INTERNATIONAL
STANDARD

**ISO**
**4527**

Second edition
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# Metallic coatings — Autocatalytic (electroless) nickel-phosphorus alloy coatings — Specification and test methods

*Revêtements métalliques — Dépôts autocatalytiques (sans courant) d'alliages de nickel-phosphore — Spécifications et méthodes d'essai*

Reference number
ISO 4527:2003(E)

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# Contents

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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 4527 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 4527:1987), which has been technically revised.

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# Introduction

Autocatalytic nickel-phosphorus alloy coatings are produced by the catalytic reduction of nickel ions in hot, usually mildly acidic solutions at atmospheric pressure using hypophosphite ion as the reducing agent. Because the deposited nickel alloy is a catalyst for the reaction, the process is self-sustaining. The coatings produced are uniform in thickness on irregularly shaped parts if the processing solution circulates freely over their surfaces.

The as-deposited coating is a thermodynamically metastable, supersaturated solid solution of phosphorus in nickel containing up to 14 % mass fraction phosphorus. The physical and chemical properties and the structure of autocatalytic nickel-phosphorus coatings are dependent on the composition of the coating, the chemical make-up of the plating solution, the pre-treatment and quality of the substrate, and heat treatment after deposition.

Autocatalytic nickel-phosphorus coatings are applied in order to improve corrosion protection and to provide wear resistance. In general, corrosion performance is significantly improved as the phosphorus content of the deposit is increased to 8 % mass fraction or higher, whereas wear resistance is improved as the phosphorus content of the coating is decreased below that level. With suitable heat treatment however, coatings with high phosphorus contents display greatly improved microhardness and hence, wear resistance.

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**INTERNATIONAL STANDARD** **ISO 4527:2003(E)**

# Metallic coatings — Autocatalytic (electroless) nickel-phosphorus alloy coatings — Specification and test methods

## 1 Scope

This International Standard specifies the requirements and test methods for autocatalytic nickel-phosphorus alloy coatings applied from aqueous solutions on to metallic substrates.

This International Standard does not apply to autocatalytic nickel-boron alloy coatings, nickel-phosphorus composites and ternary alloys.

**WARNING — The use of this International Standard may involve hazardous materials, operation and equipment. This International Standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices, and determine the applicability of regulatory limitations prior to use.**

## 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, *Metal 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[1], *Surface treatment, metallic and other inorganic coatings — Vocabulary*

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

ISO 2178, *Non-magnetic coatings on 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 2859-1, *Sampling procedures for inspection by attributes — Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection*

ISO 2859-2, *Sampling procedures for inspection by attributes — Part 2: Sampling plans indexed by limiting quality (LQ) for isolated lot inspection*

ISO 2859-3, *Sampling procedures for inspection by attributes — Part 3: Skip-lot sampling procedures*

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[1] To be published. (Revision of ISO 2080:1981)

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ISO 2859-4, *Sampling procedures for inspection by attributes — Part 4: Procedures for assessment of declared quality levels*

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

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

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

ISO 4288, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Rules and procedures for the assessment of surface texture*

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 4526[2)], *Metallic coatings — Electroplated coatings of nickel and nickel alloys for engineering purposes*

ISO 6158, *Metallic coatings — Electrodeposited coatings of chromium for engineering purposes*

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

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

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 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*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 2064, ISO 2079, ISO 2080, ISO 9587 and ISO 9588 apply. See [1] for European terminology.

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2) To be published. (Revision of ISO 4526:1984)

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# 4 Information to be supplied to the producer of the coating

## 4.1 Essential information

When ordering articles to be coated in accordance with this International Standard, the purchaser shall provide the following information for all essential items, in writing, as part of the contract, the purchase order, the detailed product specification and/or on engineering drawings:

a) the coating designation (see clause 5);

b) the tensile strength of the part and the requirements for any heat treatment before and after coating deposition (see 6.2, 6.9, 6.10, 6.11, 6.12 and Annex A);

c) the significant surface, indicated by drawings of the articles or by suitably marked samples;

d) the nature, condition and finish of the basis metal, if any of these can affect the serviceability and/or the appearance of the coating (see 6.2);

e) the location, type and dimensions where defects, such as rack marks, may be tolerated (see 6.2);

f) the finish required, e.g., bright, dull, satin or other finish and, if applicable, a sample of the desired finish keeping in mind that approved samples may deteriorate over time and may need to be replaced at regular intervals;

g) any requirements for undercoats (see 6.17);

h) sampling methods, acceptance levels, or other inspection requirements, if different from those given in ISO 4519 (see Clause 7);

i) standard methods for thickness, hardness, adhesion, porosity, corrosion resistance, wear or solderablility testing (see 6.4, 6.5, 6.6, 6.7, 6.8, 6.13, 6.14, and Annex B), and the requirements for special test specimens (see 6.1);

j) requirement for treatments to induce compressive surface stresses, e.g., shot-peening before coating (see 6.16);

k) special requirements for, or restrictions on, pretreatment;

l) special requirements for, or restrictions on, post-treatment;

m) special requirements for maximum coating thickness, especially for the build-up of worn or over-machined parts. It should also be specified whether these thicknesses shall be measured before or after machining of the coating;

n) special requirements for a coating over the autocatalytic nickel coating (see 6.17).

## 4.2 Additional information

The following additional information may be supplied by the purchaser, as appropriate:

a) the necessity for degaussing (demagnetizing) steel parts before coating to minimize the inclusion of magnetic particles in the coating;

b) the final surface roughness of the coating (see 6.3 );

c) any special requirements for the chemical composition of the coating (see 6.15);

d) any special requirements for recovering rejected articles;

e) any other special requirements.

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## 5 Designation of basis metal, metal layers and heat treatment requirements

### 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 following order, the basis metal, the specific alloy (optional), stress relief requirements, the type and thickness of undercoats, the nominal phosphorus content and thickness of the autocatalytic nickel coating, type and thickness of coatings applied over the autocatalytic layer and post-treatments including heat treatment. Double separators (//) shall be used to indicate that a step or operation has either not been specified or has been omitted.

The designation shall comprise the following:

a) the term “Autocatalytic nickel coating”;

b) the number of this International Standard, i.e., ISO 4527;

c) a hyphen;

d) the chemical symbol of the basis metal (see 5.2);

e) a solidus (/);

f) symbols for the autocatalytic nickel coating (see 5.4), as well as coatings applied prior to and after autocatalytic deposition (see 5.4), separated by solidi for each stage in the coating sequence in the order of application. The coating designation shall include the thicknesses of the coatings in µm, as well as heat treatment requirements (see 5.3).

### 5.2 Designating the basis metal

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

It is recommended that the specific alloy be designated by its standard designation, e.g., its UNS number or the local national equivalent placed between the symbols, < >; e.g., Fe<G43400> is the UNS designation for a high-strength steel. See [2] to [6].

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.).

### 5.3 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 HT, for heat treatment to increase the hardness of the coating or the adhesion of the coating to the basis metal; and the letters ER, for hydrogen embrittlement relief;

b) in parentheses, the minimum temperature, in degrees centigrade;

c) the duration of the heat treatment, in hours.

EXAMPLE

A stress relief treatment at 210 °C for 1 h is designated as follows:

**[SR(210)1]**

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### 5.4 Designating the type and thickness of metal layers

The autocatalytic nickel coating shall be designated by the symbol, NiP, followed by a whole number in parentheses giving the nominal phosphorus content of the coating, followed by a number giving the specified minimum local thickness of the autocatalytic nickel coating, in μm.

Metallic undercoats 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 μm (see 6.17). The symbol Ni is the designation for an electrodeposited nickel undercoat.

Subsequent coatings that are to be deposited upon the autocatalytic nickel layer, e.g., chromium, shall be designated by the chemical symbol of the electrodeposited coating followed by a number specifying the minimum local thickness of the coating, in μm (see 6.17).

### 5.5 Examples of designations

The following are examples of designations:

a) An autocatalytic nickel-phosphorus coating having a nominal phosphorus content of 10 % mass fraction and a thickness of 15 μm applied to G43400 steel, requiring stress relief prior to coating at 210 °C for 22 h, and subsequently electroplated with chromium, 0,5 μm thick, requiring heat treatment for hydrogen embrittlement relief at 210 °C for 22 h is designated as follows:

   **Autocatalytic nickel coating ISO 4527–Fe<G43400>[SR(210)22]/NiP(10)15/Cr0,5[ER(210)22]**

b) The same coating on an aluminium alloy, for which there are no heat treatment requirements, is designated as follows:

   **Autocatalytic nickel coating ISO 4527–Al<A96061-T6>//NiP(10)15/Cr0,5//**

c) The same coating on a copper-base alloy, for which there are no heat treatment requirements, is designated as follows:

   **Autocatalytic nickel coating ISO 4527–Cu<C10800>//NiP(10)15/Cr0,5//**

For ordering purposes, the detailed product specification shall not only comprise the designation, but shall also contain clear statements of the other essential requirements listed in Clause 4.

## 6 Requirements

### 6.1 Special test specimens

Special test specimens may be used to measure adhesion, thickness, porosity, corrosion resistance, hardness and other properties when the coated articles are of a size, shape or material that is not suitable for the test, or if it is not practical to submit the coated articles to destructive tests because the parts are few in number or too expensive. Special test specimens shall be of the same material, shall be 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.

### 6.2 Appearance

The autocatalytic nickel coating on the significant surface shall be bright, semi-bright or dull, as specified by the purchaser and, when visually inspected, shall be free from pits, blisters, exfoliation, nodular growths, cracks and other defects detrimental to the final finish, unless otherwise specified. Approved samples with the specified appearance shall be used for comparison purposes [see 4.1 f)].

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Imperfections and variations that arise from surface conditions of the basis metal (scratches, pores, roll marks, inclusions), and that persist in the finish despite the observance of good metal finishing practices, shall not be cause for rejection. The purchaser shall specify limits for the tolerable defects on the finished and unfinished product. Damaged basis metals shall not be coated.

Blisters or cracks that are visible to the naked eye and that result from heat treatment performed by the producer of the coating shall be cause for rejection.

NOTE Defects that are present in the basis metal before coating, including hidden defects, may be reproduced by the coating. In addition, stains and the formation of coloured oxides may result from post-coating heat treatment. The latter shall not be cause for rejection unless a special heat treatment atmosphere is specified. It is advisable for the interested parties to agree on the acceptability of such defects.

## 6.3 Surface finish

If a specified final surface roughness is required, the method of measurement shall be that specified in ISO 4288.

NOTE The surface finish of autocatalytic nickel coatings is not usually superior to that of the substrate before coating, except when the surface of the substrate is extremely smooth and micro-levelling occurs.

## 6.4 Thickness

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

Annex C provides guidance on the thickness requirements for corrosion protection under various conditions of service. The thickness shall be measured by one of the methods given in Annex B.

## 6.5 Hardness

When hardness is specified, it shall be measured by the method given in ISO 4516. The measured hardness of the coating shall be within ± 10 % of that specified by the purchaser.

## 6.6 Adhesion

The autocatalytic nickel coating shall be adherent to the substrate, and any metallic undercoats. Coatings shall be capable of passing one or more of the adhesion tests given in ISO 2819, as specified by the purchaser.

## 6.7 Porosity

If required, a maximum degree of porosity of the autocatalytic nickel-phosphorus alloy coating shall be specified by the purchaser together with method of testing for porosity.

## 6.8 Corrosion resistance

If required, the corrosion resistance and the corrosion test method shall be specified by the purchaser who shall specify acceptance criteria in accordance with ISO 10289. The test methods included in ISO 9227, acetic acid salt spray and copper accelerated salt spray, may be specified for evaluating the pitting corrosion resistance of the coatings.

NOTE Corrosion testing in artificial atmospheres does not necessarily relate to the service life or performance of the finished article.

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### 6.9 Stress relief heat treatment prior to coating

When specified by the purchaser [see 4.1 b)], steel parts that have an ultimate tensile strength equal to or greater than 1 000 MPa 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 in accordance with ISO 9587. Stress relief shall be carried out before any acidic or cathodic electrolytic treatments are applied.

NOTE Steels with oxide or scale should 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.

### 6.10 Hydrogen embrittlement relief heat treatment after coating

Steel parts having an ultimate tensile strength equal to or greater than 1 000 MPa, as well as surface hardened parts, shall receive hydrogen embrittlement relief heat treatment after coating according to the procedures and classes given in ISO 9588 or as specified by the purchaser [see 4.1 b)].

Any heat treatment for the relief of hydrogen embrittlement after coating shall be carried out as soon as possible, preferably within 1 h but not later than 3 h after surface finishing and before commencement of grinding or other mechanical operation.

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 method of testing threaded articles for residual hydrogen embrittlement and ISO 15724, one for measuring the diffusible hydrogen concentration in steels.

NOTE Heat treatment in accordance with the procedures and classes given in ISO 9588 does not guarantee complete freedom from hydrogen embrittlement, and tests for residual hydrogen embrittlement should be specified, whenever possible. Freedom from failure of test samples can demonstrate the effectiveness of the hydrogen embrittlement heat treatment procedure, depending on the number of samples that are tested.

### 6.11 Heat treatment to harden the coating

Table A.1 provides guidance on heat treatment to increase the hardness of autocatalytic nickel-phosphorus alloy coatings so as to improve wear resistance (see 6.13).

Heat treatment to increase the hardness of autocatalytic nickel-phosphorus alloy coatings so as to improve wear resistance shall be performed within 1 h of coating, when required. Heat treatment shall be carried out before mechanical finishing. The duration of the heat treatment shall be 1 h minimum after the part has reached the specified heat treatment temperature.

If heat treatment to harden the coating is carried out, separate hydrogen embrittlement relief heat treatment may not be necessary, provided that the requirements of ISO 9588 have been met (see 6.10).

### 6.12 Heat treatment to improve adhesion

Heat treatment to improve the adhesion of autocatalytic nickel coatings on certain basis metals shall be carried out in accordance with Table A.1, unless the purchaser specifies other procedures.

### 6.13 Wear resistance

If required, the wear resistance of the coating shall be specified by the purchaser who shall also specify the wear resistance test method to be used to ensure that the requirement has been met.

NOTE Wear resistance can be affected by heat treating autocatalytic nickel coatings (see 6.11 and Annex A).

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### 6.14 Solderability

If required, the solderability of the coating shall be specified by the purchaser who shall also specify the solderability test method to be used to ensure that the requirement has been met (see C.5).

NOTE Coatings containing greater than 10 % mass fraction phosphorus are sometimes used for soldering in order to minimize the possibility of corrosion during the soldering process, especially in electronic applications. More often, coatings containing low levels of phosphorus (1 % to 3 % mass fraction) are specified for soldering purposes.

### 6.15 Chemical composition

The phosphorus content of the nickel-phosphorus alloy coating shall be that specified in the designation (see 5.4 and Table C.2). When measured by the method given in Annex D, the mass fraction of phosphorus shall be within ± 0,5 % of that required. If the phosphorus content is not specified, it shall be within the range 1 % mass fraction to 14 % mass fraction, unless otherwise specified [see 4.2 c)].

### 6.16 Peening of metal parts

If peening prior to coating is specified by the purchaser, it shall be performed in accordance with ISO 12686 which also describes the method for measuring peening intensity, and shall be carried out before any acidic or cathodic electrolytic treatments.

NOTE Shot-peening prior to coating can minimize the reduction in fatigue strength and adhesion that occurs when high-strength steels are coated with autocatalytic nickel coatings and is recommended for parts subjected to repeated applications of complex load patterns in service. Other factors that affect fatigue strength include thickness, which should be kept as thin as is compatible with the expected service condition. The compressive stresses resulting from controlled shot-peening increase corrosion resistance and resistance to stress corrosion cracking, and can have a beneficial effect on coating adhesion.

### 6.17 Undercoats and overcoats

Electrolytic nickel undercoats [see 4.1 g)] shall comply with ISO 4526. Chromium coatings applied on top of autocatalytic nickel-phosphorus alloy coatings shall comply with ISO 6158.

NOTE Electrodeposited nickel undercoats 2 µm to 5 µm thick may be applied to basis metals (except brass and bronze) that contain more than trace quantities of antimony, arsenic, bismuth, copper, lead or tin. Autocatalytic nickel, electrodeposited nickel or electrodeposited copper undercoats 2 µm to 5 µm thick may be applied to basis metals that contain more than trace quantities of magnesium and zinc. An electrolytic nickel strike between the copper undercoat and the autocatalytic nickel coating may be applied. Electrodeposited nickel undercoats 1 µm to 2 µm thick may be applied to basis metals containing more than trace amounts of chromium, lead, molybdenum, nickel, tin, titanium or tungsten. The purpose of undercoats is to reduce the risk of contaminating the processing solution with elements that may lower the rate of deposition. In addition, electroplated metallic undercoats help prevent the diffusion of impurities from the basis metal into the autocatalytic coating, and help improve adhesion.

## 7 Sampling

The method of sampling shall be selected from the procedures specified in ISO 2859-1, ISO 2859-2, ISO 2859-3, ISO 2859-4 or ISO 4519, or the purchaser shall specify an alternate plan. The purchaser shall specify the acceptance levels.

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# Annex A
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# Heat treatment to improve adhesion and increase hardness

## A.1 Heat treatment to improve adhesion

The times and temperatures given in Table A.1 shall be used to improve the adhesion of autocatalytic nickel-phosphorus alloy coatings deposited directly on various alloys, unless otherwise specified by the purchaser. For coatings thicker than 50 µm, the heat treatment times shall be increased.

NOTE The tensile strength of heat treatable aluminium and various alloys can be reduced by heating above 130 °C. It is recommended that the effect on the mechanical properties of the substrate be considered and, if necessary, verified when the purchaser specifies heat treatment after coating in order to improve adhesion.

## A.2 Heat treatment to improve hardness and wear resistance

Autocatalytic nickel-phosphorus alloy coatings are frequently precipitation hardened by heat treatment in order to improve wear resistance. The times and temperatures given in Table A.1 shall be used.

**Table A.1 — Recommended heat treatments to improve hardness and adhesion**

| Class | Description | Temperature °C | Time h |
|---|---|---|---|
| 1 | No heat treatment; as-deposited | | |
| 2 | Heat treatment for maximum hardness, by type (see Table C.2): | | |
| | 1 | 260 | 20 |
| | | 285 | 16 |
| | | 320 | 8 |
| | | 400 | 1 |
| | 2 | 350 to 380 | 1 |
| | 3 | 360 to 390 | 1 |
| | 4 | 365 to 400 | 1 |
| | 5 | 375 to 400 | 1 |
| 3 | For adhesion on to steel | 180 to 200 | 2 to 4 |
| 4 | For adhesion on to carburized steel and age-hardened aluminium | 120 to 130 | 1 to 6 |
| 5 | For adhesion on to beryllium and non-aged hardened aluminium | 140 to 150 | 1 to 2 |
| 6 | For adhesion on to titanium and titanium alloys | 300 to 320 | 1 to 4 |
| 7 | For adhesion on to magnesium and its alloys; copper and its alloys | 180 to 200 | 2 to 2,5 |
| 8 | For adhesion on to nickel and its alloys | 220 to 240 | 1 to 1,5 |
| 9 | For adhesion on to molybdenum and its alloys | 190 to 210 | 2 to 2,5 |

In general, the hardness after heat treatment increases as the phosphorus content is decreased (Figure A.1). The hardness can be increased further by heat treating between 250 °C and 400 °C for longer than 1 h. Heat treatment above 220 °C that causes the hardness to exceed 850 KHN100 can reduce the corrosion resistance of the coatings. Heat treatment at temperatures below 200 °C to improve adhesion or to minimize the risk of hydrogen embrittlement do not impair corrosion resistance or substantially increase the hardness or improve the wear resistance of the coating. When necessary, the heat treatment should be performed in an inert or reducing atmosphere, or in vacuum to prevent coloured oxides from forming on the surface. Heat treatment above 260 °C will cause type 5 coatings to become magnetic.

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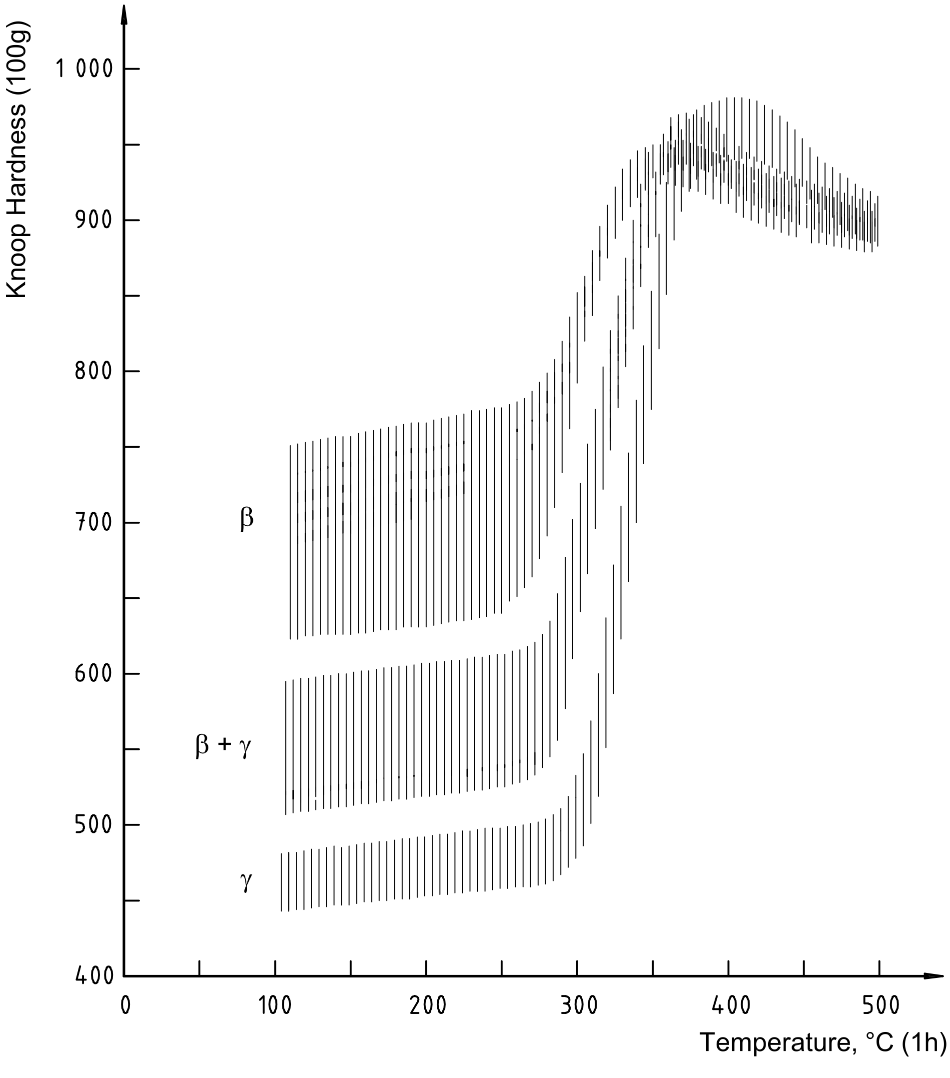
The relationship between heat treatment temperature and hardness after 1 h of heating is shown in Figure A.1 for different types of autocatalytic nickel-phosphorus coatings. The relationship between hardness and annealing time is shown in Figure A.2. The data in Figure A.2 indicate that equivalent coating hardness can be obtained by lowering the temperature and extending the annealing time.

NOTE The Knoop indenter is generally preferred for measuring the hardness of metallic coatings because the uncertainty in the measurement is reportedly less with Knoop than with Vickers. The greater variability may be due to a tendency for thin, brittle coatings to crack during Vickers hardness testing. If measured properly, the numerical values are approximately the same. However, to make proper measurements would involve using different loads to obtain the same depth of indentation. To establish a valid correlation, side-by-side measurements would have to be made on identical coatings using different loads. As far as is known, that type of study has not been performed with autocatalytic nickel-phosphorus alloy coatings.



| Phase | Alloy type | Phosphorus<br>% by mass |
|---|---|---|
| β | 2, 3 | 1 to 4 |
| β + γ | 4 | 5 to 9 |
| γ | 5 | > 10 |

Figure A.1 — Relationship between heat treatment temperature and hardness after 1 h's heating for different types of autocatalytic nikel deposits

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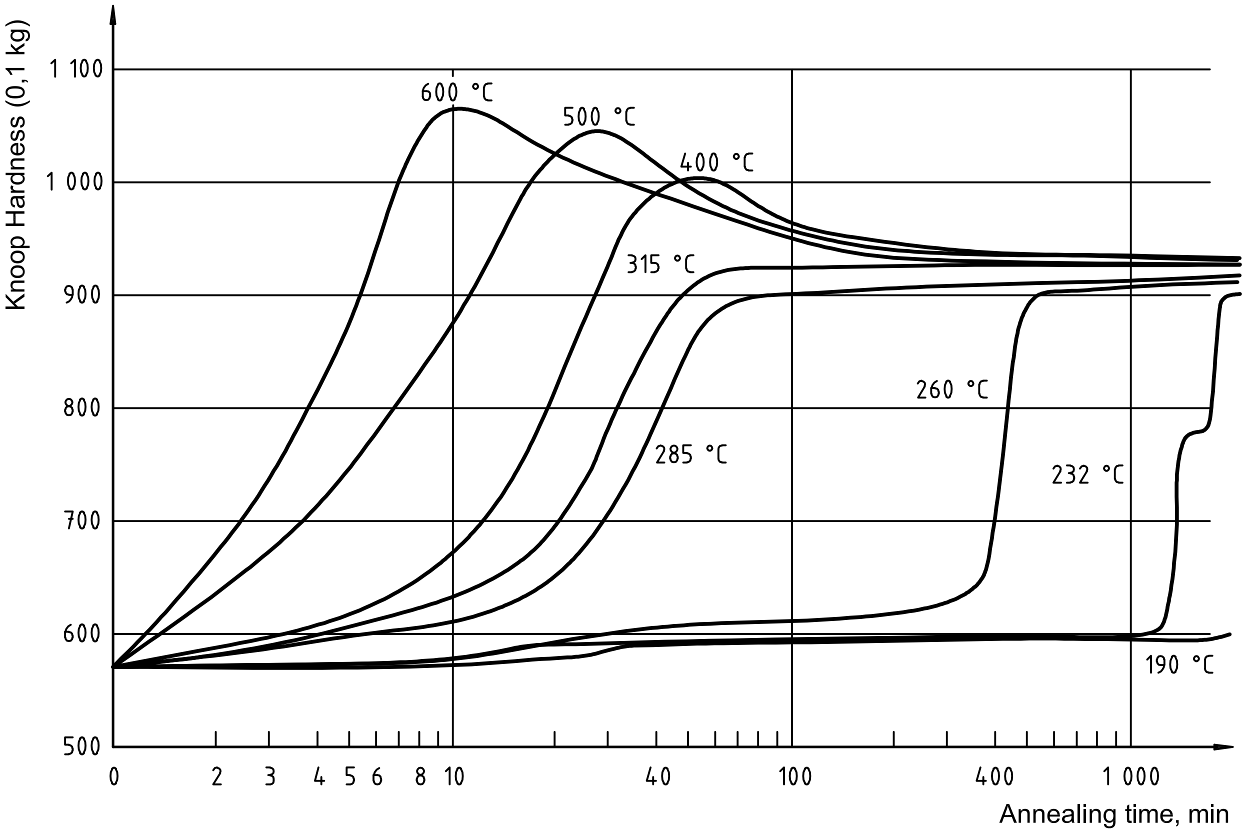

NOTE By changing the annealing time and lowering the temperature, equivalent deposit hardness can be achieved. The curves are based averages and the results may vary with deviations from standard practice.

**Figure A.2 — Relationship between hardness and annealing time**

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# Annex B
(informative)

# Thickness test methods

## B.1 General

ISO 3882 reviews methods of measuring thickness of metallic and other inorganic coatings, including methods that are not cited below.

## B.2 Destructive

### B.2.1 Microscopical method

Use the method specified in ISO 1463.

### B.2.2 Coulometric method

The coulometric method specified in ISO 2177 may be used to measure the total thickness of the autocatalytic nickel thickness and the thickness of copper and nickel underlayers, when present, at any point on the significant surface than 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 the thickness of the autocatalytic nickel and the thickness of underlayers.

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

## B.3 Non-destructive

### B.3.1 Beta backscatter method (applicable only in the absence of copper undercoats)

Use the method specified in ISO 3543. This method is suitable for measuring coatings on aluminium substrates and determines the total coating thickness.

### B.3.2 X-Ray spectrometry

Use the method specified in ISO 3497. X-ray apparatus should be calibrated with thickness standards whose coatings contain known quantities of phosphorus that are similar to or encompass the phosphorus content of the coatings being tested.

NOTE Due to possible local variation in the phosphorus content of the coating, integral methods for phosphorus determination covering a sector of the referenced area are recommended and are frequently utilized.

### B.3.3 Weigh-plate-weigh method

Using a part of known surface area (or a special test specimen with a similar substrate material of known surface area), weigh the part or test coupon to the nearest milligram before and after coating. Ensure that the part or coupon is dry and at room temperature for each measurement. Calculate the thickness from the increase in weight, the coating density, and area as follows:

$$T = \frac{10W}{\left(A \times D\right)}$$

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where

$T$ is the coating thickness, in micrometres;

$W$ is the weight gain, in milligrams;

$A$ is the total surface area, in square centimetres;

$D$ is the density, in grams per cubic centimetre.

The density of the coating depends on the phosphorus content of the deposit. The density of autocatalytic nickel coatings is given in Figure B.1, based on values reported in the technical literature.

EXAMPLE A coupon of mild steel has a weight of 3 198 mg with an area of 19,736 $cm^2$ before plating. After plating, the coupon weighs 3 583 mg. The phosphorus content of the coating is 9 % and the density is 8,01 $g/cm^3$ (from Figure B.1). The thickness is calculated as follows:

$$T = \frac{10(3\,583 - 3\,198)}{19{,}763 \times 8{,}01} = 24{,}3\,\mu\text{m}$$

### B.3.4 Magnetic method

Use the method described in ISO 2178 to measure the thickness of autocatalytic nickel-phosphorus alloy coatings containing greater than 8 % mass fraction phosphorus that are sufficiently non-magnetic to be measured by this method.

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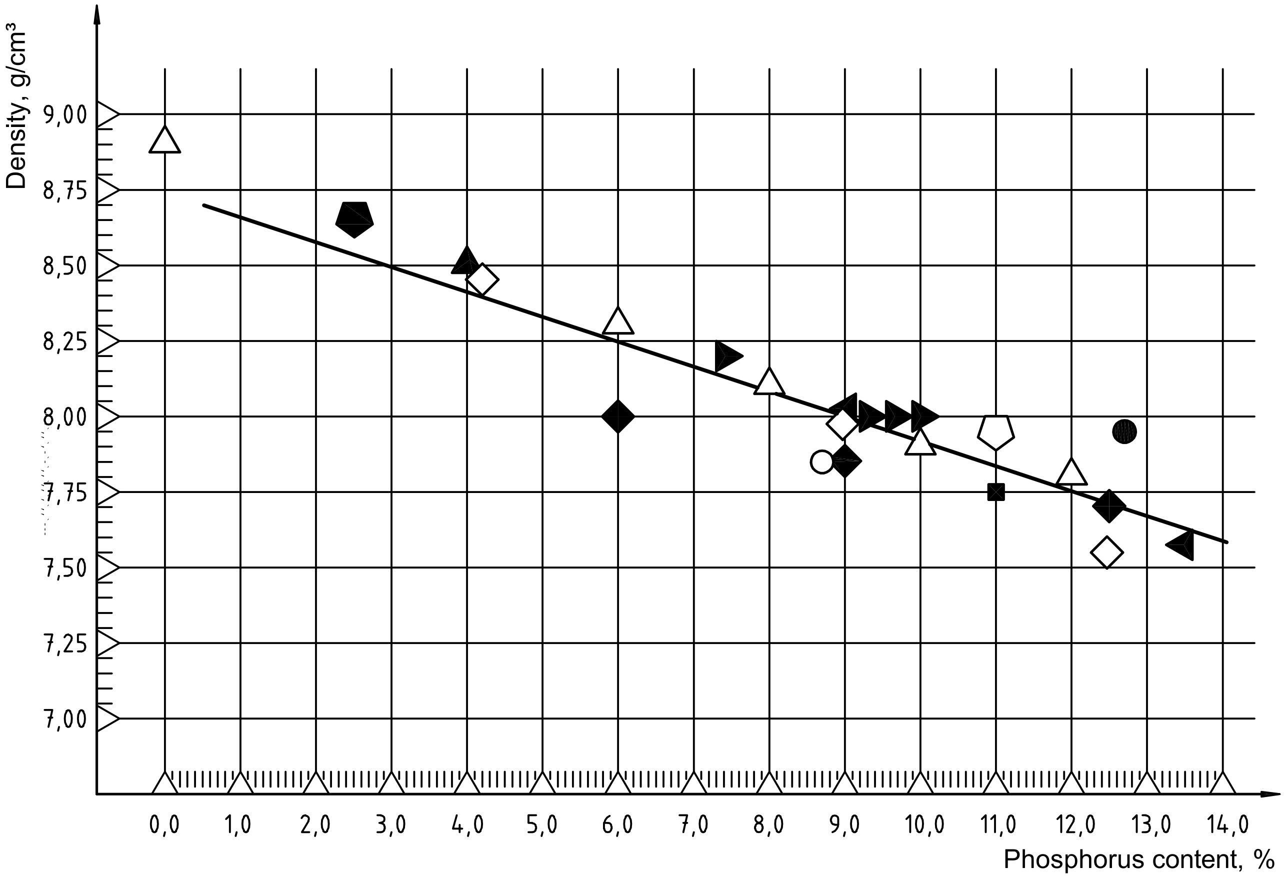

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**Figure B.1 — Density of autocatalytic (electroless) nickel-phosphorus alloy coatings**

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# Annex C

(informative)

# Guidance on thickness, composition and use of autocatalytic nickel-phosphorus coatings

## C.1 General

The properties of autocatalytic nickel-phosphorus alloy coatings depend primarily on the composition and structure of the coating. The composition and structure of the coating are in turn determined by the composition of the plating solution, the conditions of deposition and subsequent heat treatment that alters the structure of the deposit. The nature of the substrate, such as surface roughness, may also affect the properties of the coating, including its corrosion resistance.

## C.2 Corrosion resistance, coating thickness and service conditions

The minimum coating thickness required for adequate corrosion resistance in different service conditions is given in Table C.1. Coatings should be thicker on rough or porous surfaces in order to minimize the influence of the substrate on the deposit's properties. To obtain optimum corrosion resistance with the minimum thickness of coating, the surface of the substrate should be smooth and pore-free. A substrate surface roughness of approximately $Ra < 0,2$ µm may be used as a guide.

**Table C.1 — Minimum coating thickness required for corrosion resistance in service**

| **Service condition number** | **Description** | **Minimum coating thickness on ferrous base** µm | **Minimum coating thickness on aluminium base** µm |
|---|---|---|---|
| 5<br>(Exceptionally severe) | Service outdoors where frequent wetting and abrasion are factors; e.g., oil field applications. | 125 | – |
| 4<br>(Very severe) | Outdoor marine and other aggressive environments, severe abrasion, exposure to acid solutions, elevated temperature and pressure. | 75 | – |
| 3<br>(Severe) | Non-marine service outdoors where frequent wetting due to rain and dew, moderate abrasion, exposure to alkali salts at elevated temperatures are factors. | 25 | 60 |
| 2<br>(Moderate) | Service indoors where condensation can occur; indoor industrial exposures in dry or oiled environments. | 13 | 25 |
| 1<br>(Mild) | Service indoors in warm dry atmosphere; for soldering and mild abrasive wear. | 5 | 13 |
| 0<br>(Very mild) | Highly specialized electronic and semiconductor applications, thin film resistors, capacitors, inductors and diffusion welding. | 0,1 | 0,1 |

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In addition to thickness, the phosphorus content of the coating and other factors influence the corrosion performance of autocatalytic nickel-phosphorus coatings. In general, the corrosion resistance of the coatings in acidic environments improves as the phosphorus content is increased. The excellent corrosion resistance of these coatings is due to a phosphorus-rich, passive oxide film usually present on the surface. Impurities that are co-deposited with the alloy, however, can undermine the passive oxide film and, consequently, reduce corrosion resistance.

## C.3 Type and phosphorus content of coatings for different applications

The autocatalytic nickel deposition process can be controlled and adapted to yield coatings with properties that meet the requirements of different applications, thus affording engineers the opportunity to specify the properties required for a specific end-use. Table C.2 describes the types and phosphorus contents of coatings that are commonly specified for various applications.

**Table C.2 — Types of autocatalytic nickel coatings recommended for different applications**

| Type | Phosphorus, mass fraction % | Applications |
|---|---|---|
| 1 | No specified requirement for phosphorus content | General purpose coating |
| 2 (low-phosphorus) | 1 to 3 | Electrical conductivity, soldering, wire bonding |
| 3 (low phosphorus) | 2 to 4 | Adhesive and abrasive wear requiring high as-deposited hardness |
| 4 (medium phosphorus) | 5 to 9 | General purpose wear and corrosion resistance |
| 5 (high phosphorus) | > 10 | High as-deposited corrosion resistance, non-magnetic, diffusion weldable, flexible with high elongation; e.g., deposits containing 12,5 % mass fraction phosphorus as underlayers on hard discs. |

## C.4 Reclaiming worn or over-machined articles

Autocatalytic nickel coatings equal to or greater than 125 µm can be deposited to repair worn articles or to salvage ones that have been over-machined. The likelihood of nodule formation, staining, pitting and surface roughness increases with thickness, and the level of acceptance relative to those defects shall be agreed between purchaser and supplier. Because of lower internal stress, higher ductility and enhanced corrosion resistance, a coating with 10 % mass fraction or more phosphorus may be more suitable for reclaiming worn or over-machined articles than a coating with low or medium amounts. Heat treatment to improve adhesion may be performed (see Annex A).

Electrodeposited nickel undercoats are sometimes applied prior to autocatalytic deposition when coating thickness exceeds 125 µm. The electrodeposited nickel may have to be machined to size before applying the autocatalytic nickel deposit.

## C.5 Imparting solderability to metals that are difficult to solder

Autocatalytic nickel coatings are used to improve the solderability of aluminium and other alloys that are difficult to solder, the coating thickness being greater than 2,5 µm. A mildly active rosin flux is normally required for soldering.

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## C.6 Additional information for different applications

Autocatalytic nickel coatings of similar properties are not suitable for applications where adhesive wear is likely to occur, unless the coated surfaces are lubricated.

Medium- and low-phosphorus coatings are not recommended in applications where flexing or resistance to shock is required. Special care should be taken when welding items that have been coated. Welds made on coated areas may be embrittled by the diffusion of phosphorous from the coating. The coatings have a low hot hardness and are not suitable for use in cases where both wear and elevated temperatures are involved.

Some steels containing chromium and molybdenum may become passivated by anodic cleaning if a periodic reverse-current technique is used. For steels with a tensile strength below 1 000 MPa, cathodic cleaning in place of periodic reverse-current cleaning may be used.

Most metals have oxides on their surfaces that may affect the adhesion of the coating to the base metal. Special cleaning and activation procedures exist for many metals including stainless steels and aluminium. The presence of oxide films can cause adhesion failure, therefore surfaces will require removal of oxide films and any micro-constituents that may interfere with the formation of a continuous coating on the surface.

For cast iron and aluminium alloys, the presence of pores on the surfaces may cause corrosion problems due to entrapment of solutions in the pores and/or absence of continuity of the coating. The coating of cast surfaces having excessive porosity, therefore, requires special treatments for achieving the desired service life of the article.

In leaded copper alloys, lead present on the surface can contaminate the coating solution and can cause adhesion failures and porosity in the coating. Hence, special treatments should be used to cover or remove lead, prior to autocatalytic nickel deposition.

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# Annex D
(normative)

# Methods for chemical analysis of autocatalytic nickel coatings for phosphorus content

## D.1 Inductively coupled plasma (ICP) method

### D.1.1 General

Method of analysis by either emission or absorption spectra produced by inductively coupled plasma (ICP).

### D.1.2 Reagents

Analytical grade chemicals and distilled or deionized water shall be used for preparing the following test solutions:

a) nitric acid ($HNO_3$), 40 % volume fraction;

b) sodium nitrite ($NaNO_2$), 20 g/l solution;

c) potassium permanganate ($KMnO_4$), 7,6 g/l solution.

The nitric acid, 40 % volume fraction test solution is prepared by mixing 2 parts by volume of nitric acid having a specific gravity of approximately 1,42 g/ml with 3 parts by volume of water.

### D.1.3 Procedure

Carefully weigh approximately 0,2 g of the test specimen and dissolve in 50 ml of the nitric acid solution in a glass beaker. Heat gently in a fume cupboard until the specimen material is dissolved, then boil until emission of brown fumes ceases. Dilute the solution to approximately 100 ml, bring to the boil and add 25 ml of the potassium permanganate solution. Boil for 5 min and then add the sodium nitrite solution drop by drop until the precipitated manganese dioxide is dissolved. Boil the solution for 5 min and allow it to cool to ambient temperature. Transfer to a 250 ml standard flask and dilute to the mark with distilled or deionized water. Stopper the flask and shake well.

### D.1.4 Blank determination

Carry out a blank determination by preparing a blank solution as above, omitting only the test material.

### D.1.5 Spectral analysis

Carry out the determination in accordance with the operating instructions for the ICP equipment. The following spectral lines have been found to have low interference when using argon ICP techniques:

Ni 216,10 nm

P 215,40 nm

P 213,62 nm

Al 202,55 nm

Cd 214,44 nm

Co 238,34 nm

Cr 284,32 nm

Cu 324,75 nm

Fe 238,20 nm

Pb 283,30 nm

Sn 198,84 nm

Zn 206,20 nm

NOTE The inductively coupled plasma method is capable of determining the phosphorus content of the autocatalytic nickel coating to within 0,5 %.

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## D.2 Molecular absorption spectrometric method

### D.2.1 General principle

This annex specifies a molecular absorption spectrometric method for the determination of the phosphorus content of autocatalytic nickel-phosphorus coatings. A portion of the sample is dissolved in nitric acid. The solution is treated with potassium permanganate to precipitate manganese dioxide which is dissolved by adding sodium nitrite. Ammonium molybdate and ammonium vanadate are added and the absorbance of the solution is measured spectrometrically at a wavelength of 420 nm.

### D.2.2 Reagents

#### D.2.2.1 General

Analytical-grade reagents and distilled water or water of equivalent grade shall be used throughout.

#### D.2.2.2 Reagents for dissolution and oxidation

a) Nitric acid, 40 % volume fraction solution, prepared by mixing 2 parts by volume of nitric acid having a specific gravity of approximately 1,42 g/ml with 3 parts by volume of water;

b) Sodium nitrite, 20 g/l;

c) Potassium permanganate, 7,6 g/l.

#### D.2.2.3 Molybdate–vanadate solution

Dissolve separately in hot water 20 g of ammonium molybdate and 1 g of ammonium vanadate. Mix the two solutions, add 200 ml of nitric acid (specific gravity approximately 1,42 g/ml), and dilute to 1 l with water. Mix well.

#### D.2.2.4 Phosphorus standard solution (100 mg/l of P)

Weigh 0,439 2 g of potassium dihydrogen orthophosphate $(KH_2)PO_4$, dissolve it in water and transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix well.

1 ml of this solution contains 0,1 mg of P.

### D.2.3 Apparatus

#### D.2.3.1 Ordinary laboratory apparatus

**D.2.3.2 Spectrophotometer or photoelectric absorptiometer,** fitted with a filter providing maximum transmission at a wavelength of 420 nm, equipped with optical cells of path length 10 mm.

### D.2.4 Procedure

#### D.2.4.1 Preparation of the test solution

a) Weigh, to the nearest 0,1 mg, 0,19 g to 0,21 g of the test specimen. Transfer it to a beaker and dissolve it in 50 ml of nitric acid solution [D.2.2.2 a)].

b) Heat gently until the test specimen is completely dissolved. Then boil to remove brown fumes.

c) Dilute the solution to approximately 100 ml, bring to the boil and add 25 ml of the potassium permanganate solution [D.2.2.2 c)].

d) Boil the solution for 5 min.

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e) Add the sodium nitrite solution [D.2.2.2 b)] drop by drop until the precipitated manganese dioxide is dissolved.

f) Boil the solution for 5 min and then allow it to cool to ambient temperature.

g) Transfer the solution to a 250 ml one-mark volumetric flask, dilute to the mark with water and mix well.

### D.2.4.2 Blank test

Carry out a blank test in parallel with the determination, by the same procedure, using the same quantities of all reagents as in the determination, but omitting the test specimen.

### D.2.4.3 Preparation of the calibration graph

Into a series of 100 ml one-mark volumetric flasks, introduce the volumes of the phosphorus standard solution (D.2.2.4) shown in Table D.1.

**Table D.1 — Preparation of phosphorus calibration solutions**

| Volume of phosphorus standard solution ml | Corresponding mass of phosphorus (P) mg |
|---|---|
| 0 | 0 |
| 2 | 0,2 |
| 4 | 0,4 |
| 6 | 0,6 |
| 8 | 0,8 |
| 10 | 1,0 |

Treat the contents of each flask as follows:

a) Add 25 ml of the molybdate-vanadate reagent (D.2.2.3), add water to the mark and let the solution stand for 5 min. Fill one of the optical cells with the solution.

b) Carry out the spectrometric measurement using either the spectrophotometer at the wavelength of maximum absorption (about 420 nm) or the photoelectric absorptiometer fitted with suitable filters after having, in each case, adjusted the apparatus to zero absorption against water. Deduct the absorption of the calibration compensation solution from those of the other calibration solutions.

c) Plot a graph with the phosphorus mass, in milligrams, contained in the calibration solution on the abscissa versus the corresponding value of the absorbance on the ordinate.

## D.2.5 Dose

a) Transfer 10 ml of the test solution to a 100 ml one-mark volumetric flask, add 50 ml of water, 25 ml of the molybdate-vanadate solution, add water to the mark, and mix well. Allow the solution to stand for 5 min. Fill one of the optical cells with the solution. For purposes of comparison, also test a control sample that does not contain the molybdate-vanadate solution.

b) Carry out the spectrometric measurement as described in D.2.4.3 b).

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### D.2.6 Expression of results

By means of the calibration graph, determine the mass of phosphorus corresponding to the spectrometric measurement.

The phosphorus content, as a percentage by mass, is given by the following formula:

$$\text{Phosphorus content} = \frac{2{,}5\left(m_3 - m_4\right)}{m}$$

where

$m_3$ is the mass of phosphorus, in milligrams, found in the aliquot portion of the test solution used for the determination;

$m_4$ is the mass of phosphorus, in milligrams, found in the corresponding aliquot portion of the blank test solution;

$m$ is the mass, in grams, of the test portion.

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NOTE European Standards (EN) and CEN Reports (CR) are available from the European Committee for Standardization (CEN), rue de Stassart 36, B-1050, Brussels, Belgium. ASTM Standards are available from ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA, 19428-2959, USA.

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