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# International Standard



# 4526

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

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## Metallic coatings — Electroplated coatings of nickel for engineering purposes

*Revêtements métalliques — Dépôts électrolytiques de nickel pour usages industriels*

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**Descriptors:** metal coatings, electrodeposited coatings, nickel coating, specifications, tests, determination, thickness, porosity.

## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

International Standard ISO 4526 was developed by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings*, and was circulated to the member bodies in February 1982.

It has been approved by the member bodies of the following countries:

|                     |                       |                |
|---------------------|-----------------------|----------------|
| Australia           | Ireland               | Spain          |
| Czechoslovakia      | Italy                 | Sweden         |
| Egypt, Arab Rep. of | Japan                 | Switzerland    |
| France              | Netherlands           | United Kingdom |
| Germany, F. R.      | Poland                | USA            |
| Hungary             | Romania               | USSR           |
| India               | South Africa, Rep. of |                |

No member body expressed disapproval of the document.

# Metallic coatings — Electroplated coatings of nickel for engineering purposes

## 0 Introduction

Electroplated coatings of nickel can be produced with a wide range of mechanical properties and typical values, obtainable commercially, are given in annex A. Electroplated coatings for engineering applications are usually much thicker than those specified in ISO 1458 and are, therefore, often called "thick nickel" or "heavy nickel" coatings. They are applied for their physical or mechanical properties, rather than for their appearance. They are used for engineering purposes in industry mainly to take advantage of one or more of the following properties :

- a) strength, toughness and ductility;
- b) wear resistance;
- c) load-bearing qualities;
- d) corrosion resistance;
- e) resistance to heat-scaling;
- f) resistance to fretting.

Close liaison between designers, manufacturers, electroplaters and purchasers is desirable in order to obtain satisfactory electroplating and optimum corrosion resistance, and to avoid any adverse effects on the mechanical properties of the article. In particular, nickel coatings applied to some steels may lead to a reduction in fatigue strength. This effect can be minimized by peening the surface of the steel before electroplating.

Nickel coatings are used for engineering purposes :

- a) to provide special properties on new components;
- b) to replace metal lost in service due to corrosion or wear or on parts which have been machined to the wrong size;
- c) to provide an undercoat for heavy chromium coatings (see ISO 6158).

For the above purposes, there is no technical limit to the thickness of nickel which can be deposited. Undercoats are not normally required but they may be necessary on some metals.

In view of the wide variety of industrial uses of nickel coatings, it is not possible to specify thicknesses, but the following is intended to indicate typical practice.

- a) **Unmachined coatings for corrosion resistance** : coatings 50 to 250  $\mu\text{m}$  thick, depending on the finish of the basis metal and on the severity of the corrosion conditions.

Usually, the greater the roughness of the basis metal, the greater the thickness of the nickel coating should be so as to avoid porosity.

Examples of use :

- 1) Lehr rolls for glass making;
- 2) food processing equipment.

- b) **Machined coatings on new components, either alone or as an undercoat to chromium coatings** : typical finished thickness 125  $\mu\text{m}$  to 1 mm.

Examples of use :

- 1) compressor rods;
- 2) external surfaces of cylinder liners in contact with cooling liquids.

- c) **Machined coatings on worn components for salvage purposes, either alone or as an undercoat to chromium coatings** : thicknesses may be up to 15 mm or more depending on requirements.

Examples of use :

- 1) rams for use in hydraulic equipment;
- 2) machine parts in general, for example shafts.

## 1 Scope and field of application

This International Standard specifies requirements for electroplated coatings of nickel on ferrous and non-ferrous metals for engineering purposes.

Coatings primarily for decorative purposes are excluded, as these are covered by ISO 1458. For applications where corrosion resistance is also required, ISO 1458 specifies coatings of minimum thickness up to 30  $\mu\text{m}$ ; for thicker coatings, the requirements of this International Standard apply.

## 2 References

ISO 468, *Surface roughness — Parameters, their values and general rules for specifying surfaces.*

ISO 1458, *Metallic coatings — Electroplated coatings of nickel.*

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 2177, *Metallic coatings — Measurement of coating thickness — Coulometric method by anodic dissolution.*<sup>1)</sup>

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

ISO 2819, *Metallic coatings on metallic substrates — Electrodeposited and chemically deposited coatings — Review of methods available for testing adhesion.*

ISO 4516, *Metallic and related 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 6158, *Metallic coatings — Electroplated coatings of chromium for engineering purposes.*

## 3 Definitions

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

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

**3.2 nickel coating** : An electroplated coating of substantially pure nickel.

## 4 Sampling

If required, select a random sample of the size required by ISO 4519 from the inspection lot. Inspect the articles in this sample for conformity with the requirements of this International Standard and classify the lot as conforming or not conforming to each requirement according to the criteria of the sampling plans in ISO 4519.

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

2) In the case of reclaimed articles, it may be not possible to supply this information and it may, therefore, be difficult, if not impossible, to guarantee the quality of the coating.

## 5 Information to be supplied to the electroplater

The electroplater shall be supplied with the following information :

a) the number of this International Standard, i.e. ISO 4526;

b) the nominal composition, or specification, and metallurgical condition of the basis metal;<sup>2)</sup>

c) the necessity for any stress-relieving treatment before electroplating;

d) the necessity for any treatment to induce compressive stress, for example peening before electroplating;

e) the details of significant surfaces and of any surfaces that are not to be electroplated;

f) any special requirements for, or restrictions on, pre-treatment, for example vapour blasting instead of acid pre-treatment;

g) requirements for any undercoats;

h) the minimum thickness of nickel to be deposited and, if undercoats are applied, the total electroplated thickness (see clause 0 and 7.3). A maximum thickness may be quoted if desired, especially for the building-up of worn or over-machined parts. These dimensions shall be those of the finished surface after any machining of the electroplated part (see 7.3);

j) the type of nickel coating required, i.e. dull, bright, semi-bright, low stress etc.;

k) the final surface finish of the nickel coating, for example as-plated, machined or polished;

m) the type and size, or sizes, of defects with the number of defects per item, surface, or per square decimetre of surface that can be tolerated (see 7.1);

n) requirements for any heat treatment after electroplating;

p) where appropriate, any special requirements, for example hardness, controlled surface roughness (see 7.2), etc.

## 6 Treatment of basis metal before electroplating

### 6.1 General

The significant surface shall be examined by the electroplater for visible surface defects, such as porosity, cracks and undesirable coatings, or any other defects detrimental to the final finish. Any defects shall be brought to the attention of the purchaser prior to any processing.

### 6.2 Stress relief

Before being electroplated, parts shall, if specified, be stress relieved. The conditions set out in table 1 shall normally be used, but different conditions, that is, suitable combinations of a shorter time 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 1 — Stress relief conditions before electroplating**

| Maximum specified tensile strength of steel, $R_m$ (MPa) | Heat treatment                         |
|--|--|
| $R_m < 1\ 050$   | None required                          |
| $1\ 050 < R_m < 1\ 450$                                  | 1 h minimum at between 190 and 220 °C  |
| $1\ 450 < R_m < 1\ 800$                                  | 18 h minimum at between 190 and 220 °C |
| $R_m > 1\ 800$   | 24 h minimum at between 190 and 220 °C |

If stress relief is performed after peening or other cold working processes, the temperature shall not exceed 220 °C. Parts with surface-hardened areas shall be stress relieved at 130 to 150 °C for not less than 5 h, but shorter times at higher temperatures may be used if the resulting loss of surface hardness of the substrate is acceptable.

Stress-relieving heat treatment is not normally required for non-ferrous metals.

### 6.3 Peening

#### 6.3.1 Steels

If peening is necessary to improve the fatigue strength, the peening intensity, unless otherwise specified, shall be such that, when measured by the method described in annex B, the arc height is at least

- 0,3 mm for steels of tensile strength less than 1 100 MPa;
- 0,4 mm for steels of tensile strength 1 100 MPa or greater.

NOTE — Lower intensities may be necessary on thin sections to avoid distortion, but may not be fully effective in avoiding loss in fatigue strength.

Unless otherwise specified, the peening shall be performed so that the area concerned is completely covered, i.e. the ball marks completely overlap each other.

### 6.3.2 Non-ferrous metals

For non-ferrous metals, the peening intensity shall be specified by the purchaser.

## 7 Coating requirements

### 7.1 Appearance

The appearance of the significant surface in the as-plated condition shall be as described in annex A and, when inspected by the naked eye, shall be free from pits, cracks, blisters, exfoliation and growths or any other defects detrimental to the final finish.

Coatings that are to be finished by machining may have slight surface blemishes in the as-plated condition provided that these will be eliminated by the machining operation.

Blisters or cracks produced by heat treatment or grinding procedures, if carried out by the electroplater, and that are visible to the naked eye, shall be cause for rejection.

### 7.2 Surface finish

If a specified final surface roughness is required, the method of measurement shall be as specified in ISO 468.

NOTE — As a guide, for ground finishes  $0,4\ \mu\text{m}\ R_a$  may be regarded as a "commercial finish" and  $0,2\ \mu\text{m}\ R_a$  as a "good commercial finish".

### 7.3 Thickness

The finished minimum thickness of nickel on the significant surface and of any undercoat(s), shall be as specified by the purchaser [see clause 5, item h)].

### 7.4 Hardness

Hardness values, if required, shall be measured by the method specified in ISO 4516.

### 7.5 Adhesion

No universally satisfactory test for the adhesion of nickel coatings to the basis metal is known. However, a bend test on a representative sample, electroplated with 25  $\mu\text{m}$  of nickel, can be applied as a test of the effectiveness of the process. A review of methods of testing adhesion is given in ISO 2819, which includes a thermal shock test that has been found applicable in certain cases.

### 7.6 Porosity

If specified, electroplated ferrous articles shall be subjected to the porosity test described in 9.2. The results of the test used shall be evaluated in accordance with ISO 1462 and the rating so assigned shall be at least 8, unless otherwise specified.

**8 Heat treatment after electroplating**

**8.1 General**

If required by the purchaser, heat treatment after electroplating shall be performed as described in 8.2 and 8.3. The heat treatment shall be performed as soon as possible and not later than 4 h after electroplating and before any grinding or other mechanical finishing operation.

**8.2 Heat treatment of steels for reduction of hydrogen embrittlement**

The heat treatment of electroplated steel articles for the reduction of hydrogen embrittlement shall be in accordance with the requirements given in table 2.

**Table 2 — Treatment of steels after electroplating**

| Maximum specified tensile strength of steel, $R_m$ (MPa) | Heat treatment for reduction of hydrogen embrittlement |
|--|--|
| $R_m \leq 1\ 050$  | None required  |
| $1\ 050 < R_m \leq 1\ 450$                               | 8 h minimum at between 190 and 220 °C                  |
| $1\ 450 < R_m \leq 1\ 800$                               | 18 h minimum at between 190 and 220 °C                 |
| $R_m > 1\ 800$   | 24 h minimum at between 190 and 220 °C                 |

Articles shall not be treated above their tempering temperature.

Unpeened parts may be heated for shorter periods at higher temperatures if the conditions have been shown to be effective (see clause 6).<sup>1)</sup>

Parts having surface-hardened areas shall be heated at 130 to 150 °C for not less than 2 h or at a higher temperature if the resulting loss of hardness of the substrate is acceptable.

**8.3 Heat treatment of aluminium and aluminium alloys to improve adhesion**

It should be noted that the mechanical properties of some aluminium alloys may be adversely affected by heating. If heat treatment can be carried out and is required to give the desired adhesion on aluminium or aluminium alloys, the electroplated article shall be heated to give a temperature rise of 2 to 3 K/min until a temperature of 130 to 140 °C is attained. This temperature shall then be maintained for a period of not less than 2 h and not more than 3 h.

1) For springs made from hard drawn wire or strip, the duration of the heat treatment may be reduced to not less than 8 h.

**9 Test methods**

**9.1 Determination of coating thickness**

Measure the thickness at any place on the significant surface designated by the purchaser. Use a method capable of giving a measurement uncertainty of less than 10 %.

**9.1.1 Direct measurement**

Provided that there is a reference point, measurement before and after electroplating provides a direct reading of thickness. For this purpose, normal engineering instruments, for example micrometers, depth gauges etc., can be used. Measurement of the increase in diameter of cylindrical parts does not provide a true reading of thickness unless the distribution of the deposit is even. Such distribution can be checked, for example, by rotating between centres and using a dial indicator or by the use of indirect methods of measurement.

**9.1.2 Magnetic methods**

Magnetic methods are not suitable for measuring nickel coatings greater than 750 µm in thickness. If appropriate, use the method specified in ISO 2361.

**9.1.3 Metallographic sectioning and microscopical method**

Use the method specified in ISO 1463.

If etching is necessary, the following etchants are suitable :

- a) equal parts by volume of nitric acid ( $\rho = 1,42$  g/ml) and glacial acetic acid;
- b) one part by volume of nitric acid ( $\rho = 1,42$  g/ml) to 5 parts by volume of glacial acetic acid.

**WARNING — Toxic fumes are evolved when these chemicals are mixed.**

**9.1.4 Coulometric method**

Use the method specified in ISO 2177.

This method becomes decreasingly reliable for thicknesses over 50 µm. The test conditions and electrolyte are dependent on the test equipment used and the relevant information should be obtained from the manufacturer's instructions.

### 9.1.5 Profilometric method

Use the method specified in ISO 4518.

### 9.2 Testing for porosity on ferrous articles<sup>1)</sup>

Immerse strips of suitable paper, for example, wet strength filter paper, in a warm (about 35 °C) solution containing 50 g of sodium chloride and 50 g of white gelatine per litre and allow to dry. Just before use, immerse them in a solution containing 50 g of sodium chloride and 1 g of a non-ionic wetting agent per litre, press firmly to make satisfactory contact on to the

cleaned nickel surface to be tested and allow to remain for 10 min. If the papers become dry during the test, moisten them again, in place, with the sodium chloride solution.

Remove the papers and introduce them at once into a solution containing 10 g of potassium hexacyanoferrate(III) per litre.

Sharply defined blue markings will be produced on the papers wherever the basis steel was exposed by discontinuities in the coating leading to attack by the sodium chloride and transference of the iron compounds to the paper. If necessary, the same area may be re-tested.

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1) This is an improvement on earlier “ferroxyl” tests.



## Annex A

### Typical mechanical properties<sup>1)</sup> of nickel deposited from various electrolytes, compared with wrought nickel

| Description                        | Appearance as electroplated | Hardness   | Ductility     | Tensile strength | Internal stress |
|------------------------------------|-----------------------------|------------|---------------|------------------|-----------------|
|                                    |                             | HV         | Elongation, % | MPa              | MPa             |
| Hot-rolled and annealed nickel     | —                           | 90 to 140  | 40 min.       | 38 min.          | —               |
| Watts nickel <sup>2)3)4)</sup>     | Dull, matt                  | 130 to 200 | 23 to 30      | 410              | 150 (tensile)   |
| Sulfamate nickel <sup>2)3)4)</sup> | Dull, matt                  | 160 to 200 | 18            | 410              | 14 (tensile)    |

1) These properties will only be consistently obtained by the use of electrolytes which have been purified so as to remove heavy metal ions and organic contaminants from them.

2) Electroplated at 500 A/m<sup>2</sup>; pH 4,0; 60 °C.

3) By adding ammonium ions to the electrolyte, together with increasing the pH to above 5,5, hardness values of 200 to 400 HV can be achieved, normally at the expense of reduced ductility. Organic addition agents may give brightening of the deposits and may reduce resistance to corrosion.

4) Nickel coatings having higher hardnesses (up to 600 HV), greater wear resistance, resistance to compressive stresses, or levelled surfaces may be required. Small quantities of organic chemicals can be added to the electrolytes to produce these effects in the coatings. Such additives may give brightening of the deposit and may reduce resistance to corrosion. The electrodeposited coatings may contain incorporated sulfur or other substances that might adversely affect properties referred to in this International Standard. Such coatings might also be affected by heat treatment or service at high temperatures.

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## Annex B

### Method for establishing peening conditions

Use a test specimen from carbon steel sheet<sup>1)</sup>, hardness range 400 HV 30 and 500 HV 30 and of thickness 1,6 mm, which has been cut to size of 75 mm × (20 ± 0,2) mm and ground to a thickness of 1,3 ± 0,02 mm.

The deviation from flatness shall not exceed an arc height of 40 µm when measured as described below. With the specimen rigidly held in the fixture shown in the figure,peen it on the exposed side for the same period and under the same conditions as the part to be electroplated.

After peening, remove the specimen from the fixture and measure the curvature of the unpeened surface with a depth gauge, the specimen being supported on four 5 mm diameter balls, forming a rectangle 32 mm × 16 mm. Align the gauge symmetrically on the specimen with its central stylus at the centre of the specimen. Measure the arc height at the centre of the specimen over the gauge length of 32 mm, measuring to the nearest 25 µm. Then adjust the peening conditions, if necessary, to give the required arc height.

Dimensions in millimetres

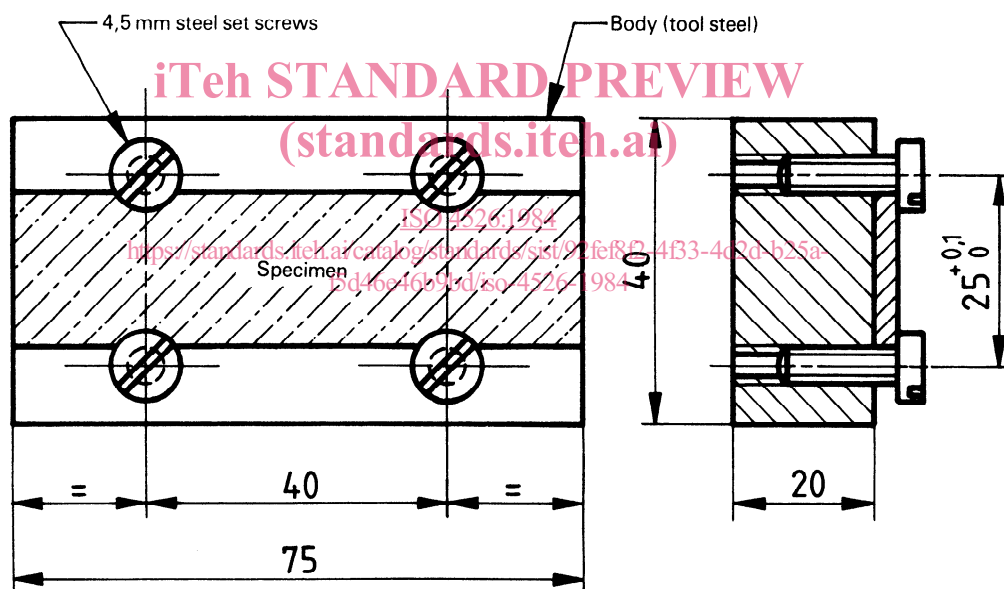


Figure — Fixture for peening test specimens

1) Carbon steel sheet will form the subject of a future International Standard.