International Standard 4521

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Metallic coatings — Electrodeposited silver and silver alloy coatings for engineering purposes

Revêtements métalliques — Dépôts électrolytiques d'argent et d'alliages d'argent pour applications industrielles

First edition — 1985-02-15

UDC 669.228.7 **Ref. No. ISO 4521-1985 (E)**

Descriptors : coatings, metal coatings, electrodeposited coatings, silver coatings, specifications, designation, tests, adhesion tests, dimensional measurements, thickness.

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 4521 was prepared by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings*.

Metallic coatings — Electrodeposited silver and silver alloy coatings for engineering purposes

0 Introduction

This International Standard specifies a range of electrodeposited silver and silver alloy coatings for electrical, electronic and other engineering purposes. Within this International Standard, engineering purposes are defined as those for which the coating has to fulfil primarily non-decorative functional tasks, while decorative purposes are defined as those for which the appearance of the finished article is of prime importance. However, protection against corrosion may be a requirement in both cases.

Electrodeposited silver and silver alloy coatings are used in many applications and are chosen for their extremely good electrical conductivity. However, in many conditions of service, sulfide films are liable to form on the coatings, consequently increasing the contact resistance of the silver electroplated mating surface and making them unsuitable for use in low voltage electronic circuits. Nevertheless, because the silver sulfide films are not completely insulating, they are not particularly detrimental to other electrical contacts where higher voltages and/or higher contact pressures are used.

Packaging materials, such as paper and cardboard, frequently contain small amounts of sulfur compounds which can cause excessive tarnishing of silver and silver alloy electrodeposited articles. Cognizance should be taken of this when silver and silver alloy electrodeposited articles are packed, stored and transported, and the packaging materials should be free from significant contamination by sulfur compounds. Anti-tarnish treatments can be applied to the silver and silver alloy coatings to prevent or delay tarnishing (see clause 12), but these treatments increase contact resistance to a greater or lesser extent and so may only prove useful in certain cases.

Attention is drawn to the possibility that

a) ionic migration of silver will occur in electronic circuits, particularly if the insulating material is damp;

b) galling (cold welding) will occur when two similar silver surfaces (for example two "bright" or two "dull" coatings) are used in sliding contact.

Attention is also drawn to the expression in certain contexts of silver contents in terms of parts per thousand (by mass), also known as "thousandths" (*millièmes*) or "fineness". In this International Standard, percentages by mass to one decimal place are used.

1 Scope and field of application

This International Standard specifies requirements for electrodeposited coatings of silver and its alloys for electrical, electronic and other engineering applications on metallic and non-metallic materials. Such coatings may or may not be subject to subsequent machining.

It does not apply to coatings on screw threads [see ISO 4042, *Threaded components — Electroplated coatings components* (at present at the stage of draft)] or to coatings on sheet or strip in the unfabricated form.

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.* [1)]

ISO 4516, *Metallic and related coatings — Vickers and Knoop microhardness tests.*

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

ISO 4522, *Metallic coatings — Test methods for electrodeposited silver and silver alloy coatings —*

Part 1 : Determination of coating thickness.

Part 2 : Adhesion tests.

Part 3 : Residual salts test. [2)]

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

1) At present at the stage of draft. (Revision of ISO 2177-1972.)

2) At present at the stage of draft.

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

3 Definitions

For the purpose of this International Standard, the definitions of ISO 2064, especially the following, apply.

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.

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 (ISO 4521);

b) the classification number of the coating required (see clause 8 and 11.2);

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

d) the surface appearance (see 11.1);

e) the method(s) of adhesion testing to be employed (see 11.5);

f) the sampling procedure to be adopted (see clause 7).

4.2 Additional information

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

a) the minimum silver content of the coating [see 8 c)] and details of any intentional alloying elements (see 11.4);

b) the nature of the basis material, its surface condition and roughness (see clause 5);

c) any requirements for stress relief before electroplating and/or hydrogen embrittlement relief after electroplating (see clause 9);

d) any special requirements, including the thickness, for undercoats (see clauses 6 and 10);

e) any requirements for corrosion resistance and/or porosity testing and the methods of test to be used (see 11.3);

f) the electrical properties of the coating and the methods of test to be used (see 11.6);

g) the microhardness of the coating and the test method from ISO 4516 to be used (see 11.7);

h) any requirements for anti-tarnish treatment, the type of treatment and methods of test to be applied;

j) requirements for solderability and the method of test to be used (see 11.8);

k) any requirements for freedom from contamination of the finished product (see 11.10).

5 Basis material

This International Standard specifies no requirements for the condition, finish or surface roughness of the basis material prior to electroplating. However, it should be recognized that the surface roughness of the coating will be dependent on the initial surface roughness of the basis material and this shall not, therefore, be a cause for rejection of the silver or silver alloy coating.

6 Pretreatment

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

6.2 To avoid poor adhesion, it is usual to use a silver strike to prevent the chemical deposition of silver and silver alloys on to the basis material. For electroplating certain alloys, a gold strike may be preferable.

7 Sampling

A random sample of the size required by 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 and the lot shall be classified as conforming or not conforming to each requirement according to the criteria of the sampling plans in ISO 4519.

8 Classification number

The classification number comprises :

a) the chemical symbol for the basis metal or for the principal metal, if an alloy, or in the case of non-metallic materials the letters NM followed by an oblique stroke;

b) if appropriate, the chemical symbol(s) for the undercoat metal(s), as shown by the examples given in table 1, followed by an oblique stroke;

c) the chemical symbol for silver, Ag, followed if required by a number in parentheses representing the minimum silver content, expressed as a percentage by mass, of the silver coating to one decimal place;

d) a number indicating the minimum thickness, in micrometres, of the silver or silver alloy coating on the significant surface.

Table 1 — Symbols

Symbol	Significance
Ni	Nickel
Cu	Copper
Cu/Ni	Nickel on copper
Sn-Ni	Tin-nickel alloy

Examples of complete classification numbers are

— a silver coating, having a thickness of 20 µm (minimum), on brass, has the classification number Cu/Ag 20

— a 99,9 % silver coating, having a thickness of 10 µm (minimum), on steel, using a nickel undercoat, has the classification number Fe/Ni/Ag(99,9) 10.

9 Heat treatment of steels

9.1 General

Heat treatment as specified in 9.3 and 9.4 shall be performed on certain basis metals to reduce the risk of damage by hydrogen embrittlement. In all cases, the duration of heat treatment shall commence from the time at which the whole of each part attains the specified temperature.

Parts made from steels with maximum specified tensile strengths greater than 1 050 MPa[1] (corresponding hardness values approximately 34 HRC, 340 HV or 325 HB) and surface-hardened parts will require heat treatment. Preparation involving cathodic treatments in alkaline or acid solutions shall be avoided. Additionally, the selection of electroplating solutions with high cathodic efficiencies is recommended for steel components with tensile strengths greater than 1 450 MPa (corresponding hardness values approximately 45 HRC, 440 HV or 415 HB).

9.2 Categorization of steels

9.2.1 With the exception of surface-hardened parts (see 9.3.2 and 9.4.2), the heat-treatment conditions shall be selected on the basis of the specified maximum tensile strength. Steels shall be categorized according to specified maximum tensile strength according to table 2. If the steel specification is only in terms of minimum tensile strength, the corresponding maximum tensile strength shall be determined from table 2.

Table 2 — Categories of steels and maximum tensile strengths corresponding to specified minimum tensile strengths

Minimum specified tensile strength, $R_{m\,min}$ (MPa)	Corresponding maximum tensile strength, $R_{m\,max}$ (MPa)
$R_{m\,min} \leqslant 1\,000$	$R_{m\,max} \leqslant 1\,050$
$1\,000 < R_{m\,min} \leqslant 1\,400$	$1\,050 < R_{m\,max} \leqslant 1\,450$
$1\,400 < R_{m\,min} \leqslant 1\,750$	$1\,450 < R_{m\,max} \leqslant 1\,800$
$1\,750 < R_{m\,min}$	$1\,800 < R_{m\,max}$

9.2.2 If neither the maximum nor the minimum tensile strength is specified for the steel, Vickers hardness values of 340, 440 and 560 HV shall be regarded as equivalent to maximum tensile strengths 1 050, 1 450 and 1 800 MPa, respectively, and these strengths shall be used to select the heat-treatment conditions.

9.3 Stress relief before electroplating

9.3.1 The conditions given in table 3 are recommended if the purchaser requires the parts to be stress relieved before electroplating, although different conditions, namely suitable combinations of shorter periods at appropriate higher temperatures, may be used if they have been shown to be effective. The heat treatment shall be carried out before the commencement of any preparation or cleaning treatment using aqueous solutions.

Table 3 — Heat-treatment conditions for stress relief before electroplating (excluding surface-hardened parts)

Maximum specified tensile strength, $R_{m\,max}$	Temperature	Time
MPa	°C	h
$R_{m\,max} \leqslant 1\,050$	None required	—
$1\,050 < R_{m\,max} \leqslant 1\,450$	190 to 220	1
$1\,450 < R_{m\,max} \leqslant 1\,800$	190 to 220	18
$1\,800 < R_{m\,max}$	190 to 220	24

9.3.2 Surface-hardened parts shall be heat treated at 130 to 150 °C for not less than 5 h or for shorter periods at higher temperatures if the resulting loss of surface hardness of the substrate is acceptable.

9.3.3 If stress relief is given after shot peening or other cold working processes, the temperature shall not exceed 220 °C.

9.4 Hydrogen embrittlement relief after electroplating

9.4.1 The treatment shall be carried out as soon as possible, and in any case not later than 4 h after electroplating, according to the conditions specified in table 4.

Table 4 — Heat-treatment conditions for hydrogen embrittlement relief after electroplating (excluding surface-hardened parts)

Maximum specified tensile strength, $R_{m\,max}$	Temperature	Time
MPa	°C	h
$R_{m\,max} \leqslant 1\,050$	None required	—
$1\,050 < R_{m\,max} \leqslant 1\,450$	190 to 220	8
$1\,450 < R_{m\,max} \leqslant 1\,800$	190 to 220	18
$1\,800 < R_{m\,max}$	190 to 220	24

1) 1 MPa = 1 N/mm²

9.4.2 Surface-hardened parts shall be heat treated at a temperature between 190 and 220 °C for not less than 2 h.

9.4.3 Other temperatures and durations may be specified and used if they have been shown to be effective for the particular part, and are acceptable to the purchaser. However, parts shall not be heat treated above their tempering temperature.

NOTE — Attention is drawn to the fact that the heat treatments specified can cause interdiffusion of the silver coating and the substrate. Undercoats of nickel reduce this effect. However, see also note 3 to clause 10.

10 Requirements for undercoats

The thickness requirements for undercoats given in table 5 are minimum values. For arduous conditions of service, greater thicknesses may be required and shall be specified by the purchaser [see 4.2 d)].

NOTES

1 See 6.2 for guidance on avoidance of poor adhesion.

2 An undercoat of low stress may be essential.

3 For certain applications, undercoats of copper and/or nickel may be undesirable. In such cases, the undercoat may be replaced by at least the same thickness of silver or silver alloy.

Table 5 — Requirements for undercoats

Basis material	Undercoat(s)	Minimum thickness of undercoat(s) (μm)
Copper	None	—
Copper alloys, notably free-cutting brass containing lead	Cu (copper) or Ni (nickel)1) may be required	To be agreed
Ferrous materials (other than austenitic stainless steel)	Ni (nickel)1)	10
	Cu/Ni (copper + nickel)1)	10 (Cu) + 5 (Ni)
Austenitic stainless steel	An acid nickel strike [Wood's bath2)] will normally be required	Thin coating, to promote adhesion of the silver coating
Zinc and zinc alloys	Cu/Ni (copper + nickel)1)	8 (Cu) + 10 (Ni)
Aluminium and aluminium alloys	Ni (nickel)1) 3)	20
Other basis materials and basis metal with soldered joints	Nickel1) or copper may be required to meet functional or other requirements of this International Standard	To be agreed

1) Low stress nickel undercoats should normally be used.

2) A Wood's bath consists of a solution of 240 g of nickel chloride hexahydrate ($NiCl_2 \cdot 6H_2O$) and 85 ml of 36 % (*m/m*) hydrochloric acid per litre. The parts are made anodic for not more than 2 min and then cathodic for 6 min, using depolarized nickel electrodes, without anode bags, at room temperature and a current density of 300 A/m^2. If current reversal is not feasible, the anodic treatment can be replaced by immersion in the solution, without current flow, for 15 min.

3) An initial copper coating may be employed under the nickel coating but the thickness of the nickel coating should not be reduced.

If required, the thickness of any undercoat shall be determined by the microscopical method specified in ISO 1463 or the coulometric method specified in ISO 2177.

11 Requirements for silver and silver alloy coatings

If the tests given in 11.2, 11.3, 11.5 and 11.7 are to be carried out, this should be done in the absence of any anti-tarnish treatment referred to in clause 12. The tests specified in 11.4 and 11.6 shall be carried out after the final treatment.

11.1 Appearance

The electroplated article shall be free from extraneous soil and from damage.

Over the significant surface, the electroplated article which is not to be machined subsequently shall be free from electroplating defects such as pits, roughness, cracks, or uncoated areas visible when viewed at a magnification of X 8 (see the note). In addition, blistering or other signs of poor adhesion cannot be tolerated on any surface of the article.

Silver and silver alloy electroplated articles which are to be machined subsequently shall be free from excessive nodulation and treeing at the edges and from imperfections detrimental to subsequent fabrication.

On articles where a contact mark on the significant surface is unavoidable, its position shall be the subject of agreement between the supplier and the purchaser.

If required, a preliminary sample with the required finish shall be supplied or approved by the purchaser.

NOTE — If using illuminated viewers, a X 4 magnification is acceptable.

11.2 Thickness

Silver and silver alloy coatings are classified by thickness and the preferred range for general use is given in table 6. Any other coating thickness, above the minimum of 2 µm and expressed in whole numbers of micrometres, may be called up by specifying the required minimum local thickness at the appropriate place in the classification number.

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

The thickness of the coatings shall be measured over a reference area (see ISO 2064) by an appropriate method selected by the purchaser from those specified in ISO 4522/1 on any part of the significant surface.

11.3 Porosity and corrosion resistance

Where porosity and/or corrosion resistance are important, and if specified by the purchaser, the parts shall be subjected to one or more porosity and/or corrosion tests which relate to the environment in which the silver and silver alloy electroplated parts are to be exposed.

Table 6 — Typical thickness of silver and silver alloy coatings (not subsequently machined) for engineering applications

Minimum thickness
µm
2
5
10
20
40

11.4 Composition

The minimum silver content of the coating shall if required be specified by the purchaser as a percentage by mass to one decimal place and included at the appropriate place in the classification number [see 4.2 a) and clause 8].

NOTE — Metallic or organic brighteners used for grain refinement may be present in the electrodeposited coating, provided that they do not interfere with the intended function of the coating and are acceptable to the purchaser.

If required, the purchaser shall specify the method to be used for the determination of the silver content of the coating.

11.5 Adhesion

Coatings shall be capable of passing one or more of the tests given in 11.5.1 to 11.5.4 and 11.5.6, as specified by the purchaser. Coatings greater than 125 µm in thickness shall be capable of passing the shear test given in 11.5.5.

NOTE — The preparation of cross-sections for metallographic thickness measurement often provides an indication of poor adhesion. The action of grinding and polishing creates a shear force which may cause the separation of the deposit from the substrate, and this separation can be observed in the measuring microscope.

11.5.1 Burnishing test

When the specimen is tested by the burnishing test specified in ISO 4522/2, there shall be no sign of blistering of the coating.

11.5.2 Barrel burnishing test

When the specimen is tested by the barrel burnishing test specified in ISO 4522/2, there shall be no sign of blistering or peeling of the coating.

11.5.3 Peel test

When the specimen is tested by the peel test specified in ISO 4522/2, there shall be no sign of detachment of the coating — failure shall occur in the solder layer only.

11.5.4 Bend test

When the specimen is tested by the bend test specified in ISO 4522/2, it shall withstand three bends without detachment of the coating.

Failure of the substrate due to microcracking or macrocracking shall not be cause for rejection provided that the coating has not exfoliated.

11.5.5 Shear test

When the specimen is tested by the shear test specified in ISO 4522/2, there shall be no sign of separation from the basis metal at the interface, nor shall there be any sign of blistering, flaking or peeling of the coating.

11.5.6 Shot peening

When the specimen is tested by the shot peening test specified in ISO 4522/2, there shall be no sign of blistering or detachment of the coating.

11.5.7 Thermal shock test

When the specimen is tested by the thermal shock test specified in ISO 4522/2, there shall be no sign of blistering or detachment of the coating.

11.6 Electrical properties

If the electrical properties of the coating are important, these shall be specified by the purchaser together with the method or methods of assessing such properties.

11.7 Hardness

If a value for hardness is specified the hardness of the coating shall be determined by one of the methods specified in ISO 4516.

NOTE — The hardness of silver and silver alloy coatings deposited from many types of bright silver electroplating solutions decreases considerably during the first 24 h after electrodeposition. In such cases, the hardness test should not be carried out until this time has elapsed or, alternatively, until an accelerated ageing treatment, such as maintaining the specimen at 100 °C for 1 h, has been carried out.

11.8 Solderability

If specified, silver coatings shall be subjected to a solderability test. The form of test and any artificial ageing treatment carried out before the test shall be appropriate to the intended service of the electroplated product and details shall be agreed between the parties. A test is described in IEC Publication 68-2-20.

11.9 Wear resistance

If the wear resistance of the coating is important, it shall be specified by the purchaser together with its method of measurement.

11.10 Freedom from contamination

The silver or silver alloy coated parts shall be thoroughly rinsed and dried after electroplating. If specified by the purchaser, the parts shall be subjected to the residual salts test specified in ISO 4522/3.

An increase in conductivity of not more than 150 µS/m shall be considered acceptable.

12 Anti-tarnish treatment

There are a number of treatments available which retard the onset of tarnishing of silver and silver alloys. If such a treatment is required, this shall be specified by the purchaser together with any necessary associated tests. A suitable test is given in ISO 4538 and, if applied, the duration of test shall be specified by the purchaser.

Treated paper strips, intended for packaging with silver electroplated items, that react with oxides of sulfur and sulfide gases, can inhibit tarnish formation during shipment and storage without affecting the surface properties of the silver.

NOTE — Many anti-tarnish treatments increase the surface electrical resistivity of the silver and silver alloy coatings and may also impair their solderability.