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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*

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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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# 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<sup>1)</sup>, *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)

## ISO 4527:2003(E)

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<sup>2)</sup>, *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* <https://standards.iteh.ai/catalog/standards/sist/00a915ee-443a-44ef-9306-213f7aad186f/iso-4527-2003>

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)



## 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 solderability 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.

## 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  $\mu\text{m}$ , 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]**

## 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  $\mu\text{m}$ .

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  $\mu\text{m}$  (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  $\mu\text{m}$  (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  $\mu\text{m}$  applied to G43400 steel, requiring stress relief prior to coating at 210 °C for 22 h, and subsequently electroplated with chromium, 0,5  $\mu\text{m}$  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)].

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  $\pm 10\%$  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.