International Standard 7587

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Electroplated coatings of tin-lead alloys — Specification and test methods

Dépôts électrolytiques d'alliage étain-plomb — Spécifications et méthodes d'essai

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Foreword

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International Standard ISO 7587 was prepared by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings.*

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.

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Electroplated coatings of tin-lead alloys — Specification and test methods

0 Introduction

Tin-lead alloy coatings are used extensively in the telecommunications industry and their use has improved the integrity and reliability of soldered electrical circuits. Tin-lead alloy coatings are also known to be more resistant to whisker growth and allotropic change than pure tin.

Annex D 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 7587 without this information is insufficient.

1 Scope and field of application

This International Standard specifies requirements for electroplated coatings of tin-lead alloy containing between 50 % (*m*/*m*) and 70 % (*m*/*m*) of tin (see 10.3). It may be used for tin-lead alloys of other compositions but the properties of such coatings may be different from those of the alloy range quoted.

A classification scheme is included by which the nature of the basis metal and the coating composition, within these limits, can be defined and which also contains provision for flow-melting and bright deposited coatings.

The coatings are intended for use on fabricated metal articles and on printed circuit boards.

It does not apply to

a) threaded components;

b) bearing overlays;

c) coatings on sheet, strip or wire in unfabricated form, or of articles made from them;

d) electroplating of steels with tensile strength greater than 1 000 MPa[1] (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.*

1) 1 MPa = 1 N/mm^2

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

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 D.4).

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) and the requirements for alloy composition (see 10.3);

d) whether verification of the coating composition is required (see 10.3);

e) the significant surface of the article to be electroplated indicated, for example, by drawings or by the provision of suitably marked samples;

f) the sampling procedure to be adopted (see clause 6);

g) the positions where unavoidable contact marks and other defects are acceptable (see 10.1);

h) the method of adhesion testing to be used (see 10.4);

i) any special post-plating treatment (see D.3.1).

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 10.5);

c) any requirement for solderability testing and the test methods and conditions to be applied (see 10.6);

d) any special requirements for undercoats (see clause 9);

e) a sample showing the required finish;

f) any special pretreatment required;

g) any special packaging requirements for plated components.

5 Basis material

This International Standard specifies no requirements for the condition, finish or surface roughness of the basis material prior to electroplating (see D.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 (see clause D.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 and also 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-lead alloys are susceptible to damage in abrasive environments or in those containing certain organic vapours. See also annex D.

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 c)];

c indicates the composition of the coating by means of the symbol, Sn, with the percentage by mass of tin in the coating, followed by a hyphen and the symbol, Pb, 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 5 Sn60-Pb 10 f

which represents an iron or steel basis metal, 5 µm nickel undercoat, tin-lead electroplated with a nominal tin content 60 % (m/m) having a thickness of 10 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-lead coatings is very slow, and also because the coatings would melt at the temperatures required, 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:

a) to prevent diffusion (see D.2.2 and D.2.3);

b) to retain solderability (see D.2.2, D.2.3 and D.2.4);

c) to ensure adhesion (see D.2.4 and D.2.5);

d) 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 (see 10.2 and D.2.3) of tin-lead alloy; such coatings may also be necessary to retain good appearance and adhesion.

If an undercoat is specified, its nature (see annex D) 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 B.

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 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. However, the occurrence of a cellular pattern on the surface may be met but is not detrimental.

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

10.2 Thickness

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

The thickness of the coating shall be measured over a reference area (see ISO 2064) by the appropriate method given in annex B on any part of the significant surface that can be touched

Table — Coating thicknesses

Service condition number	Copper basis materials[1]		Other basis materials[2]	
	(Partial) classification number	Minimum thickness µm	(Partial) classification number	Minimum thickness µm
4	Snx[3]-Pb 30	30	Snx[3]-Pb 30	30
3	Snx[3]-Pb 15	15	Snx[3]-Pb 20	20
2	Snx[3]-Pb 8	8	Snx[3]-Pb 12	12
1	Snx[3]-Pb 5	5	Snx[3]-Pb 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 D.2.4 and D.2.5 regarding the need to undercoat certain basis metals.

3) x is the nominal tin content of the coating.

with a 20 mm diameter ball. In the case of articles having a significant surface area of 100 mm^2 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 mm^2, 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 also apply to the surface within the holes, and not only to the areas that can be touched with a 20 mm diameter ball (see B.0.2.6).

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

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

10.3 Composition

This specification is based on coatings with a tin content in the range 50 to 70 % (m/m) of tin.

The nominal tin content shall be shown in the classification number and the tolerance on the composition indicated in the information to be supplied to the electroplater (see the note).

A suitable method for analysis of the tin-lead coating is given in annex A and shall be used in case of dispute.

NOTE — Annex D gives guidance on the use of other alloy compositions.

10.4 Adhesion

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

10.5 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 materials, 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 X 3 (see clause D.1).

10.6 Solderability (see clause D.2)

10.6.1 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.

10.6.2 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.

Annex A

Analysis of coatings

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

A.1 General

The method described is *not* intended for use on plated components because of the difficulty of ensuring complete removal of the coating from the substrate (see the note).

If a referee method is required for the determination of the coating composition it is necessary to electroplate dummy cathodes under the same processing conditions as the work being processed. The tin content of this coating is determined as specified in clause A.3.

NOTE — For the routine determination of coating composition on plated components, the beta backscatter technique is suitable.

A.2 Preparation of dummy cathodes

A.2.1 Vat electroplated articles

Deposit 25 to 30 µm of the alloy on to a piece of austenitic stainless steel approximately 100 mm × 80 mm × 0,5 mm. The deposit should be easily removable by scraping and peeling. Adherent coatings should be discarded and the process repeated.

A.2.2 Barrel electroplated articles

A.2.2.1 To prepare a stripping solution, add 50 ml of 6 % (m/m) hydrogen peroxide solution to 50 ml of 40 % (m/m) borofluoric acid solution. Use the freshly prepared solution.

A.2.2.2 Include a number of copper test pieces (rods approximately 50 mm × 12 mm diameter are suitable) with the work to be electroplated.

A.2.2.3 After electroplating, weigh, to the nearest 0,001 g, a sufficient number of test pieces to give approximately 0,5 g of deposit. Remove the coating by immersion in 50 ml of the stripping solution (A.2.2.1), rinse, collecting the rinsings in a separate beaker, dry and reweigh. Combine the stripping solution and rinses and determine their tin content as specified in clause A.3.

A.3 Determination of tin

A.3.1 Principle

The tin is reduced to tin(II) and determined iodometrically.

A.3.2 Range

10 to 90 % (m/m) of tin.

Reproducibility ±0,5 % (m/m) of tin.

A.3.3 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity which has been freshly boiled and cooled.

A.3.3.1 Hydrochloric acid, ϱ = 1,16 to 1,18 g/ml.

A.3.3.2 Hydrogen peroxide, 6 % (m/m), solution.

A.3.3.3 Sodium hydrogen carbonate, saturated solution.

A.3.3.4 Iron powder, hydrogen reduced, tin free.

A.3.3.5 Nickel coils, for assay (see A.3.8.1).

A.3.3.6 Starch, 10 g/l, solution.

Make a paste of 1 g of soluble starch and water, and stir into 100 ml of boiling water. Cool before use.

A.3.3.7 Potassium iodate/iodate, standard volumetric solution, for use with alloys containing more than 25 % (m/m) of tin.

Dissolve 6,01 g of potassium iodate, previously dried at 105 °C, in 400 ml of water containing 1 g of sodium hydroxide and 30 g of potassium iodide. Dilute to the mark in a 1 000 ml one-mark volumetric flask (see A.3.8.2).

1 ml of this solution corresponds to 0,010 g of Sn.

A.3.4 Apparatus

All volumetric glassware used should be class A accuracy to the appropriate International Standard.

A 750 ml conical flask fitted with a rubber bung and a suitable method for producing and maintaining an inert atmosphere, for example a Göckel safety trap with sodium hydrogen carbonate solution, a Bunsen valve or the passage of an inert gas (nitrogen, argon or carbon dioxide) from a cylinder with a suitable reducing valve.

A.3.5 Preparation of test solution

A.3.5.1 Vat electroplated articles

Weigh, to the nearest 0,001 g, 0,5 to 0,8 g of the sample for analysis and transfer to a 750 ml conical flask. Add 75 ml of the hydrochloric acid (A.3.3.1) and warm to dissolve, adding a few drops of the hydrogen peroxide solution (A.3.3.2) periodically to aid dissolution.

A.3.5.2 Barrel electroplated articles

Transfer the stripping solution quantitatively to a 750 ml conical flask and add 60 ml of the hydrochloric acid (A.3.3.1).

A.3.6 Procedure

Add sufficient water to the test solution (A.3.5) to give a total volume of 250 ml, add 0,5 g of the iron powder (A.3.3.4), cover and simmer to dissolve.

Add a nickel coil (A.3.3.5) to the flask and insert the rubber bung assembly (A.3.4) into the flask to maintain an inert atmosphere.

Heat the solution to boiling and boil gently for a further 30 min.

Whilst maintaining the inert atmosphere, remove the flask to a sink and cool to below 20 °C.

Remove the bung, add approximately 20 ml of sodium hydrogen carbonate solution (A.3.3.3), 2 to 3 ml of starch solution (A.3.3.6) and titrate with potassium iodate/iodide solution (A.3.3.7) to a permanent blue colour.

Carry out a simultaneous blank determination on the reagents following the same procedure and using the same quantities of reagents, but omitting the test solution.

A.3.7 Expression of results

The tin content of the coating, expressed as a percentage by mass of Sn, is given by the formula

$$\frac{(V_1 - V_0) \times m_1}{m_0} \times 100$$

where

V_0 is the volume, in millilitres, of potassium iodate/iodide solution used for the titration of the blank test solution;

V_1 is the volume, in millilitres, of potassium iodate/iodide solution used for the titration of the test solution;

m_0 is the mass, in grams, of sample taken;

m_1 is the mass, in grams, of tin corresponding to 1 ml of the potassium iodate/iodide solution used.

A.3.8 Notes on procedure

A.3.8.1 A suitable nickel coil is made from a piece of pure nickel sheet approximately 150 mm × 50 mm. A new coil should be etched before use by boiling for 10 min in 50 % (m/m) hydrochloric acid containing 20 % (m/m) of sodium chloride.

A.3.8.2 For the precision generally required for this International Standard, the potassium iodate may be considered to be a primary standard. However, if required, the solutions can be standardized by carrying out the determination in triplicate on about 0,4 g, weighed to the nearest 0,001 g, of 99,9 % (m/m) tin, treating it as a vat electroplated article (A.3.5.1).

Annex B

Determination of coating thickness

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

B.0 Introduction

B.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.

B.0.2 Referee methods

B.0.2.1 General

In cases of dispute, the methods designated for referee purposes shall be in accordance with B.0.2.2. to B.0.2.6. For the coulometric and analytical methods, the alloy composition shall be determined by the method given in annex A. Use the formula given in B.2.5 to calculate the density even though this may result in a thickness value less than the true thickness.

B.0.2.2 Local thickness greater than 9 µm

Use the microscopical method specified in B.1.1.

B.0.2.3 Local thickness less than 9 µm

Use the coulometric method specified in B.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 B.1.1.

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

B.0.2.4 Average thickness of tin-lead over copper, nickel, or steel

Use the analytical method specified in B.2.

B.0.2.5 Average thickness of undercoat and of tin-lead over undercoats or substrates other than copper, nickel or steel

Use the coulometric method specified in B.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 B.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.

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

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

B.1 Measurement of local thickness

B.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 ±0,8 µm or, for thicknesses greater than 25 µm, to within 5 %.

B.1.2 Coulometric method

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

B.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, the effective atomic number of the basis material and any variations in the alloy composition.

B.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.

B.2 Measurement of average thickness

B.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 D.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).

B.2.2 Reagents

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

B.2.2.1 For stripping iron basis materials and nickel undercoats

Dissolve 20 g of antimony trioxide in 1 000 ml of cold concentrated hydrochloric acid (ϱ = 1,18 g/ml).

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

B.2.2.2 For stripping copper and copper alloys

Add 50 ml of 6 % (m/m) hydrogen peroxide solution to 50 ml of 40 % (m/m) borofluoric acid solution. This solution shall be freshly prepared.

B.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 organic solvent or by vapour degreasing.

B.2.4 Procedure

B.2.4.1 For iron basis materials and nickel undercoats

Weigh the cleaned test specimen (B.2.3) to the nearest 0,001 g, immerse it in the stripping solution (B.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.

B.2.4.2 For copper and copper alloys

Weigh the cleaned test specimen (B.2.3) to the nearest 0,001 g, immerse it in the stripping solution (B.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.

B.2.5 Expression of results

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

$$\frac{(m_1 - m_2)}{A} \times 117\,400$$

where

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

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

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

117 400 is a factor based on the density of a 60/40 tin-lead alloy, 8,52 g/cm^3.

NOTE — For coatings of different compositions and hence different densities, the factor should be calculated accordingly.

Annex C

Adhesion tests

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

C.1 Burnishing test

Apply the method described in ISO 2819 to an area of not more than 600 mm^2 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, sharpened to a slightly radiused edge has been found to be a very satisfactory burnishing tool.

C.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.

C.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.