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Metallic and other inorganic coatings — Electrodeposited gold and gold alloy coatings for electrical, electronic and engineering purposes — Specification and test methods

Revêtements métalliques et autres revêtements inorganiques — Dépôts électrolytiques d'or et d'alliages d'or pour usages électrique, électronique et industriels — Spécification et méthodes d'essai

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Contents

Page

Foreword

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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 27874 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 3, *Electrodeposited coatings and related finishes*.

This first edition of ISO 27874 cancels and replaces ISO 4523:1985, ISO 4524-1:1985, ISO 4524-4:1985 and ISO 4524-5:1985, of which it constitutes a technical revision.

Introduction

The engineering uses of electrodeposited gold and gold alloy coatings have expanded with the growth of the electrical and electronic industries. Low voltages and currents, dry circuits and microwave frequencies require low-resistance interconnection systems, connectors and waveguides. Non-tarnishing, low-resistance gold coatings were the logical choice for connectors where the stability of contact surfaces was critical. The need to improve the wear resistance of gold coatings led to the development of new electroplating solutions containing controlled amounts of metallic and non-metallic additives that either changed the composition or altered the crystal structure of the coating. The special needs of the printed-circuit industry led to the development of acid gold electroplating solutions that contained no free cyanide, yielding coatings that are hard, bright and solderable. Formulations for high-speed electroplating up to current densities of 200 A/dm^2 were introduced for continuous strip, stripe or spot gold and gold alloy coatings.

The high cost of gold metal has led to the development of selective and thickness profile plating techniques to limit the use of the metal to the active areas only of the components, where the gold is required. Designers will therefore often specify the area requiring gold electroplating as well as the thickness profile, if required, by reference to suitably marked drawings.

With the introduction of many new gold electroplating formulations and the proliferation of engineering applications, the need for technical standards that specify the requirements of electrodeposited gold and gold alloy coatings, as well as the test methods to ensure that the specified requirements are met, is critical. Composition, appearance, hardness, thickness, purity, porosity, wear resistance, solderability, electrical contact resistance, infrared reflectivity and other properties must be controlled to produce high-quality gold and gold alloy coatings for engineering purposes.

Metallic and other inorganic coatings — Electrodeposited gold and gold alloy coatings for electrical, electronic and engineering purposes — Specification and test methods

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1 Scope

This International Standard specifies the requirements for electrodeposited gold and gold alloy coatings for electrical, electronic and other engineering applications on metallic and non-metallic substrates. It also specifies test methods for measuring the properties of the coatings.

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 gold or gold alloy coatings depends on the condition of the basis material. It is essential that the purchaser specify 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 threaded articles or to coatings on sheet or strip in 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 2819, *Metallic coatings on metallic substrates — Electrodeposited and chemically deposited coatings — Review of methods available for testing adhesion*

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 3882, *Metallic and other inorganic coatings — Review of methods of measurement of thickness*

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

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

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

ISO 4524-2, *Metallic coatings — Test methods for electrodeposited gold and gold alloy coatings — Part 2: Mixed flowing gas (MFG) environmental tests*

ISO 4524-3:1985, *Metallic coatings — Test methods for electrodeposited gold and gold alloy coatings — Part 3: Electrographic tests for porosity*

ISO 4524-6, *Metallic coatings — Test methods for electrodeposited gold and gold alloy coatings — Part 6: Determination of the presence of residual salts*

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 or steel to reduce the risk of hydrogen embrittlement*

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 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*

IEC 60068-2-20, *Environmental testing — Part 2-20: Tests — Test T: Test methods for solderability and resistance to soldering heat of devices with leads*

3   Terms and definitions

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

3.1
gold or gold alloy coating
electrodeposited gold or gold alloy having intentional alloying elements in its composition

3.2
double-layer gold or gold alloy coating
gold or gold alloy coating consisting of two discrete layers of differing gold contents

3.3
multilayer gold or gold alloy coating
gold or gold alloy coating consisting of more than two discrete layers of differing gold contents

4 Information to be supplied to the electroplater

4.1 Essential information

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

a) the designation (see Clause 5);

b) the significant surface of the article, indicated, for example, as dimensioned areas 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 (see Clause 1);

d) the position on the surface of any 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 method of porosity testing to be used and the permitted number and location of acceptable pores (see 6.4);

g) the tensile strength of the part and the requirements for any heat treatment prior to, or after, electroplating (see 6.7 and 6.8);

h) sampling methods, acceptance levels and any other inspection requirements if different from those specified in ISO 4519 (see Clause 7);

i) the requirements for coating thickness, including positions of measurement as indicated on dimensioned drawings (see 6.3);

j) the requirements for adhesion testing (see 6.9).

4.2 Additional information

The following additional information may be required and, if so, shall be specified by the purchaser in writing, for example in the contract or purchase order, or on drawings:

a) the composition of the coating and details of intentional alloying elements and undesirable impurities (see 6.6);

b) any special cleaning procedures to be used;

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

d) any requirements for the composition and thickness of each layer in double or multilayer coatings (see Clause 3);

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

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

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

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

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

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

k)   the agreed mean density of a gold alloy coating if the thickness measurement method requires a density correction (see Annex B);

l)   any requirements for accelerated-corrosion testing (see 6.5);

m)   any other requirements, such as a residual-salts test, (see 6.16).

5   Designation

5.1   General

The designation shall appear on engineering drawings, in the purchase order, in the contract or in the detailed product specification.

The designation specifies, in the following order, the basis metal, the specific alloy (optional), stress relief requirements, the type(s) and thickness(es) of undercoats (when present), the thickness and composition of the gold or gold alloy coating or coatings (when double or multilayer coatings are specified), and supplementary treatments such as heat treatment to reduce susceptibility to hydrogen embrittlement.

5.2   Designation specifications

The designation shall comprise the following:

a)   the term, "electrodeposited coating";

b)   the number of this International Standard (ISO 27874);

c)   a hyphen;

d)   the chemical symbol of the basis metal (see 5.3);

e)   a solidus (/);

f)   if appropriate, the chemical symbol for an undercoat metal followed, if necessary, 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 gold, Au, or the standard designation for a gold alloy, including the symbol of the alloying element and a figure in parentheses giving the mean content of that element expressed as a mass fraction in percent to one decimal place;

i)   a number giving the minimum local thickness requirement for the gold or gold alloy coating in micrometres;

j)   for double and multilayer gold coatings, repeat h) and i), followed by a solidus (/), for each subsequent gold or gold alloy coating required.

5.3   Designating the basis material

The basis material shall be designated by its chemical symbol or the chemical symbol of its principal constituent if an alloy, for example:

Fe for iron and steel;

Zn for zinc alloys;

Cu for copper and copper alloys;

Al for aluminium and aluminium alloys.

In the case of non-metallic materials, the letters NM shall be used.

A specific alloy shall be identified by its standard designation, for example its UNS number or the local, national, equivalent placed between the symbols < >.

For example, Fe<G43400> is the UNS designation for a particular high-strength steel (see Reference [5] in the Bibliography).

5.4 Designation of heat treatment requirements

The heat treatment requirements shall be in brackets and designated as follows:

a) the letters SR for stress relief heat treatment prior to electroplating, the letters ER for hydrogen embrittlement relief heat treatment after electroplating, and the letters HT for heat treatment for other purposes;

b) in parentheses, the minimum temperature, in °C;

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

The structure and composition of gold and gold alloy coatings may be modified and the coating properties substantially altered by heat treatment. Designers should be aware of these effects before specifying gold coatings on high-tensile-strength basis material.

5.5 Examples

A pure gold coating, Au, with a minimum thickness of 5 µm on nickel-electroplated steel, Fe/Ni, will have the following designation:

Electrodeposited coating ISO 27874 – Fe/Ni/Au5

An alloy coating containing 98,0 % gold and 2 % silver, AuAg(2,0), with a minimum thickness of 5 µm on a zinc alloy, Zn, with copper and nickel undercoats will have the following designation:

Electrodeposited coating ISO 27874 – Zn/Cu/Ni/AuAg(2,0)5

An alloy coating containing 99,5 % gold and 0,2 % nickel, AuNi(0,2), with a minimum thickness of 0,5 µm deposited over a pure-gold coating, Au, with a minimum thickness of 1 µm on a Cu alloy will have the following designation:

Electrodeposited coating ISO 27874 – Cu/Au1/AuNi(0,2)0,5

A pure-gold coating with a minimum thickness of 5 µm, Au5, deposited over a copper undercoat that is 5 µm thick, Cu5, on a steel that has an ultimate tensile strength of 1 200 MPa and is 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 12 h, ER(190)12, will have the following designation:

Electrodeposited coating ISO 27874 – Fe/SR(200)3/Cu5/Au5ER(190)12

The designation describes the heat treatment and electroplating steps in the order in which they are performed. The standard designation of the basis material could be placed after the chemical symbol, 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 and test methods

6.1 General

Gold and gold alloy coatings normally consist of a single layer of gold metal, usually with a strike undercoat of unspecified thickness, but double or multilayer coatings may be specified by the purchaser [see 4.2 c)].

6.2 Appearance

Although this International Standard does not specify the condition, finish or surface roughness of the basis material prior to electroplating, the appearance of electroplated gold and gold alloy coatings depends on the condition of the basis material (see the Bibliography for surface preparation methods). Over the significant surface, the electroplated article shall be free from clearly visible blisters, pits, roughness, cracks and 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 and extent shall be specified by the purchaser [see 4.1 d)].

In the case of selectively plated articles, the degree and extent of discoloration at the boundary between the areas that have been electroplated and those that are not electroplated shall be specified on the product drawing.

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

The types of article which are selectively electroplated with gold are commonly also selectively electroplated with other metals, such as a nickel undercoat or solderable tin alloy coating. Agreements on visual standards will thus extend to all such areas and boundaries. It is therefore essential that such parameters be specified on the product drawing.

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 within the reference area or at a site specified on the component drawing. The minimum thickness of the gold or gold alloy coating shall be that specified by the purchaser.

One or more of the methods given in Annex B shall be used to measure the thickness of the gold or gold alloy coating.

A profile of the minimum-thickness distribution may alternatively be specified on suitably prepared drawings.

Thicknesses of gold and gold alloy coatings commonly specified for electrical, electronic or engineering applications are given in Table 1.

Table 1 — Examples of commonly specified thicknesses of gold and gold alloy coatings for various applications

Application	Minimum thickness µm
Solderability retention, low-reliability electrical contacts	0,1
Medium-reliability electrical connector and switch contacts (electroplated acid gold alloy)	0,25
Semiconductor bonding (pure gold)	0,5
High-reliability consumer electrical contacts	0,75
High-frequency devices and waveguides (pure gold)	1,0
High-reliability electrical contacts for safety-critical applications	2,5 or 5,0

The thickness values given are approximate only. The thickness which a designer specifies for an application will be the minimum necessary for satisfactory performance in porosity and/or wear tests.

6.4 Porosity

When specified by the purchaser, the parts shall be subjected to one or more of the environmental and porosity tests given in ISO 4524-3, ISO 10308, ISO 12687 or ISO 14647. The method to be used and the acceptable number and location of pores shall be specified by the purchaser.

The electrographic tests given in ISO 4524-3:1985, Clauses 2 to 5, can be used for articles with flat surfaces. The tests given in ISO 12687 and ISO 14647 involving gas exposure and the electrographic test given in ISO 4524-3:1985, Clause 6, can be used for articles with curved surfaces.

6.5 Accelerated-corrosion testing

Where the corrosion resistance of the coated articles is important and accelerated-corrosion testing is specified, one of the tests given in ISO 4524-2 shall be used. The acceptable corrosion rating after testing shall be specified by the purchaser in accordance with ISO 10289.

The duration and results of accelerated-corrosion tests may 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.

Accelerated-corrosion tests are frequently used as part of the qualification testing of electrical subassemblies containing gold or gold alloy electroplated components. Such operational tests typically involve multiple operations of the assembly followed by exposure to a corrosive atmosphere. Qualification depends upon the satisfactory results of electrical tests, such as contact resistance measurements, conducted before and after the operational tests. Corrosion testing on its own is not to be used as a guide to performance in qualification tests.

6.6 Composition

When required, the minimum gold content of the coating shall be specified by the purchaser in the designation. Where alloy coatings are required, the gold and alloyed-metal contents shall be specified. The nature and amount of non-metallic inclusions may be specified, especially in the case of electrical and electronic applications. The properties of the coating may be significantly affected by the presence of non-metallic inclusions.

If specified by the purchaser, the gold content of the coating shall be determined by the method given in Annex D.

Pure-gold coatings or gold alloy coatings electroplated from sulfite-containing solutions produce deposits with a tendency to cold welding. They are not recommended for use as finishes for connector or switch contacts.

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 coil-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 (strength 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 inducing hydrogen embrittlement during cleaning.

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.

The effectiveness of the hydrogen embrittlement relief heat treatment may be determined by test methods specified by the purchaser or by test methods described in International Standards (see the Bibliography).

The properties of some gold alloy coatings may be changed by heat treatment procedures. It is, however, unlikely that high-strength steels of strength equal to or greater than 1 000 MPa will be used for any such application.

6.9 Adhesion

Coatings shall pass one or more of the adhesion tests (see ISO 2819) given in Annex C, as specified by the purchaser. The individual layers shall not separate when tested by the methods given in this International Standard.

Aluminium alloys may be 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.

The preparation of sectioned metallographic specimens for the microscopical determination of thickness may give an indication of poor adhesion because grinding and polishing of the specimen may cause separation of the coating from the substrate which can be observed in the microscope. Misleading results may also be obtained if grinding or polishing are not properly carried out during metallographic preparation.

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 e)].

6.11 Microhardness

If the microhardness of the coating is specified, it shall be measured by one of the methods given in ISO 4516. Typical microhardness ranges for gold and gold alloy coatings are given in Table 2.

Table 2 — Microhardness ranges for gold and gold alloy coatings for various uses

Knoop microhardness range HK25	Type of coating and typical applications
90 maximum	Pure gold for semiconductor bonding and microwave applications
91 to 130	Alloyed acid gold coatings for sliding contacts and connectors
91 to 200	Alloyed-gold coatings from neutral or alkaline plating solutions for switches or heavy-duty connectors
> 200	Other applications requiring wear resistance

The microhardness of electroplated gold and gold alloy coatings is dependent on the solution formulation and how the process is controlled and operated. In general, variation in microhardness is achieved by the addition of alloying elements and organic additives.

6.12 Solderability

If specified, the solderability of the gold or gold alloy coating shall be measured by the method described in IEC 60068-2-20, or by an alternative test method specified by the purchaser. The type of test and any artificial-ageing treatment carried out before testing shall be specified [see 4.2 g)].

The formation of brittle joints and the loss of solderability of thin, porous gold coatings during storage is possible. Soft-soldered joints on gold coatings may contain intermetallic compounds that are hard and brittle and may lower shear, fatigue and impact resistance. The risk of developing brittleness becomes greater with increase in soldering temperature or gold thickness and may be greater with some alloy coatings. If the thickness exceeds 1,5 μm, special soldering techniques will need to be used. When thin coatings are specified, the loss of solderability that may occur in storage may be avoided by specifying a suitable undercoat.

6.13 Wear resistance

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

6.14 Ductility

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

6.15 Undercoats

Undercoats applied prior to electroplating with gold or gold alloy coatings may be specified to improve corrosion resistance, adhesion and solderability; to prevent interdiffusion between the basis metal and the coating; to prevent contamination of electroplating solutions; or to reduce surface roughness or porosity. Typical undercoats include copper, nickel, palladium, palladium-nickel, palladium-cobalt and combinations of these undercoats. When undercoats are specified, they shall meet the requirements given in Annex A.

6.16 Freedom from contamination

If specified by the purchaser, the articles shall be subjected to the residual-salts test specified in ISO 4524-6. An increase in conductivity of not more than 150 μS/m, as measured by that test, shall be acceptable.

Articles electroplated with gold or gold alloy coatings shall be thoroughly rinsed after each electroplating process. It is preferable to use de-ionized water for each rinse and to dry the plated articles thoroughly after the final rinse.

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 this International Standard. The lot shall be considered conforming or non-conforming for each requirement, in accordance with 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 requirement for an electrodeposited undercoat applied prior to gold or gold alloy electroplating is given in Table A.1 for different basis materials. The minimum thickness of the undercoat shall be that given in Table A.1. The thickness of any undercoat, when specified, shall be determined by the microscopical method (ISO 1463) or the coulometric method (ISO 2177) or, when local thickness determination is required on selectively plated articles, by the X-ray spectrometric method (ISO 3497).

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

Basis material	Application	Undercoats	Minimum thickness µm
Copper Copper alloys (notably those containing lead)	Interdiffusion prevention	Nickel Copper or nickel	2 Specified by the purchaser
Ferrous materials (other than austenitic stainless steel)	Corrosion prevention	Nickel [a]	10
		Copper plus nickel [a]	10 of Cu plus 5 of Ni (i.e. Cu10/Ni5)
Austenitic stainless steel [c]	Adhesion	An acid nickel strike (Woods bath) will normally be required [b].	Thin, not normally specified
Zinc and its alloys	Adhesion, corrosion protection	Copper plus nickel [a]	8 of Cu plus 10 of Ni (i.e. Cu8/Ni10)
Aluminium and its alloys	Adhesion, corrosion protection	Nickel [a,d]	20
Other basis materials with soldered or brazed joints	Adhesion, interdiffusion prevention	Nickel or copper may be required.	Specified by the purchaser
Non-metallic basis materials	Adhesion, strength, conductivity	Copper and/or nickel will be required.	Specified by the purchaser

[a]   Ductile, low-stress nickel undercoats may be essential.

[b]   If gold-electroplated austenitic stainless steel is to be used in a chloride environment, a substantial nickel undercoat will be necessary and its thickness shall be specified.

[c]   An initial copper coating may be used under the nickel coating, but the thickness of the nickel shall not be reduced.

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

A.2 Chemical symbols for common undercoats

For common undercoats, the chemical symbols in Table A.2 shall be used in the coating designation when required.

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

Chemical symbol	Undercoat
Ni	Nickel
Cu	Copper
Cu/Ni	Nickel applied over copper

Annex B
(normative)

Methods of measuring the thickness of gold and gold alloy coatings

B.1 Uncertainty of thickness measurements

The methods given below have adequate precision, that is the uncertainty in the measurement is less than 10 % when the method is 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 Clauses B.3, B.4 and B.5. The most reliable method (see ISO 3882) shall be selected taking into consideration the expected coating thickness, the shape and size of the components, the nature of the coating material and the nature of 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 gold or gold 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 (by mass) and 40 % silver (by mass) would have the following calculated density, in g/cm^3:

$$\rho = 100/[60/19,3 + 40/10,5] = 14,5$$

where

ρ is the calculated density of the alloy coating, in g/cm^3;

19,3   is the density of pure gold, in g/cm^3;

10,5   is the density of pure silver, in g/cm^3.

A value of the density is required for the beta backscatter, X-ray spectrometric, coulometric, gravimetric and chemical analytical methods described in Clauses 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 gold electroplating solutions may be able to estimate the density of the gold 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 gold coatings electrodeposited from different solutions are compared to values calculated from deposit purity in Table B.1. Table B.1 illustrates the fact that calculated density values may introduce large errors.

These true density values are for illustrative purposes only and are not to be used as density factors when converting from mass per unit area to thickness.

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

Type of gold electroplating solution	Deposit purity % (by mass)	True density g/cm^3	Calculated density g/cm^3
Alkaline cyanide (matt)	99,9	18,9	19,3
Alkaline non-cyanide (bright)	99,9	19,2	19,3
"Alloy" (Ag) alkaline cyanide (bright)	99,0	16,7	19,1
"Alloy" (Cd) alkaline non-cyanide (bright)	98,6	18,9	19,0
Acid hard gold (Co) (bright)	99,5	17,8	19,2

B.3 Non-destructive methods

B.3.1 Beta backscatter (see ISO 3543)

This method has a measurement uncertainty of less than 10 % for gold thicknesses corresponding to a 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 gold or gold alloy coating is required for precise measurements.

B.3.2 X-ray spectrometric (see 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.4 Semi-destructive methods

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

B.4.1 Coulometric (see ISO 2177)

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

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

B.4.2 Profilometric (see ISO 4518)

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

B.4.3 Interferometric (see ISO 3868)

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

B.5 Destructive methods

B.5.1 Microscopical (see ISO 1463)

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

B.5.2 Gravimetric

B.5.2.1 Principle

The substrate is dissolved by chemical or electrochemical means which do not attack the gold or gold alloy coating and the mass of the coating determined. The average thickness of the coating is then calculated from its area, mass and density.

B.5.2.2 Test specimen

Carefully cut or punch a test specimen from the article in such a way that the area and the mass of the gold coating can be determined with a precision of 98 % or better. It may be necessary to square and sand the edges of the test specimen to remove gold smeared over the edges by the cutting or punching operation.

B.5.2.3 Procedure

Measure the area of the coating. Before stripping, remove as much as possible of the substrate by mechanical means to minimize potential attack of the coating. Dissolve the substrate material without attacking the coating. For many substrates, a 25 % solution by volume of nitric acid ($\rho = 1,42$ g/ml) may be used at approximately 20 °C, but this acid may dissolve some elements that could be present in gold alloy coatings.

Take the gold out of the solution, rinse it and dry at 100 °C for about 30 min. Weigh the gold coating. If the gold breaks into small pieces in the solution, it may be necessary to separate the gold from the solution by filtration, using standard analytical techniques, before rinsing, drying and weighing it.

B.5.2.4 Thickness calculation

The average coating thickness is calculated from the following equation:

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

where

    d is the average coating thickness, in µm;

    m is the mass, in mg, of the coating;

    A is the surface area, in cm^2, of the coating;

    ρ is the density, in g/cm^3, of the coating (unless the true value is known, a value of 19,3 g/cm^3 shall be used in the calculation).

B.5.3 Chemical analysis

B.5.3.1 Principle

The basis material is removed from a small, known area of the test specimen (taken from a suitable location on the finished article). The gold is then dissolved in aqua regia and the mass of gold in the test solution determined by spectrophotometric or atomic spectrometric absorption. The average thickness of the gold coating on the test specimen is calculated from the area of the coating and its mass, density and purity.

If the gold content of the coating is to be determined, the washed and dried coating is weighed before dissolution in aqua regia.

B.5.3.2 Reagents

Use only analytical-grade reagents and distilled water or water of equivalent purity.

B.5.3.2.1   Nitric acid, 25 % solution with a density of about 1,2 g/ml.

B.5.3.2.2   Aqua regia, prepared by adding 25 ml of concentrated nitric acid ($\rho = 1,42$ g/ml) to 75 ml of concentrated hydrochloric acid ($\rho = 1,18$ g/ml).

B.5.3.2.3   Standard gold solution (0,05 g/l of Au), prepared by dissolving 0,050 g of gold, purity 99,99 % (by mass), in 20 ml of aqua regia (B.5.3.2.2) in a one-mark volumetric flask of capacity 1 000 ml and diluting with water to the mark. One millilitre of this standard solution contains 50 µg of gold.

B.5.3.3 Apparatus

Thoroughly clean all glassware, including the spectrophotmetric cells, with aqua regia (B.5.3.2.2) and rinse with water before use. It is preferable to reserve glassware solely for these analyses.

B.5.3.4 Test specimen

Carefully cut or punch a test specimen from the article in such a way that its area and the mass of the gold coating can be determined with a measurement uncertainty of less than 2 %. It may be necessary to square and sand the edges to remove any gold smeared over the edges by the cutting or punching operation. The precision of the thickness measurement depends mainly on the precision with which the area of the test specimen can be measured. For thin articles of narrow cross-section, precisely sized specimens may be taken by using a punch and die set to give discs of known diameter. For gold-electroplated pads on printed wiring boards, the copper foil can usually be detached mechanically. If necessary, the pads can be detached from the laminate by boiling in sodium hydroxide solution containing about 200 g/l of NaOH.

B.5.3.5 Preparation of test and standard solutions

B.5.3.5.1 Test solution

The quantities of reagents and the dilutions specified in this preparation procedure are based on an area of 0,1 cm^2, the smallest test specimen that should be taken. If larger areas are taken, adjust the dilutions and the volumes of the aliquot portions of the test solution accordingly.

Before stripping, remove as much of the basis metal as possible from the test specimen (B.5.3.4) by mechanical means to minimize potential attack of the coating. Separate the coating from the rest of the basis metal by dissolving the basis metal in nitric acid solution (B.5.3.2.1). Wash and dry the stripped coating, and dissolve it in 3 ml of hot aqua regia (B.5.3.2.2) in a 50 ml beaker.

B.5.3.5.2 Standard solutions

Into a series of six 50 ml squat beakers, place the volumes of standard gold solution (B.5.3.2.3) shown in Table B.2.

Determine the gold content of the test solution by the spectrophotometric (B.5.3.6) or the atomic absorption (B.5.3.7) method.

Table B.2 — Standard gold solutions

Volume of standard gold solution (B.5.3.2.3) ml	Corresponding mass of gold mg	Concentration of gold in final solution mg/l
0 [a]	0 [a]	0 [a]
1,0	0,05	0,005
2,0	0,10	0,010
4,0	0,20	0,020
6,0	0,30	0,030
8,0	0,40	0,040
[a] Compensation solution.		

B.5.3.6 Spectrophotometric method

B.5.3.6.1 Principle

Potassium chloride is added to the test solution to give stable gold potassium chloride. The solution is evaporated to dryness followed by spectrophotometric determination of the gold content.

Basis metals that have soluble colourless chlorides, and copper, nickel cobalt and iron that have coloured chlorides, do not interfere. In the case of gold coatings that contain silver or other metals that form insoluble chlorides, it may be necessary to filter the solution before measuring its absorbance.

B.5.3.6.2 Reagents

B.5.3.6.2.1 Potassium chloride solution, 10 g/l.

B.5.3.6.2.2 Hydrochloric acid solution, prepared by diluting 200 ml of concentrated HCl ($\rho = 1,18$ g/ml) to 1 000 ml.

B.5.3.6.3 Apparatus

Ordinary laboratory apparatus plus the following:

B.5.3.6.3.1 Spectrophotometer, fitted with silica cells of 10 mm, and 40 mm or 50 mm, optical path lengths.

B.5.3.6.3.2 Beakers, squat form, capacity 50 ml. At least six are required.

B.5.3.6.3.3 Microfilter funnel, fitted with a sintered glass plate or a suitable filter stick.

B.5.3.6.3.4 Electric oven, capable of maintaining a temperature of 110 °C $\pm$ 2 °C.

B.5.3.6.4 Preparation of calibration graph

B.5.3.6.4.1 Preparation of calibration solutions

Treat the contents of each beaker of dilute standard gold solution (see B.5.3.5.2) as follows: add 1 ml of potassium chloride solution, evaporate carefully to incipient dryness on a hotplate or water bath, dry in the oven at 110 °C ± 2 °C and allow to cool. Re-dissolve the residue in hydrochloric acid in a one-mark 10 ml volumetric flask and dilute to the mark with the same acid.

B.5.3.6.4.2 Spectrophotometric measurements

Carry out the spectrophotometric measurements of the calibration solutions prepared in B.5.3.6.4.1 at a wavelength of 312 nm using 10 mm cells, after having adjusted the instrument to zero absorbance against water.

B.5.3.6.4.3 Plotting the calibration curve

Deduct the absorbance of the compensation solution (see Table B.2) from those of the other calibration solutions. Plot a graph of the concentrations, in milligrams of gold per millilitre, as abscissa versus the corresponding net values of the absorbance as ordinates. A straight line should be obtained.

B.5.3.6.5 Determination

Carry out the determination in duplicate, starting from two different test specimens (see B.5.3.4). Add 1 ml of potassium chloride solution to the test solution (see B.5.3.5.1) and evaporate carefully to incipient dryness on a hotplate or water bath. Dry in the electric oven at 110 °C ± 2 °C and allow to cool. Re-dissolve the residue in hydrochloric acid in a one-mark volumetric flask of capacity given in Table B.3, and then dilute to the mark with the same acid.

Measure the absorbance of the solution in the spectrophotometer at a wavelength of 312 nm using the appropriate cell indicated in Table B.3, after having adjusted the instrument to zero absorbance against the blank solution (see B.5.3.6.6). By means of the calibration curve, determine the mass of gold corresponding to the spectrophotometric measurement, taking into account the blank test (see B.5.3.6.6).

Table B.3 — Volumes of dilution and cell sizes for various thicknesses of coating

Coating thickness, d μm	Capacity of one-mark volumetric flask ml	Cell size mm
$5{,}0 < d < 10{,}0$	20	10
$1{,}25 < d < 5{,}0$	10	10
$0{,}1 < d < 1{,}25$	10	40 or 50

B.5.3.6.6 Blank test

Carry out a blank test in parallel with the determination by the same procedure and using the same quantities of all the reagents but omitting the test specimen.

B.5.3.7 Atomic absorption spectrometric method

B.5.3.7.1 Principle

Lanthanum chloride is added to the test solution to suppress interferences, followed by measurement of the atomic absorption.

B.5.3.7.2   Reagent

B.5.3.7.2.1   Lanthanum chloride solution, 100 g/l, prepared by dissolving approximately 58,6 g of lanthanum oxide in 250 ml of concentrated hydrochloric acid ($\rho \approx 1,18$ g/ml) and diluting to 500 ml with water.

B.5.3.7.3   Apparatus

Ordinary laboratory apparatus plus an atomic absorption spectrometer.

B.5.3.7.4   Preparation of calibration graph

B.5.3.7.4.1   Preparation of calibration solutions

Treat the contents of each beaker of dilute gold standard solution (see B.5.3.5.2) as follows: transfer to a series of six 100 ml one-mark volumetric flasks, then add 4 ml of lanthanum chloride solution and dilute to the mark with water.

B.5.3.7.4.2   Spectrometric measurements

Aspirate the calibration solutions directly into the flame of the atomic absorption spectrometer, using the conditions for gold determinations specified by the instrument manufacturer, and record the absorbance readings.

B.5.3.7.4.3   Plotting the calibration graph

Deduct the absorbance of the compensation solution from those of the other calibration solutions. Plot a graph of the concentrations, in milligrams of gold per millilitre, as abscissa versus the corresponding net values of absorbance as ordinates.

B.5.3.7.5   Determination

Transfer the test solution (see B.5.3.5.1) to a one-mark volumetric flask of capacity given in Table B.4, add the volume of lanthanum chloride solution (B.5.3.7.2.1) indicated and dilute to the mark with water. Measure the absorbance of the resultant solution as described in B.5.3.7.4.2. By means of the calibration graph, determine the mass of gold corresponding to the spectrometric measurement, taking into account the blank test (see B.5.3.7.6).

Table B.4 — Volumes of dilution and of lanthanum chloride solution for various masses of gold

Mass of gold, m_{Au}, in test specimen mg	Capacity of one-mark volumetric flask ml	Volume of lanthanum chloride solution ml
$m_{Au} < 0,2$	10	0,4
$0,2 < m_{Au} < 2$	100	4
$2 < m_{Au} < 20$	1 000	40

B.5.3.7.6   Blank test

Carry out a blank test in parallel with the determination by the same procedure and using the same quantities of all reagents but omitting the test specimen.

B.5.3.8   Thickness calculation

The average coating thickness is calculated from the following equation:

$$d = (10m_1/A\rho)(100/w_{\text{Au}})$$

where

d is the average coating thickness, in µm;

m_1 is the mass, in mg, of gold in the test solution, calculated from the calibration curve (see B.5.3.6.4 or B.5.3.7.4) and the total volume of the final test solution (see Tables B.2 and B.3), taking into account the blank test (see B.5.3.6.6 or B.5.3.7.6);

A is the surface area, in cm^2, of the test specimen (see B.5.3.4);

ρ is the density, in g/cm^3, of the coating;

w_{Au} is the gold content, expressed as a percentage by mass of the coating (see Annex D).

B.5.3.9   Test report

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.