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Metallic coatings — Electroplated coatings of nickel plus chromium on plastics materials

*Revêtements métalliques — Dépôts électrolytiques de nickel plus chrome
sur matières plastiques*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 4525 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 3, *Electrodeposited coatings and related finishes*.

This second edition cancels and replaces the first edition (ISO 4525:1985), which has been technically revised.

Annexes A, C, D, E, F and G form a normative part of this International Standard. Annex B is for information only.

Introduction

The traditional method of preparing plastics for electroplating includes electrodeposition of a ductile acid copper layer, before electroplating with nickel plus chromium, to meet thermal cycle requirements. The elimination of copper and its substitution with *ductile* nickel is a new trend aimed at facilitating the reclamation of electroplated plastics at the end of the product-life cycle. Nickel plus chromium metal can be readily separated from plastics and utilized directly in the production of stainless steel, whereas copper plus nickel plus chromium coatings would first require separation and complete removal of copper because of its detrimental effects on the properties of stainless steel. Although the traditional method is still the one most widely applied, the automotive and plumbing industries are now specifying ductile nickel as replacement for copper undercoats in Europe, because of the reclamation benefits. These recent trends have been taken into account in revising this document which permits the specification of decorative, electroplated nickel plus chromium coatings on plastics materials with either copper or nickel undercoats when thermal cycle resistance is a requirement.

New developments in preparing plastics for electroplating, e.g., the use of ionic palladium catalysts, and the elimination of electroless deposition and chromic/sulfuric acid etchants, make it more essential than ever that the instructions provided by the suppliers of proprietary processes for preparing plastics for electroplating be followed. Proper surface preparation is essential to obtain satisfactory performance of electroplated coatings on plastics materials.

No distinction is made between the types of plastics suitable for electroplating and no detailed requirements are laid down concerning the surface condition of the plastics material or the level of moulding stresses. However, where plastics articles are produced by some technique that involves a change of phase, such as moulding, then it is essential that the electroplating operation not take place until at least 24 h have elapsed after production.

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Metallic coatings — Electroplated coatings of nickel plus chromium on plastics materials

1 Scope

This International Standard specifies requirements for decorative, electroplated coatings of nickel plus chromium with and without copper undercoats on plastics materials. It permits the use of either a copper or ductile nickel undercoat to satisfy thermal cycle requirements.

This International Standard is not applicable to such coatings on plastics to be used for engineering purposes.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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, *Surface treatment, metallic and other inorganic coatings — Vocabulary*

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

ISO 2361, *Electrodeposited nickel coatings on magnetic and non-magnetic substrates — Measurement of coating thickness — Magnetic method*

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

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

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

ISO 8401, *Metallic coatings — Review of methods of measurement of ductility*

ISO 9227, *Corrosion tests in artificial atmospheres — Salt spray tests*

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 16348, *Metallic and other inorganic coatings — Definitions and conventions concerning appearance*

ASTM B764-94, *Standard Test Method for Simultaneous Thickness and Electrochemical Potential Determination of Individual Layers in Multilayer Nickel Deposit (STEP Test)*

3 Terms and definitions

For the purposes of this International Standard, the following terms and definitions given in ISO 2064, ISO 2080 and ISO 16348 apply.

4 Information to be supplied to the electroplater

4.1 Essential information

When ordering articles to be electroplated in accordance with this International Standard, the purchaser shall provide the following information in writing, e.g., in the contract or purchase order, or on engineering drawings:

- a) the designation (see clause 6);
- b) the appearance required, e.g. bright, dull or satin; alternatively, a sample showing the required finish shall be supplied or approved by the purchaser and used for comparison purposes in accordance with 7.2;
- c) the significant surfaces to be indicated on drawings of the parts, or by providing samples that are suitably marked;
- d) additional portions of the significant surface where local thickness requirements are to be applied (see 7.4);
- e) the positions on significant surfaces for rack or contact marks where such marks are unavoidable (see 7.2);
- f) whether copper or nickel undercoats shall be applied to meet the thermal cycle requirements (see 7.3, 7.6 and 7.8);
- g) whether corrosion testing is to be done continuously or cyclically (see 7.7);
- h) whether corrosion and thermal cycle tests (see 7.6 and 7.7) shall be done individually on separate specimens or sequentially using the same specimens (see 7.8), and whether the specimens shall be mounted or unmounted in a manner simulating assembly during testing (annex A);
- i) any requirements for STEP testing (see 7.9);
- j) sampling methods and acceptance levels (see clause 8);
- k) the designation of the type of plastic to be electroplated (see 7.1).

4.2 Additional information

The following additional information may be provided by the purchaser, when appropriate.

- a) The limitations on the extent of tolerable surface defects resulting from moulding (see 7.1).
- b) The extent to which defects are to be tolerated on non-significant surfaces (see 7.2).

5 Service condition number

The service condition number shall be used by the purchaser to determine the degree of protection required as related to the severity of the conditions to which a product is to be subjected, in accordance with the following scale:

- | | |
|---|----------------------|
| 5 | Exceptionally severe |
| 4 | Very severe |
| 3 | Severe |
| 2 | Moderate |
| 1 | Mild |

Typical service conditions for which the various service condition numbers are appropriate are defined in annex B.

6 Designation

6.1 General

The designation is a means of specifying the types and thicknesses of coatings appropriate for each service condition (see Table 1) and comprises the following:

- a) the term, "Electroplated coating", the number of this International Standard, ISO 4525, followed by a hyphen;
- b) the letters, PL, indicating a plastics base material followed by a solidus (/);
- c) the chemical symbol, Cu, for the copper undercoat (or the chemical symbol, Ni, when the undercoat is nickel); copper or nickel undercoats shall only be omitted when there are no requirements for thermal cycle resistance, as specified by the purchaser;
- d) a number giving the minimum local thickness (see ISO 2064), in micrometres, of the copper (or nickel) undercoat;
- e) a lower-case letter designating the type of copper or nickel undercoat (see 6.2);
- f) the chemical symbol, Ni, for nickel;
- g) a number indicating the minimum local thickness (see ISO 2064), in micrometres, of the nickel coating;
- h) a letter designating the type of nickel coating (see 6.3);
- i) the chemical symbol, Cr, for chromium;
- j) a letter or letters designating the type and thickness of the chromium deposit (see 6.5).

Table 1 — Coatings on plastics materials

Service condition number	Partial coating designation for copper plus nickel plus chromium coatings	Partial coating designation for nickel plus chromium coatings
5	PL/Cu15a Ni30d Cr mp (or mc)	PL/Ni20dp Ni20d Cr mp (or mc)
4	PL/Cu15a Ni30d Cr r	PL/Ni20dp Ni20d Cr r
	PL/Cu15a Ni25d Cr mp (or mc)	PL/Ni20dp Ni20b Cr mp (or mc)
3	PL/Cu15a Ni25d Cr r	PL/Ni20dp Ni15b Cr r
	PL/Cu5a Ni20d Cr mp (or mc)	
2	PL/Cu15a Ni15b Cr r	PL/Ni20dp Ni10b Cr r
	PL/Cu15a Ni10b Cr mp (or mc)	
1	PL/Cu15a Ni7b Cr r	PL/Ni20dp Ni7b Cr r

6.2 Type of copper or nickel undercoat

The type of copper undercoat shall be designated by the following symbol:

- a for ductile, levelling copper electrodeposited from acid-type solutions.

The type of nickel undercoat shall be designated by the following symbol:

- dp for ductile, columnar nickel electrodeposited from special pre-electroplating solutions.

NOTE The type of nickel required for thermal cycle resistance may be obtained by electroplating from Watts or nickel sulfamate solutions containing no organic additives or brighteners, as well as from special proprietary formulations available from suppliers of electroplating processes. See [3], [4], [5] for additional background information.

6.3 Type of nickel

The type of nickel applied over the copper or nickel undercoat shall be designated by the following symbols:

- b for nickel deposited in the fully bright condition;
- s for dull or semi-bright nickel that shall not have been mechanically polished;
- d for double- or triple-layer nickel coatings, the requirements for which are given in Table 2.

6.4 Double- and triple layer coatings

The requirements for double- and triple-layer coatings are summarized in Table 2.

Table 2 — Requirements for double- and triple-layer nickel coatings

Layer (type of nickel coating)	Specific elongation ^a %	Sulfur content ^b % mass fraction	Thickness ^c as a percentage of total nickel thickness	
			double-layer	triple-layer
Bottom (s)	> 8	< 0,005	≥ 60	50 to 70
Middle (high sulfur layer)	—	> 0,15	—	≤ 10
Top (b)	—	> 0,04 and < 0,15	10 to 40	≥ 30

^a The test method for determination of specific elongation (or ductility) is specified in annex C.

^b The sulfur contents are specified to indicate the type of nickel plating solution that is to be used. No simple method exists for determining the sulfur content of a nickel deposit on a coated article. However, an accurate determination is possible on a specially prepared test specimen (see annex D).

^c It will usually be possible to identify the type and determine the ratios of nickel layers by microscopical examination of a polished and etched section of an article prepared in accordance with ISO 1463, or by means of the STEP.

6.5 Types and thicknesses of chromium

The types and thicknesses of chromium shall be designated by the following symbols placed after the chemical symbol, Cr, as follows:

- r regular (i.e., conventional) chromium having a minimum local thickness of 0,3 µm;
- mc micro-cracked chromium having more than 250 cracks per centimetre in any direction, forming a closed network over the whole of the significant surface when determined by one of the methods specified in annex E and having a thickness of 0,3 µm. With some processes, a substantially greater thickness (about 0,8 µm) of chromium may be required to achieve the necessary crack pattern, in which case the minimum local thickness shall be included in the coating designation as follows: Cr mc (0,8);
- mp micro-porous chromium, containing a minimum of 10 000 pores per square centimetre when determined by the method specified in annex E and having a minimum local thickness of 0,3 µm. The pores shall be invisible to the unaided eye or corrected vision.

NOTE 1 Micro-porous chromium is often achieved by depositing the chromium over a special thin nickel layer that contains inert non-conducting particles, the special nickel layer being applied on top of b or d nickel.

NOTE 2 There may be some loss of lustre after a period of service in the case of mp or mc chromium deposits which may be unacceptable in some applications. This tendency can be reduced by increasing the minimum chromium coating thickness to 0,5 µm in every case where micro-porous or micro-cracked chromium is specified in Table 1.

6.6 Example of a designation

An electroplated coating on a plastics base (PL) comprising 15 μm (minimum) bright acid copper (Cu15a) and 10 μm (minimum) bright nickel (Ni10b) plus 0,3 μm (minimum) microporous or microcracked chromium [Cr mp (or mc)] shall be designated as follows:

Electroplated coating ISO 4525 - PL/Cu15a Ni10b Cr mp (or mc)

An electroplated coating on a plastics base (PL) comprising 20 μm (minimum) ductile nickel (Ni20dp) and 20 μm (minimum) double layer nickel (Ni20d) plus 0,3 μm (minimum) microporous chromium (Cr mp) shall be designated as follows:

Electroplated coating ISO 4525 - PL/Ni20dp Ni20d Cr mp

For ordering purposes, the detailed product specification should not only comprise the designation, but also include clear written statements of other requirements that are essential for the serviceability of the particular product (see clause 4).

7 Requirements

7.1 Substrate

The plastics materials shall be plateable and formulated to enable the metallic coatings to conform to this International Standard when the coatings are correctly applied [see 4.1 k)].

Defects in the surface of the moulded plastics such as cold shots, ejection marks, flash, gate marks, parting lines, splay and others, may adversely affect the appearance and performance of coatings on plastics materials. Accordingly, the electroplater's responsibility for defects in the coating resulting from the plastic-moulding operation is waived, unless the electroplater is also the moulder. Alternatively, the specifications covering the items to be electroplated should contain appropriate limitations on the extent of tolerable surface defects resulting from moulding [see 4.2 a)].

7.2 Appearance

Over the significant surface, there shall be no clearly visible plating defects such as blisters, pits, roughness, cracks, non-plated areas, stains or discolorations. The extent to which defects may occur on non-significant surfaces shall be specified by the purchaser. Where rack marks on the significant surface are unavoidable, their position shall be specified by the purchaser. The appearance shall be uniform and of an agreed colour and approved samples of artefacts shall be used for comparison purposes [see 4.1 b) and ISO 16348].

7.3 Thickness of copper or nickel undercoat

The minimum local thickness of a copper undercoat shall be 15 μm and the minimum local thickness of a nickel undercoat shall be 20 μm [see 4.1 f) and Table 1].

7.4 Local thickness

The thickness of a coating specified in the designation shall be the minimum local thickness. The minimum local thickness of an electrodeposited coating shall be measured at any point on the significant surface that can be touched by a ball 20 mm in diameter.

The thickness of the electrodeposited coatings shall be measured by one of the methods given in annex F.

7.5 Ductility

The minimum value of the ductility shall be 8 % for copper, for dp nickel and for semi-bright nickel when tested by the method given in annex C. There shall be no cracks passing across the convex surface of the tested specimen. Small edge cracks shall not constitute a failure.

7.6 Thermal cycling

The thermal cycle test assesses adhesion and monitors the effectiveness of processes for preparing plastics for electroplating. The magnitude of temperature fluctuations in service shall be taken into account when selecting the service condition number and the thermal cycling requirements. The temperature limits appropriate for each service condition number are given in Table A.1.

After having been subjected to three cycles of the thermal cycle test specified in A.3, the coated article shall show no defects visible to the unaided eye or corrected vision, such as cracking, blistering, peeling, sink marks or distortion.

NOTE The use of the thermal cycle test eliminates the need for a separate adhesion test.

7.7 Accelerated corrosion testing

Coated articles shall be subjected to the CASS corrosion test specified in ISO 9227, no earlier than 24 h after electroplating, for the duration stated in Table G.1 as appropriate for the particular service condition number.

NOTE The duration of the corrosion test given in Table G.1 provides a means of controlling the continuity and quality of the coatings and does not necessarily relate to the life or performance of the finished article in actual service.

The duration given in Table G.1 shall be either continuous or shall consist of an appropriate number of 8 h or 16 h periods separated by rest periods of between 1 h and 16 h, as agreed between the purchaser and the electroplater.

A protection rating shall be assigned in accordance with ISO 10289 to each article tested representing the extent to which the nickel plus chromium coating prevents corrosion of the copper or nickel undercoat, and exposure of the plastic substrate. Alternatively, a one-number rating based solely on the appearance of the parts after corrosion testing may be assigned. For compliance with this International Standard, the appearance rating shall be no less than 8 h after corrosion testing.

NOTE Surface deterioration of the coating itself is expected to occur during the testing of some coatings.

7.8 Combined thermal cycle and accelerated corrosion testing

Corrosion testing may be combined with thermal cycle testing for components that are electroplated to the requirements of service conditions numbers 5, 4 and 3. For articles electroplated to service conditions 5 and 4, three cycles are required; for those electroplated to service condition 3, two cycles are required.

The coated articles shall be examined for defects after each cycle of combined thermal cycle-corrosion testing conducted in accordance with annex G.

NOTE The use of combined thermal cycle and corrosion testing replaces the individual tests described in 7.6 and 7.7.

7.9 STEP test requirements

When specified by the purchaser, the electrode potential differences between individual nickel layers shall be measured for multilayer coatings by means of the STEP test described in ASTM B764-94.

In triple-layer nickel coatings, the STEP potential difference between the special high-activity nickel layer and the bright nickel layer is within the range of 15 mV to 35 mV, and the high-activity layer is always more active (anodic) than the bright nickel layer.

The STEP potential difference between the thin nickel layer immediately below the chromium (applied, e.g., to induce microporosity or microcracking) and the bright nickel layer is 0 mV to 30 mV, and the bright nickel layer is always more active (anodic) than the thin nickel layer applied prior to chromium.

NOTE Although universally accepted STEP values have not been established, some agreement exists for required ranges. For example, the STEP potential difference between the semi-bright and bright nickel layer is within the range of 100 mV to 200 mV, and the semi-bright nickel layer is always more noble (cathodic) to the bright nickel layer.

8 Sampling

The method of sampling shall be selected from the procedures given in ISO 4519. The acceptance levels shall be stated by the purchaser [see 4.1 j)].

9 Test methods

With the exception of the methods given in annex E and in annex F, all methods of test shall be carried out no earlier than 24 h after electroplating.

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Annex A (normative)

Thermal cycle test

A.1 Apparatus

The apparatus shall consist of a circulating air heating chamber and cooling chamber sufficiently powered, insulated and controlled to closely maintain the preset temperature.

NOTE The two chambers may be separate or may be built so as to constitute one chamber.

The controller and recorder used for chamber control, calibration and records shall be accurate to $\pm 1\text{ }^{\circ}\text{C}$ of the set temperature. All points within the working area of the test chamber shall remain within $\pm 3\text{ }^{\circ}\text{C}$ of the set temperature. The air circulation shall be controlled to permit a consistent rate of heating or cooling of the parts during testing.

A.2 Elapsed time after electroplating

The elapsed time between completion of the electroplating operation and thermal cycle testing influences the results. The elapsed time shall be $24\text{ h} \pm 2\text{ h}$.

A.3 Test procedure

Parts may be introduced into the chamber un-mounted, or mounted in a manner simulating assembly, as specified by the purchaser. The chamber is loaded with the desired quantity of parts to be tested. The location of the parts within the chamber is recorded, as well as the number and size of the parts. The thermal cycle temperature limits corresponding to the specified service condition number shall be chosen from Table A.1.

One full thermal cycle shall consist of either placing the parts in a room-temperature chamber and heating the chamber to the high limit, or placing the parts directly into the chamber at the high limit, and performing steps a) to d).

- a) expose the parts for 1 h at the high temperature limit;
- b) allow the parts to return to $20\text{ }^{\circ}\text{C} \pm 3\text{ }^{\circ}\text{C}$ and maintain at this temperature for 1 h (this is usually done by removing the parts from the chamber);
- c) expose the parts for 1 h at the low temperature limit;
- d) allow the parts to return to $20\text{ }^{\circ}\text{C} \pm 3\text{ }^{\circ}\text{C}$ and maintain at this temperature for 30 min.

Table A.1 — Thermal cycle temperature limits

Service condition number	Temperature limits $^{\circ}\text{C}$	
	High	Low
5	85	−40
4	80	−40
3	80	−30
2	75	−30
1	60	−30

Annex B

(informative)

Examples of service conditions for which the various service condition numbers are appropriate

B.1 Service condition number 5

Extended service outdoors in exceptionally severe conditions where long-time preservation (greater than five years) of the decorative coating is required.

B.2 Service condition number 4

Service outdoors in very severe conditions.

B.3 Service condition number 3

Service outdoors where occasional or frequent wetting by rain or dew may occur.

B.4 Service condition number 2

Service indoors where condensation may occur.

B.5 Service condition number 1

Service indoors in warm, dry atmospheres.

Annex C (normative)

Ductility test

C.1 Preparation of test specimen

Prepare an electroplated test specimen 150 mm long, 10 mm wide and 1 mm thick as follows.

Polish a sheet of soft brass, the length and breadth of which both exceed those of the final specimen by about 50 mm. Plate the sheet on one side with nickel (or copper) to a thickness of 25 µm under the same conditions and in the same bath as the corresponding articles.

Cut the test specimen from the plated sheet using a guillotine. Round or chamfer the longer edges of the test specimen at least on the plated side, by careful filing or grinding.

C.2 Procedure

Bend the test specimen with the plated side in tension, by steadily applied pressure, through 180° over a mandrel of diameter 11,5 mm until the two ends of the test specimen are parallel. Ensure that contact between the test specimen and the mandrel is maintained during bending.

C.3 Evaluation

If there are no cracks across the convex surface of the tested specimen, the percent elongation of the coating is greater than 8, as calculated from the following formula:

$$E = 100 \times T / (D + T)$$

where

E is the elongation expressed as a percentage;

T is the total thickness of the base metal and deposit;

D is the diameter of the mandrel.

To calculate E , identical units for T and D shall be used.

For comparison purposes, all test specimens shall have approximately the same coating and total thickness.

This method is an adaptation of the method described in ISO 8401.

Annex D

(normative)

Determination of sulfur content of electrodeposited nickel

D.1 Determination by combustion and iodate titrimetry

The sulfur content of electrodeposited nickel shall be determined, when required, by combustion of a test portion of the nickel in a stream of oxygen in an induction furnace. The sulfur dioxide that is evolved is absorbed in an acidified potassium iodide/starch solution. The solution is then titrated with potassium iodate solution that has been freshly standardized against steels of known sulfur content to compensate for day-to-day variations in sulfur dioxide recovery. Compensation is made for the blank to allow for the effects of crucibles and accelerators.

This method is applicable to electrodeposited nickel having sulfur contents, expressed as S, in the range of 0,005 % mass fraction to 0,5 % mass fraction.

NOTE Commercial instruments are available that utilize infra-red and thermal conductivity detection methods to measure the sulfur dioxide produced by combustion and that have computer facilities that permit direct read-out of sulfur content.

D.2 Determination by sulfide formation and iodate titrimetry

Alternatively, the sulfur content of electrodeposited nickel shall be determined by converting the sulfur in the nickel to hydrogen sulfide by treatment with hydrochloric acid containing dissolved hexachloroplatinic acid, as an accelerator for dissolution. The hydrogen sulfide that is evolved is reacted with ammoniacal zinc sulphate. The zinc sulfide that is formed is titrated with standard volumetric potassium iodate solution. Results are based on potassium iodate as the primary standard.