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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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Published in Switzerland

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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 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 4526 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 4526:1984), which has been technically revised.

## Introduction

Engineering nickel coatings are specified for various applications such as improved hardness, wear and corrosion resistance, load-bearing characteristics, heat-scaling resistance, corrosion fatigue resistance and other improvements in surface properties. Electrodeposited nickel is also, used in engineering applications to salvage worn or incorrectly machined manufactured articles, and function as diffusion barriers in combination with other metallic coatings. Engineering nickel coatings usually contain greater than 99 % nickel and are most frequently electrodeposited from additive-free Watts or nickel sulfamate solutions. Typical solution compositions, operating conditions and mechanical properties of electrodeposits from these solutions are given in Annex A.

When increased hardness, greater wear resistance, modified deposit internal stress values and enhanced levelling characteristics are required, particles of organic additives such as silicon carbide, tungsten carbide, aluminium oxide, chromium carbide and other substances may be introduced into these solutions. The use of sulfur-containing organic additives to increase hardness and to lower residual internal stress is feasible only when the end-use involves exposure to low or moderate temperatures. High temperature exposure of nickel coatings that contain sulfur may result in embrittlement and cracking of the coating. The effect is time-dependent and may become evident at 150 °C if the time of heating is sufficiently long.

A notable trend is the growing utilisation of nickel alloy electroplating processes for engineering applications. These include binary alloys of nickel with cobalt, iron, manganese, molybdenum, phosphorus and tungsten.



# Metallic coatings — Electroplated coatings of nickel for engineering purposes

## 1 Scope

This International Standard specifies requirements for electroplated nickel and nickel alloy coatings applied to ferrous and non-ferrous basis metals for engineering purposes.

Binary nickel alloys in which nickel is a minor constituent are outside the scope of this International Standard.

The designation provides a means of specifying the type and thickness of nickel and nickel alloy coatings appropriate for engineering applications.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 2064, *Metallic and other inorganic coatings — Definitions and conventions concerning the measurement of thickness*

ISO 2079, *Surface treatment and metallic coatings — General classification of terms*

ISO 2080, *Electroplating and related processes — 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 2819, *Metallic coatings on metallic substrates — Electrodeposited and chemically deposited coatings — Review of methods available for testing adhesion*

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

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

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

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

ISO 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 9220, *Metallic coatings — Measurement of coating thickness — Scanning electron microscope method*

ISO 9587, *Metallic and other inorganic coatings — Pretreatments of iron or steel to reduce the risk of hydrogen embrittlement*

ISO 9588, *Metallic and other inorganic coatings — Post-coating treatments of iron or steel to reduce the risk of hydrogen embrittlement*

ISO 10289, *Methods for corrosion testing of metallic and other inorganic coatings on metallic substrates — Rating of test specimens and manufactured articles subjected to corrosion tests*

ISO 10587, *Metallic and other inorganic coatings — Test for residual embrittlement in both metallic-coated and uncoated externally-threaded articles and rods — Inclined wedge method*

ISO 12686, *Metallic and other inorganic coatings — Automated controlled shot-peening of metallic articles prior to nickel, autocatalytic nickel or chromium plating, or as a final finish*

ISO 15724, *Metallic and other inorganic coatings — Electrochemical measurement of diffusible hydrogen in steels — Barnacle electrode method*

EN 12508, *Corrosion protection of metals and alloys — Surface treatment, metallic and other inorganic coatings — Vocabulary*

### 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 2064, ISO 2079, ISO 2080 and EN 12508 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 5).
- b) The requirements for special test specimens (see 6.1).
- c) The significant surface, indicated by drawings of the articles or by suitably marked samples (see 6.2).
- d) The final surface finish, e.g. as-plated, ground, machined or polished. Alternatively, samples showing the required finish shall be supplied or approved by the purchaser, and used for comparison purposes (see 6.2 and 6.3).
- e) The type and size of defects with the number of defects that can be tolerated per item, for the surface or per square decimetre of surface (see 6.2).
- f) Additional portions of the surface where minimum thickness requirements apply (see 6.4).
- g) The test methods to be used to measure thickness, adhesion and porosity and, if required, those for internal stress and ductility (see 6.4, 6.6, 6.7, 6.11 and 6.12, respectively).
- h) The tensile strength of parts and the requirement for stress relief heat treatment before electroplating (see 6.8).
- i) The requirement for hydrogen embrittlement relief after electroplating, and the hydrogen embrittlement test methods (see 6.9).
- j) The sampling plan and acceptance levels (see Clause 7).

## 4.2 Additional information

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

- a) the nominal composition or specification, and metallurgical condition of the basis metal including hardness (see 5.3);

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

- b) the necessity for peening before or after electroplating (see 6.10);
- c) any special requirements for, or restrictions on, pre-treatment, e.g., vapour blasting instead of acid pre-treatment;
- d) requirements for undercoats and/or overcoats (see 5.5);
- e) where appropriate, any special requirements for surface finish, hardness and adhesion (see 6.3, 6.5 and 6.6, respectively).

## 5 Designation

### 5.1 General

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

The designation specifies, **in the order given**, the basis material, its standard designation (optional), stress relief requirements, the type and thickness of undercoats, the type and thickness of the nickel or nickel alloy coating, the type and thickness of coatings applied over the nickel or nickel alloy coating, and post-treatments including heat treatment.

### 5.2 Components

The designation shall comprise the following:

- a) the term, "Electrodeposited coating";
- b) the number of this International Standard, i.e ISO 4526;
- c) a hyphen;
- d) the chemical symbol of the basis metal (see 5.3);
- e) a solidus (/);
- f) symbols for the nickel or nickel alloy coating, as well as coatings applied prior to and after electroplating, separated by solidi (/) for each stage in the coating sequence in the order of application. The coating designation shall include the thickness of the coatings in micrometres (see 5.5) as well as heat treatment requirements (see 5.4). Double solidi (//) shall be used to indicate that a step has been omitted or is not a requirement.

NOTE It is recommended that the specific alloy be identified by its standard designation following the chemical symbol of the basis metal; e.g., its UNS number, or the national or regional equivalent may be placed between the symbols < >.

EXAMPLE Fe<G43400> is the UNS designation for a high-strength steel.

See [1] to [5].

### 5.3 Designation of the basis metal

The basis metal shall be designated by its chemical symbol or, if an alloy, its principal constituent.

For example;

- a) Fe for iron and steel;
- b) Zn for zinc alloys;
- c) Cu for copper and copper alloys;
- d) Al for aluminium and aluminium alloys.

NOTE To ensure proper surface preparation and hence adherence of the coating to the substrate, it is important to identify the specific alloy and its metallurgical condition (tempered, nitrided, etc.).

### 5.4 Designation of heat treatment requirements

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

- a) the letters SR, for heat treatment for stress relief purposes; the letters ER, for heat treatment for the purpose of reducing susceptibility to hydrogen embrittlement; the letters HT, for heat treatment for other purposes;
- b) in parentheses, the minimum temperature, in degrees centigrade;
- c) the duration of the heat treatment, in hours.

EXAMPLE [SR(210)1] designates stress relief heat treatment at 210 °C for 1 h.

When heat treatment is specified, the requirements shall be included in the designation. See the last example in 5.6.

### 5.5 Type and thickness of metal layers

The electroplated nickel shall be designated by the chemical symbol for nickel, Ni, followed by a number giving the specified minimum local thickness of the coating in micrometres. The type of nickel shall be designated by the symbols given in Table 1 placed after the number that designates coating thickness.

In the case of nickel alloy coatings, the symbols for the alloy coating given in Table 2 shall be followed by a whole number in parentheses giving the nominal composition of the alloy coating, followed by a number giving the specified minimum local thickness of the coating, in micrometres.

EXAMPLE NiCo(35)25 designates a nickel-cobalt alloy coating containing 35 % mass fraction cobalt and which is 25 µm thick.

Metallic undercoats and overcoats deposited electrolytically or by other means shall be designated by the chemical symbol(s) for the deposited metal(s) followed by a number specifying the minimum local thickness of the layer in micrometres.

**Table 1 — Symbols, sulfur content and ductility of different types of nickel coatings**

| Type of nickel                                                                 | Symbol | Sulfur content<br>% mass fraction | Ductility<br>% |
|--------------------------------------------------------------------------------|--------|-----------------------------------|----------------|
| Sulfur-free                                                                    | sf     | < 0,005                           | > 8            |
| Sulfur-containing                                                              | sc     | > 0,04                            | —              |
| Sulfur-free nickel containing particles dispersed throughout the nickel matrix | pd     | < 0,005                           | > 8            |

**Table 2 — Symbols and nominal compositions of electrodeposited binary nickel alloys**

| Nickel alloy                    | Symbol | Nominal compositions<br>% mass fraction |
|---------------------------------|--------|-----------------------------------------|
| Nickel-cobalt                   | NiCo   | 5 to 50 cobalt                          |
| Nickel-iron                     | NiFe   | 10 to 30 iron                           |
| Nickel-manganese                | NiMn   | Approximately 0,5 manganese             |
| Nickel-molybdenum               | NiMo   | 5 to 40 molybdenum                      |
| Nickel-tungsten                 | NiW    | 5 to 40 tungsten                        |
| Electroplated nickel-phosphorus | NiP    | 1 to 30 phosphorus                      |

NOTE Nickel coatings for engineering purposes are most often electrodeposited from Watts and nickel sulfamate solutions, typical compositions of which are given in Annex A. The symbol, sf, refers to nickel electroplated from solutions not containing hardeners, brighteners or stress-reducing agents, the deposits from which are sulfur-free. The symbol, sc, refers to nickel electrodeposits that may contain sulfur or other co-deposited elements or compounds that are present to increase the hardness, refine the grain structure or to control the internal stress of the electrodeposited nickel. Watts and nickel sulfamate solutions can be modified in order to deposit alloys of nickel with cobalt, iron, manganese or phosphorus. The solutions for depositing nickel alloy coatings with either molybdenum or tungsten however, are significantly different from Watts or sulfamate solutions. Proprietary solutions for electrodepositing nickel-molybdenum and nickel-tungsten alloy coatings are reportedly available. For additional technical information on nickel alloy electroplating, see [6] and [7].

## 5.6 Examples of designations

An engineering nickel coating that has a minimum local nickel thickness of 50 µm, that is sulfur-free and that is electrodeposited on carbon steel is designated as follows:

### **Electrodeposited coating ISO 4526 - Fe//Ni50sf**

An engineering nickel coating that has a minimum local thickness of 75 µm, that is sulfur-free, that contains co-deposited silicon carbide particles dispersed throughout the nickel, and that is electrodeposited on an aluminium alloy is designated as follows:

### **Electrodeposited coating ISO 4526 - Al//Ni75pd**

An engineering nickel coating that has a minimum local thickness of 25 µm, that is sulfur-free and that is deposited on a high strength steel that is stress relieved prior to electroplating at 210 °C for 2 h and is also heat treated for embrittlement relief purposes at 210 °C for 22 h is designated as follows:

### **Electrodeposited coating ISO 4526 - Fe/[SR(210)2]/Ni25sf/[ER(210)22]**

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

NOTE The double separators in the first two examples indicate that there are no heat treatment requirements before and after electroplating.

## 6 Requirements

### 6.1 Special test specimens

Special test specimens may be used to measure adhesion, thickness, porosity, corrosion, hardness and other properties when the coated articles are not suitable for the test, or if it is not practical to submit the coated article to destructive tests because the parts are few in number or expensive. Special test specimens shall be of the same material and in the same metallurgical condition, shall have the same surface condition as the coated articles and shall be processed along with the coated articles that they represent.

The use of special or representative test specimens to determine that the requirements of this International Standard have been met, the number of test specimens to be used, the material from which they shall be made, and their shape and size shall be specified by the purchaser [see 4.1 b)].

### 6.2 Appearance

The coating on the significant surface shall be smooth and free from visible defects, such as pits, cracks, blisters, exfoliation, flaking, burned deposits and non-coated areas. The boundaries of electroplating that cover only a portion of the surface, after being machined or otherwise finished as specified by the purchaser, shall be free of beads, nodules, growths, jagged edges, and other defects [see 4.1 c)].

Approved samples of artefacts shall be used for comparison purposes [see 4.1 d)].

Imperfections in the coating which arise from surface conditions of the basis metal and which persist despite the observance of good electroplating practice shall not be cause for rejection. The acceptance limits for defects of the basis metal shall be specified by the purchaser [see 4.1 e)].

Coatings that are to be finished by machining are allowed to have slight surface blemishes in the as-deposited condition provided that these can be eliminated by the machining operation. For articles that are electroplated and subsequently ground to size, the grinding operation shall be performed with a sulfur-free liquid coolant, and with a sufficiently light cut to prevent cracking. Blisters or cracks, visible to the naked eye, produced by heating or grinding operations performed by the electroplater, shall be cause for rejection.

### 6.3 Surface finish

See 4.1 d) and 4.2 e).

NOTE For ground finishes, a surface roughness value,  $R_a$ , of 0,4  $\mu\text{m}$  is termed a commercial finish and 0,2  $\mu\text{m}$  is termed a good commercial finish.

### 6.4 Thickness

The coating thickness specified in the designation shall be the minimum local thickness. The minimum local thickness of an electrodeposited coating shall be measured at any point of the significant surface that can be touched by a ball 20 mm in diameter, unless otherwise specified [see 4.1 f)].

The minimum local thickness of nickel shall be measured by one of the methods given in Annex B. The minimum local thickness is typically 5  $\mu\text{m}$  to 200  $\mu\text{m}$ , depending on the specific engineering application [see 4.1 g)].

Most of the methods given in Annex B are applicable to nickel alloy coatings provided suitable primary or secondary thickness standards are available.

**NOTE** Although there is no technical limitation to the thickness of nickel that can be electroplated, there are practical limitations caused by the size and geometry of the articles that make it difficult to achieve smooth surfaces and uniformity of nickel thickness, especially when nickel thickness is increased. The electroplating operation may have to be interrupted at intermediate stages of processing in order to machine the coated surface so as to meet appearance and surface roughness requirements. If the electroplating operation is interrupted for this purpose, the machined nickel surface must be properly re-activated to ensure adhesion of subsequent nickel deposits. Auxiliary anodes and/or selective shielding may have to be used in order to achieve uniformity of deposit thickness.

## 6.5 Hardness

When hardness is specified, it shall be measured by the method given in ISO 4516 [see 4.2 e)].

## 6.6 Adhesion

The coated article or a special test specimen shall pass the bend, file or thermal shock tests described in ISO 2819. The specific test or tests to be used shall be specified by the purchaser [see 4.1 g)].

**NOTE 1** It is the responsibility of the electroplater to determine that the method for surface preparation prior to electroplating results in a surface capable of meeting the requirements of this subclause.

**NOTE 2** Aluminium alloys may be given a post-electroplating heat treatment at 130 °C to improve adhesion of the coating. That treatment is not recommended for those alloys that would suffer deterioration at or above that temperature.

## 6.7 Porosity

Electroplated ferrous articles or special representative test specimens shall be subjected to the hot water porosity test described in Annex C, or the modified ferroxyl test described in Annex D. After testing, the articles shall be rated in accordance with ISO 10289. The part shall have failed if any degree of porosity is detected, unless otherwise specified by the purchaser [see 4.1 g)].

## 6.8 Stress relief heat treatments prior to coating

When specified by the purchaser, steel parts that have an ultimate tensile strength equal to or greater than 1 000 MPa (31 HRC) and that contain tensile stresses caused by machining, grinding, straightening or cold-forming operations shall be given a stress relief treatment prior to cleaning and metal deposition. The procedures and classes for stress relief treatment shall be as specified by the purchaser or the purchaser may specify appropriate procedures and classes from ISO 9587 [see 4.1 h)].

**NOTE** Steels with oxide or scale shall be cleaned before application of the coatings. For high strength steels, non-electrolytic alkaline and anodic alkaline cleaners, as well as mechanical cleaning procedures, are preferred in order to avoid the risk of producing hydrogen embrittlement during cleaning operations. The possibility of overheating should be considered in the case of mechanical cleaning of high-strength steels (tensile strength > 1 400 MPa).

## 6.9 Hydrogen embrittlement relief heat treatment

Steel parts having an ultimate tensile strength  $\geq$  1 000 MPa (31 HRC), as well as surface hardened parts, shall receive hydrogen embrittlement relief heat treatment according to the procedures and classes of ISO 9588 or as specified by the purchaser [see 4.1 i)].

The effectiveness of the embrittlement relief treatment may be determined by a test method specified by the purchaser or by test methods described in ISO Standards; e.g., ISO 10587 describes a test method for testing threaded articles for residual hydrogen embrittlement relief heat treatment and ISO 15724 for measuring the relative, diffusible hydrogen concentration in steels.

Electroplated springs or other parts subject to flexure shall not be flexed before the hydrogen embrittlement relief heat treatment.

NOTE Nickel and nickel alloy electrodeposits that contain small amounts of sulfur (sc nickel) discolour and become embrittled when heated above 200 °C. The exact temperature of embrittlement depends on the sulfur content of the nickel, and the time at elevated temperature.

## 6.10 Peening

If controlled automatic shot peening, before or after electroplating, is specified by the purchaser, it shall be performed in accordance with ISO 12686. The method of measuring shot peening intensity is also described in that International Standard [see 4.2 b)].

NOTE Shot peening prior to and after electroplating minimizes the reduction in fatigue strength that occurs when high-strength steels are electroplated with coatings that are stressed in tension, and is recommended for parts that are subject to repeated applications of complex load patterns in service. Other factors that affect fatigue life include the thickness of the coating that should be as thin as is compatible with the expected service conditions, and deposit residual internal stress that should be as low as possible. The compressive stresses that result from shot peening increase corrosion resistance and resistance to stress corrosion cracking.

## 6.11 Internal stress

The internal stress of electrodeposited nickel and nickel alloy coatings varies over a wide range. In general, high tensile or compressive stresses in excess of 100 MPa may result in processing difficulties. Electrodeposits from Watts solutions usually have higher internal stresses than deposits from nickel sulfamate solutions. Organic additives that reduce internal stress are available, but must be used with caution because they increase the sulfur content of the electrodeposited coating and may also cause compressive stress. Nickel and nickel alloy coatings containing sulfur are likely to be embrittled when heated above 200 °C during processing or in service, depending on the time at elevated temperature. Levelling agents tend to increase the internal stress of the deposit in the tensile direction [see 4.1 g)].

The spiral contractometer, rigid strip and other methods for measuring the internal stress of coatings are available but have not been standardized at an international level. National standards for the measurement of internal stress exist. The suppliers of equipment for measuring internal stress can also provide details on how the internal stress of electrodeposited coatings may be measured.

## 6.12 Ductility

The ductility of electrodeposited nickel and nickel alloy coatings is an important consideration in applications where bending or forming is necessary after electroplating; e.g., in many electrical and electronic applications. Inadequate ductility may lead to cracking of the coating during forming operations. The ductility of sulfur-free nickel deposits is usually greater than 8 %. The purchaser shall specify the ductility required (see NOTE) and its method of measurement (see ISO 8401). Ductility is often measured by the cylindrical mandrel bending method described in ISO 8401 [see 4.1 g)].

NOTE As indicated in Annex A, the ductility of electrodeposited nickel as measured by percent elongation varies from 10 % to 30 % in the case of deposits from Watts solutions, and from 5 % to 30 % for deposits from nickel sulfamate solutions, depending on pH, temperature and current density. The ductility of sulfur-free nickel deposits can be increased by heat treatment after electroplating.

## 7 Sampling

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

## Annex A

(informative)

### Typical composition and operating conditions of Watts and nickel sulfamate solutions, and mechanical properties of nickel electrodeposited from those solutions

|                                                                                           | Watts Nickel         | Nickel sulfamate  |
|-------------------------------------------------------------------------------------------|----------------------|-------------------|
| <b>Electrolyte composition</b><br>g/l                                                     |                      |                   |
| Nickel sulfate, $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$                                 | 225 to 400           | —                 |
| Nickel sulfamate, $\text{Ni}(\text{SO}_3\text{NH}_2)_2$                                   | —                    | 300 to 450        |
| Nickel chloride, $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$                                | 30 to 60             | 0 to 10           |
| Boric acid, $\text{H}_3\text{BO}_3$                                                       | 30 to 45             | 30 to 45          |
| Temperature, °C                                                                           | 50 to 65             | 40 to 65          |
| Agitation                                                                                 | Air or mechanical    | Air or mechanical |
| <b>Operating conditions</b>                                                               |                      |                   |
| Cathode current density, $\text{A/dm}^2$                                                  | 3 to 8               | 0,5 to 30         |
| Anodes                                                                                    | Nickel               | Nickel            |
| pH                                                                                        | 3,5 to 4,5           | 3,8 to 4,2        |
| <b>Typical mechanical properties</b>                                                      |                      |                   |
| Tensile strength, MPa                                                                     | 345 to 485           | 415 to 610        |
| Elongation, %                                                                             | 10 to 30             | 5 to 30           |
| Vickers hardness, 100 g load                                                              | 130 to 200           | 170 to 230        |
| Internal stress, MPa                                                                      | 125 to 185 (tensile) | 0 to 55 (tensile) |
| NOTE Anti-pitting agents for nickel electroplating are normally added to control pitting. |                      |                   |

## **Annex B** (informative)

### **Test methods for determining thickness**

#### **B.1 General**

ISO 3882 reviews methods of measuring the thickness of metallic and other inorganic coatings.

#### **B.2 Destructive tests**

##### **B.2.1 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 copper plus nickel, a solution of 1 part by volume of nitric acid (relative density = 1,4 g/ml) to 5 parts by volume of glacial acetic acid.

##### **B.2.2 Coulometric method**

The coulometric method specified in ISO 2177 may be used to measure the total thickness of the nickel and the thickness of a copper underlayer, when present, at any point on the significant surface which can be touched by a ball 20 mm in diameter.

##### **B.2.3 Scanning electron microscope method**

The scanning electron microscope method described in ISO 9220 may be used to measure nickel thickness and the thickness of underlayers.

**NOTE** In cases of dispute, the coulometric method may be used for measuring the thickness of nickel coatings less than 10 µm thick, and the microscopical method may be used for measuring the thickness of nickel coatings and undercoats 10 µm thick and above.

#### **B.3 Non-destructive tests**

##### **B.3.1 Magnetic method** (applicable to nickel coatings only)

Use the method specified in ISO 2361.

**NOTE** This method is sensitive to variations in the permeability of coatings.

##### **B.3.2 Beta backscatter method** (applicable only in the absence of copper undercoats)

Use the method specified in ISO 3543. This method is suitable for measuring a nickel coating on an aluminum substrate. This method determines the total coating thickness, including that of a copper undercoat, if present.

##### **B.3.3 X-Ray spectrometry**

Use the method specified in ISO 3497.

## Annex C (normative)

### Hot water porosity test

#### C.1 General

This method reveals discontinuities such as pores, in electroplated nickel on ferrous articles. It is non-corrosive to nickel.

#### C.2 Materials

A stainless steel or rubber-lined or glass vessel equipped to suspend the part that shall be insulated from contact with metal vessels shall be used. The significant electroplated areas shall be totally immersed in clean distilled or deionized water. The pH of the water shall be maintained between 6,0 and 7,5 and the conductivity shall be no greater than 0,5  $\mu\text{S/m}$ . Additives required for pH control shall be non-corrosive to nickel and shall be approved by the purchaser; e.g., pH can be adjusted by introducing  $\text{CO}_2$  or by additions of  $\text{H}_2\text{SO}_4$  or acetic acid or  $\text{NaOH}$ . A source of oil-free air shall be available to aerate the water with agitation vigorous enough to prevent air bubbles from clinging to the significant surfaces of the parts.

#### C.3 Procedure

Clean and degrease the electroplated surface to be tested in order to provide a water break-free surface. Totally immerse the electroplated areas of the part in the water which has been heated to 85 °C. The 60 min test period starts when the water temperature is in equilibrium with the part at 85 °C  $\pm$  3 °C. This temperature shall be maintained throughout the 60 min test period. At the end of the test period, remove the part from the hot water, and allow the part to drain and dry. Oil-free air pressure may be used to speed the drying. Black spots or red rust indicate basis metal corrosion or porosity.

#### C.4 Test report

The following information shall be included in the test report:

- a) the area of surface tested;
- b) the total number and diameter of all spots visible to the unaided eye;
- c) the highest number of spots visible within a square area as defined and specified by the purchaser.