method is stated to have an accuracy of $\pm 0,8$ µm or, for thicknesses greater than 25 µm, to within 5 %.

A.1.2 Coulometric method

Use the method specified in ISO 2177. This method is stated to be normally accurate to within 10 %.

A.1.3 Beta backscatter method

Use the method specified in ISO 3543, which requires the equipment and its operation to be such that the coating thickness can be determined to within 10 % of its true value; this accuracy is dependent on the mass per unit area of the coating and the effective atomic number of the basis material.

A.1.4 X-ray spectrometric method

Use the method specified in ISO 3497, which requires the instrument, its calibration and its operation to be such that the coating thickness can be determined to within 10 % of its true value.

A.2 Measurement of average thickness

A.2.1 Principle

A suitable coated specimen (or number of specimens, if small) of known surface area is cleaned, weighed, stripped of its coating by chemical dissolution and re-weighed.

The method is not generally suitable for coatings on small parts or on certain metals (see C.2.5). In appropriate cases, the average of a number of microsection determinations should be used for the determination of average thickness (see ISO 2064).

A.2.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

A.2.2.1 For stripping from iron basis materials and nickel undercoats

Dissolve 20 g of antimony trioxide in 1 000 ml of cold concentrated hydrochloric acid ($\rho = 1,16$ to $1,18$ g/ml).

NOTE — Articles stripped using this solution may not be suitable for reprocessing.

A.2.2.2 For stripping from copper and copper alloys

Hot (minimum 90 °C) concentrated hydrochloric acid ($\rho = 1,16$ to $1,18$ g/ml).

A.2.3 Test specimen

Use a test specimen or specimens of total surface area sufficient to give a mass loss of not less than $0,1$ g, the area of which can be measured to an accuracy of 2% or better. Remove all soil from the test specimen by washing it in a suitable solvent or by vapour degreasing.

A.2.4 Procedure

A.2.4.1 For iron basis materials and nickel undercoats on copper and copper alloys

Weigh the cleaned test specimen (A.2.3) to the nearest $0,001$ g, immerse it in the stripping solution (A.2.2.1) and leave

immersed for 2 min after the evolution of gas has ceased. Remove from the solution, wash thoroughly in running water, brushing off any smut. Dry and weigh to the nearest $0,001$ g.

A.2.4.2 For copper and copper alloys

Weigh the cleaned test specimen (A.2.3) to the nearest $0,001$ g, immerse it in the stripping solution (A.2.2.2) and remove immediately the coating has completely dissolved. Wash thoroughly in running water, dry and weigh to the nearest $0,001$ g.

A.2.5 Expression of results

The coating thickness, in micrometres, is given by the formula

$$\frac{(m_1 - m_2)}{A} \times 137 \times 10^3$$

where

m_1 is the mass, in grams, of the test specimen before stripping;

m_2 is the mass, in grams, of the test specimen after stripping;

A is the surface area, in square millimetres, of the test specimen;

137×10^3 is a factor based on the density, $7,30$ g/cm³, of tin.

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Annex B

Adhesion tests

(This annex forms an integral part of the Standard.)

B.1 Burnishing test

Apply the method described in ISO 2819 to an area of not more than 600 mm² of the significant surface.

NOTE — An agate dental spatula with a handle 60 to 100 mm long and agate blade 30 to 50 mm long, 5 to 10 mm wide, and sharpened to a slightly radiused edge has been found to be a very satisfactory burnishing tool.

B.2 Bend test

Place the sample in a suitable machine, capable of applying a bend of radius 4 mm to the sample (or in the jaws of a suitable

vice). Bend the sample through 90° and back to its original position. Carry out this procedure three times. Examine the specimen for signs of detachment of the coating.

B.3 Thermal shock test

CAUTION — This test may have an adverse effect on the mechanical properties of the article tested. Accordingly, the thermal shock test specimen shall not be used for other tests.

Use the method described in ISO 2819.

Annex C

Guidance notes

(This annex does not form part of the Standard.)

These notes are intended to draw the attention of the user to

- a) certain properties of tin which, if not understood, may lead to inappropriate use of the coating;
- b) properties and preparation of the substrate;
- c) electroplating practice.

C.1 Properties of the coating

C.1.1 General

Tin coatings are soft and easily abraded. Some corrosion of tin may be expected in certain outdoor exposure conditions and, therefore, deposit thicknesses considerably greater than those specified for a given service condition may be required. The thicknesses specified in the table are minimum values and the use of thicknesses greater than those specified may be required. In normal indoor exposure, tin gives protection to most metals except, especially on ferrous metals, at discontinuities and pore sites in the coating. The porosity of an electroplated coating is governed not only by its thickness but also by such variables as the condition of the basis material and general electroplating practice, and this should be borne in mind when specifying the porosity test.

Electroplated coatings covered by this International Standard can provide thinner or thicker coatings than those normally obtained by hot-dipping.

C.1.2 Whisker growth

Electroplated tin is liable to the spontaneous growth of metal 'whiskers' (filaments), especially on stressed coatings. If the possibility of whisker growth is considered to be a liability, flow-melting of the coatings or the use of tin-lead alloy coatings should be considered. The use of suitable undercoats, for example nickel, may retard the growth of whiskers.

C.1.3 Allotropic changes

High-purity tin coatings are subject to allotropic change (to α - or grey-tin) if subjected to subzero temperatures. For such conditions the use of tin-lead or other suitable tin alloy coatings should be considered.

C.2 Properties and preparation of the basis material

C.2.1 Surface condition

The surface condition of the coating will depend partly on the surface condition of the basis material.

C.2.2 Formation of intermetallic compounds

The interdiffusion of the coating with copper and copper-base alloys by a solid/solid diffusion process is dependent on time and temperature, and can lead to darkening and poor solderability with thin coatings. The rate of deterioration depends on the storage conditions but in poor conditions storage life may be only a few months.

C.2.3 Diffusion of zinc

Zinc, from zinc-containing alloys such as brass, diffuses through the tin coating to the surface and degrades the solderability, adhesion and appearance (see clause 9).

C.2.4 "Difficult" basis materials

Some basis materials, for example phosphor-bronze, beryllium-copper and nickel-iron alloys, are difficult to prepare chemically clean because of the nature of the surface oxide film. If solderability is a requirement of the tin coating, an undercoat of nickel or copper of a minimum local thickness of 2,5 μm may be advantageous.