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# International Standard



# 4525

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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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**Descriptors** : plastics, coatings, metal coatings, electrodeposited coatings, nickel coating, chromium plating, specifications.

## 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.

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 4525 was prepared by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings*.

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

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

## 0 Introduction

This International Standard specifies requirements for a range of electroplated coatings of nickel plus chromium on plastics materials for various conditions of service. No distinction is made between the types of plastics material that are suitable for electroplating and no detailed requirements are laid down concerning the surface condition of the plastics material or the level of moulding stresses.

It is essential that the purchaser should state the service condition number, and if desired, the classification number. Merely to ask for plating to be carried out in accordance with ISO 4525 is insufficient.

## 1 Scope and field of application

This International Standard specifies requirements for electroplated coatings of nickel plus chromium, with a copper undercoat in many cases, on plastics materials. It is not applicable to such coatings on plastics used for engineering purposes.

## 2 References

ISO 1462, *Metallic coatings — Coatings other than those anodic to the basis metal — Accelerated corrosion tests — Method for the evaluation of the results.*

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

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

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

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

ISO 2859, *Sampling procedures and tables for inspection by attributes.*<sup>1)</sup>

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

ISO 3769, *Metallic coatings — Acetic acid salt spray test (ASS test).*

ISO 3770, *Metallic coatings — Copper-accelerated acetic acid salt spray test (CASS test).*

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

## 3 Definition

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

(Definition taken from ISO 2064.)

## 4 Information to be supplied to the electroplater

When ordering articles to be electroplated in accordance with this International Standard, the purchaser shall state, in addition to the number of this International Standard, ISO 4525, the service condition number denoting the severity of the conditions the coating is to withstand (see clause 5) and, if desired, the classification number of the particular coating required (see clause 7). If the service condition number is quoted and not the classification number, the supplier shall be free to supply any of the classes of coating corresponding to the service condition number, but shall inform the purchaser of the classification number of the coating supplied.

The purchaser shall state the appearance required, for example bright or dull or satin. Alternatively, a sample showing the required finish shall be supplied or approved by the purchaser.

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

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

## 5 Grading of service conditions

The service condition number comprises

a) a number from 1 to 4 indicating the severity of the conditions that the coating is likely to encounter in service (the highest number denoting the most severe conditions). Typical service conditions for which the various service condition numbers are appropriate are

- service condition No. 4 — service outdoors in exceptionally severe corrosive conditions
- service condition No. 3 — service outdoors in normal conditions
- service condition No. 2 — service indoors in places where condensation may occur, for example kitchens and bathrooms
- service condition No. 1 — service indoors in warm dry atmospheres, for example offices;

b) a letter A, B or C (see 8.6) indicating the temperature conditions that are to be used in the thermal cycling test.

## 6 Substrate requirements

The plastics materials to be electroplated with nickel and chromium shall have properties which enable the electroplated coatings of nickel and chromium to conform to the requirements of this International Standard, provided that those coatings have been correctly applied. Where plastics artefacts are produced by some technique which involves a change of phase, such as moulding, then the electroplating operation shall not take place until at least 24 h has elapsed after production.

## 7 Classification number

The classification number comprises

- a) the letters PL, indicating a plastics basis material, followed by an oblique stroke;
- b) if a copper undercoat is specified,<sup>1)</sup> the chemical symbol Cu, followed by a number indicating the minimum local thickness (see ISO 2064), in micrometres, of that undercoat (see 8.1);
- c) the chemical symbol Ni for nickel;
- d) a number indicating the minimum local thickness (see ISO 2064), in micrometres, of the nickel coating;
- e) a letter designating the type of nickel deposit (see 8.2);
- f) the chemical symbol Cr for chromium;

g) a letter or letters designating the type of chromium deposit (see 8.3).

For example the complete classification number for a coating comprising 15 µm (minimum) bright nickel plus 0,3 µm (minimum) micro-cracked chromium is

PL/Ni15b Cr mc

## 8 Coating requirements

NOTE — Because of the difference in the thermal coefficient of expansion of the plastics material and the coating, it is essential that the coating should possess sufficient ductility. This may be achieved by using either ductile copper or ductile nickel for the first electro-deposited layer.

### 8.1 Undercoats

To ensure good adhesion and compliance with 8.6, certain plastics materials require a ductile undercoat. In the case of a copper undercoat, the minimum local thickness at any point on the significant surface that can be touched by a ball 20 mm in diameter shall be 15 µm, but not less than 20 µm is recommended for thermal cycles B and C.

### 8.2 Nickel coating

The number following the chemical symbol Ni indicates the minimum local thickness, in micrometres, of the nickel coating, measured by one of the methods given in 10.1, at any point on the significant surface that can be touched by a ball 20 mm in diameter. The minimum local thickness requirement may also be applied to additional portions of the significant surface if specified by the purchaser.

The type of nickel<sup>2)</sup> shall be designated by the following symbols :

- b : for nickel deposition in the fully bright condition
- s : for dull or satin or semi-bright nickel which shall not have been mechanically polished
- d : for double- or triple-layer nickel coatings, which shall have the properties indicated in table 1

### 8.3 Chromium coating

The thickness of the chromium coating shall be measured by the coulometric method given in 10.1.2 at any point on the significant surface that can be touched by a ball 20 mm in diameter. The minimum local thickness requirement may also be applied to additional portions of the significant surface if specified by the purchaser.

The type of chromium shall be designated by the following symbols placed after the chemical symbol Cr :

1) This item would be omitted if a copper undercoat was not specified or used, but see the note to clause 8.

2) It will usually be possible to identify the type of nickel by microscopical examination of a polished and etched cross-section of an article prepared according to ISO 1463.

r : for regular (i.e. conventional) chromium, having a minimum local thickness of 0,3 µm

mc : for 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 10.3, and having a minimum local thickness of 0,3 µm. With some processes, a substantially greater thickness (about 0,8 µm) will be required to achieve the necessary crack pattern, in which case the minimum local thickness shall be included in the classification number, for example Cr mc (0,8)

mp : for micro-porous chromium, containing a minimum of 10 000 pores per square centimetre when determined by the method specified in 10.3.2 and having a minimum local thickness of 0,3 µm.<sup>1)</sup> The pores shall be invisible to the unaided eye or corrected vision

There may be some loss of lustre after a period of service in the case of mc and mp chromium deposits of 0,3 µm thickness which could be unacceptable in some applications. This tendency can be reduced by increasing the chromium deposit thickness to 0,5 µm in which case the minimum local thickness shall be included in the classification number, as follows

Cr mc (0,5)  
Cr mp (0,5)

### 8.4 Coatings appropriate for each service condition number

Table 2 shows the coating classification numbers appropriate for each service condition number. See clause 5 and 8.6 for the significance of the designations.

### 8.5 Appearance

The electroplated article shall be clean and free from damage. Over the significant surface, the electroplated article shall be free from visible electroplating defects such as blisters, pits, roughness, cracks or areas not coated by any layer, and shall not be stained or discoloured.

### 8.6 Thermal cycling

The thermal cycling test is intended primarily to assess adhesion. Temperature fluctuations in service may cause premature adhesion failures and the magnitude of these fluctuations should be taken into consideration when selecting the thermal cycling requirements. The purchaser shall select one of the following requirements and include the appropriate letter in the service condition number :

- A : temperature limits of 75 °C and 20 ± 5 °C
- B : temperature limits of 75 °C and -20 °C
- C : temperature limits of 75 °C and -40 °C

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**Table 1 – Properties of double- and triple-layer nickel coatings**

Layer	Elongation (%)	Sulfur content* [% (m/m)]	Thickness, as a percentage of total nickel thickness	
			Double layer	Triple layer
Bottom	> 8	< 0,005	> 60	> 50
Middle	—	> 0,15	—	< 10
Top	—	> 0,04 and < 0,15	< 40	< 40
Test method	See 10.2	—	See 10.1.1	

\* The sulfur contents are specified in order to indicate the type of nickel electroplating solution that is to be used. No simple method exists for determining the sulfur content of a nickel deposit on a coated article. An accurate determination is possible on a specially prepared test specimen.

**Table 2 – Coatings on plastics materials \***

Service condition number	Classification number
4A, 4B or 4C	PL/Ni 25 b (or s) Cr mc (or mp) PL/Ni 25 d Cr r (or mc or mp)
3A, 3B or 3C	PL/Ni 20 b (or s or d) Cr r PL/Ni 15 b (or s or d) Cr mc (or mp)
2A, 2B or 2C	PL/Ni 10 b (or s or d) Cr r (or mc or mp)
1A, 1B or 1C	PL/Ni 5 b (or s) Cr r (or mc or mp)

\* Where a copper undercoat is specified, it is not permissible to reduce the thickness of nickel given. See clause 7 for the manner of specifying a copper undercoat.

1) The specified porosity pattern at a minimum chromium thickness of 0,3 µm may be achieved by depositing chromium over a nickel layer which contains a large number of small non-conducting particles.

After having been subjected to the appropriate test specified in 10.4, the coated article shall show no defects, which are visible to the unaided eye or corrected vision, such as cracking, blistering, peeling, sink marks or distortion.

NOTE — The use of this test eliminates the need for a separate adhesion test.

**8.7 Corrosion resistance of the coating metal**

Coated articles shall be subjected to the CASS corrosion test specified in ISO 3770, not earlier than 24 h after electroplating, for the duration stated in table 3 as appropriate for the particular service condition number.

The durations of the corrosion test given in table 3 provide a means of controlling the continuity and quality of the coatings and do not necessarily relate to the service life of the finished article.

**Table 3 — Corrosion test durations appropriate to each service condition number**

Service condition number	Duration of CASS test (h)
4	32
3	16
2	8
1*	—

\* Although no test duration is specified for service condition number 1, such coatings may be subjected, by agreement, to the acetic salt spray specified in ISO 3769 for an agreed period of not more than 8 h.

The durations given in table 3 shall be either continuous or shall consist of an appropriate number of 8 or 16 h periods separated by rest periods of between 1 and 16 h, as agreed between the purchaser and the electroplater.

Assign a rating to each article, tested in accordance with ISO 1462, representing the extent to which the nickel plus chromium coating has remained unpenetrated, thus preventing corrosion of the copper undercoat or exposure of the plastics material. For compliance with this International Standard, the rating for each article shall be not less than 8.

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

**9 Sampling**

The method of sampling shall be selected from the procedures given in ISO 2859 or ISO 4519. The acceptance levels shall be specified by the purchaser.

**10 Test methods**

With the exception of the methods specified in 10.1 and 10.3, all methods of test shall be carried out not earlier than 24 h after electroplating.

**10.1 Measurement of thickness**

**10.1.1 Microscopical method**

Use the method specified in ISO 1463 with, if required, the nitric acid/glacial acetic acid etchant specified therein or, for coatings of nickel on copper, a solution of 1 part by volume of nitric acid ( $\rho \approx 1,42 \text{ g/ml}$ ) to 5 parts by volume of glacial acetic acid.

NOTE — The use of these etchants enables the thickness of the different layers in double- and triple-layer nickel coatings to be distinguished and hence measured.

**10.1.2 Coulometric method**

Use the method specified in ISO 2177.

**10.1.3 Magnetic method** (applicable to nickel coatings only)

Use the method specified in ISO 2361.

**10.1.4 Beta backscatter method** (applicable only in the absence of copper undercoats)

Use the method specified in ISO 3543.

NOTE — This method determines the total coating thickness, including that of a copper undercoat, if present. The thickness of this undercoat can, however, be distinguished from that of the outer coating by using this method in conjunction with that specified in ISO 2177, for nickel and chromium coatings, or in conjunction with that specified in ISO 2361, for nickel coatings.

**10.2 Ductility test**

**10.2.1 Preparation of test specimen**

Prepare an electroplated test specimen 150 mm long, 10 mm wide and 1 mm thick by the following method.

Polish a sheet of soft brass, the length and breadth of which both exceed those of the final test specimen by at least 50 mm. Plate the sheet on one side with nickel to a thickness of 25  $\mu\text{m}$  under the same conditions and in the same bath as the corresponding articles.

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

**10.2.2 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.

### 10.2.3 Assessment

The plating is deemed to comply with the minimum requirement of an elongation of 8 % provided that, after testing, there are no cracks passing completely across the convex surface. Small cracks at the edges do not signify failure.

## 10.3 Determination of cracks and pores in chromium coatings

### 10.3.1 General

Micro-cracking can usually be detected by direct microscopical examination without pre-treatment. However, the copper deposition method (10.3.3) is recommended for use, in cases of dispute, as a means of revealing cracks and is necessary to reveal micropores.

### 10.3.2 Microscopical examination for cracks without pre-treatment

Examine the surface for cracks in reflected light under an optical microscope at a suitable magnification. Use a micrometer eyepiece or similar device for indicating the distance over which cracks are counted.

Carry out the determination over a measured length so that at least 40 cracks are counted.

### 10.3.3 Copper deposition method for cracks and pores

#### 10.3.3.1 Principle

Electrodeposition of copper from an acid sulfate solution at low current density or low voltage occurs only on the underlying nickel which is exposed through cracks, pores and other discontinuities.

This method may be used as a rapid means of visually assessing the uniformity of cracks or pores or for counting them. In the latter case a microscope should be used.

#### 10.3.3.2 Procedure

The test is best applied immediately on completion of the electroplating process. If there is any delay, degrease the test specimen thoroughly prior to testing, avoiding any electrolytic treatment.

Using the test specimen as the cathode, deposit copper on to it for approximately 1 min in a bath containing a solution of approximately 200 g/l of copper(II) sulfate pentahydrate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) and 20 g/l of sulfuric acid ( $\text{H}_2\text{SO}_4$ ) maintained at  $20 \pm 5$  °C and using an average current density of 30 A/m<sup>2</sup>. (See the note.)

In cases where the test is applied several days after chromium deposition, immerse the test specimen in a solution containing

10 to 20 g of nitric acid per litre for 4 min at approximately 65 °C before the copper deposition stage, to help reveal the cracks or pores.

Carry out the determination over a measured length so that at least 40 cracks or at least 200 pores are counted.

NOTE — Before immersing them in the bath, the test specimen and the anodes should first be connected to the current supply.

## 10.4 Thermal cycling

### 10.4.1 Apparatus

**10.4.1.1 Oven**, capable of being maintained at a temperature of  $75 \pm 2$  °C.

**10.4.1.2 Refrigerator**, capable of being maintained at a temperature of  $-40 \pm 2$  °C.

### 10.4.2 Procedure

Subject the electroplated articles, not less than 24 h after plating, to one of the following sets of conditions, according to the thermal cycling requirements indicated in the service condition number (see 8.6).

#### 10.4.2.1 Thermal cycle A

Four cycles, each cycle consisting of

- 1 h at a temperature of 75 °C;
- 1 h (minimum) at a temperature of  $20 \pm 5$  °C.

#### 10.4.2.2 Thermal cycle B

Four cycles, each cycle consisting of

- 1 h at a temperature of  $-20$  °C;
- 1 h (minimum) at a temperature of  $20 \pm 5$  °C;
- 1 h at a temperature of 75 °C;
- 1 h (minimum) at a temperature of  $20 \pm 5$  °C.

#### 10.4.2.3 Thermal cycle C

Four cycles, each cycle consisting of

- 1 h at a temperature of  $-40$  °C;
- 1 h (minimum) at a temperature of  $20 \pm 5$  °C;
- 1 h at a temperature of 75 °C;
- 1 h (minimum) at a temperature of  $20 \pm 5$  °C.

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