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Electrodeposited coatings and related finishes — Electroless Ni-P-ceramic composite coatings

Dépôts électrolytiques et finitions apparentées — Revêtements composites céramiques-Ni-P sans courant

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 3, *Electrodeposited coatings and related finishes*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Electroless nickel-phosphorus (Ni-P) alloy coatings are produced by the controlled chemical reduction of nickel ions onto a catalytic surface in hot, usually mildly acidic or occasionally alkaline solutions using hypophosphite ion as the reducing agent. Because the deposited nickel alloy is a catalyst for the reaction, the process is self-sustaining or autocatalytic.

Fine ceramic (nano-sized) particles dispersed in the electroless nickel plating bath can be incorporated with the deposition of a Ni-P layer, which produces a nickel-phosphorus-ceramic (Ni-P-ceramic) composite coating. There is no molecular bonding between the Ni-P matrix and the incorporated ceramic particles. The incorporation phenomenon basically results from the impact and settling of the particles on the surface of the work piece and the subsequent surrounding of these particles by the growing Ni-P matrix. The deposits produced are uniform in thickness on irregularly shaped articles as the processing solution circulates freely over their surfaces. The physicochemical properties and the structure of electroless Ni-P-ceramic composite coatings are dependent on the size, type, chemical inertness and deposition homogeneity of the ceramic particles as well as plating conditions such as pH, temperature, plating bath composition, surfactants used, quality of the substrates and their pre-treatment, activation methods and post-heat treatment.

Electroless Ni-P-ceramic composite is applied to improve hardness as well as wear, and corrosion-resistant properties of the coating depending on the nature of the ceramics.

Electrodeposited coatings and related finishes — Electroless Ni-P-ceramic composite coatings

1 Scope

This document specifies the requirements and recommendations for electroless Ni-P-ceramic composite coatings applied from aqueous solutions onto metallic and non-metallic substrates.

This document does not apply to ternary nickel alloys and nickel-boron ceramic composite coatings.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 2080, *Metallic and other inorganic coatings — Surface treatment, metallic and other inorganic coatings — Vocabulary*

ISO 2081, *Metallic and other inorganic coatings — Electroplated coatings of zinc with supplementary treatments on iron or steel*

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*

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 thickness — Beta backscatter method*

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, *Metallic coatings — Electroplated coatings of nickel for engineering purposes*

ISO 4527, *Metallic coatings — Autocatalytic (electroless) nickel-phosphorus alloy coatings — Specification and test methods*

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

ISO 9587, *Metallic and other inorganic coatings — Pretreatment 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 27307, *Thermal spraying — Evaluation of adhesion/cohesion of thermal sprayed ceramic coatings by transverse scratch testing*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 2064, ISO 2080, ISO 4527, ISO 9587 and ISO 9588 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Designation

4.1 General

The designation shall comprise the following:

a) the term, “electroless Ni-P-ceramic composite coating”;

b) the number of this document, i.e. ISO 23363;

c) a hyphen;

d) the chemical symbol of the basis metal;

e) a solidus (/);

f) symbols for the nickel-phosphorus and ceramics as well as coatings applied prior to and after electroless coating, 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 µm.

4.2 Examples of designations

The following are examples of designations.

a) 20 µm thick electroless nickel-phosphorus silicon carbide composite coating having a nominal phosphorus content of 8 % mass fraction, and 25 volume % of SiC in average size of 300 nm on 10800 copper-base alloy is designated as follows:

Electroless Ni-P-ceramic composite coating ISO 23363-Cu<10800>/NiP(8)20/SiC300(25)/

b) The same coating applied to G43400 steel requiring stress relief prior to coating at 210 °C for 22 h and subsequently electroplated with 0,5 µm chromium, requiring heat treatment for hydrogen embrittlement relief at 210 °C for 22 h, is designated as follows:

Electroless Ni-P-ceramic composite coating ISO 23363-Fe<G43400>[SR(210)22]//NiP(8)20/ SiC 300(25)// Cr0,5[ER(210)22]

5 Requirements

5.1 Special test specimens

Special test specimens may be used to measure adhesion, thickness, porosity, corrosion resistance, hardness and other properties of Ni-P-ceramic composite coatings. 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 required.

5.2 Surface finish

The incorporation of ceramic particles into the deposit can increase the surface roughness of the coating, especially in the case of hard and sub-micron-sized ceramic particles. If a specified final surface roughness is required, the method of measurement shall be as specified in ISO 4288.

NOTE The surface finish of electroless nickel composite coatings is not usually superior to that of the substrate before coating.

5.3 Thickness

The coating thickness shall be measured using one of the test methods given in ISO 2178, ISO 1463, ISO 3497, ISO 3543, ISO 4527 and ISO 9220 unless otherwise specified by the purchaser.

5.4 Hardness

In addition to the heat treatment, the hardness of the Ni-P-ceramic composite coatings shall be different to the electroless Ni-P coatings depending on the type, size, nature and the homogeneity of the dispersed ceramic particles into the deposit, in accordance with Annex A. 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.

5.5 Adhesion

Prior to the electroless nickel composite coating, an electro/electroless nickel or other metallic undercoat is recommended to improve adhesion to the substrate. Coatings shall be capable of passing one or more of the adhesion tests given in ISO 2819 and ISO 27307 or as specified by the purchaser.

5.6 Porosity

If required, a maximum degree of porosity of the electroless Ni-P-ceramic composite coating shall be specified by the purchaser together with a method of testing for porosity.

5.7 Corrosion resistance

Corrosion resistance can be affected by the nature, types and the amount (volume %) of incorporated ceramic particles into the Ni-P matrix (see Annex A). Similarly, surface conditions such as surface smoothness, hydrogen embrittlement, porosity and the interface between the ceramics and nickel matrix as well as phosphorus content in the deposit can affect the corrosion behaviour of the coating. The corrosion resistance and the corrosion test method shall be specified by the purchaser. If required,

the acceptance criteria for corrosion protection shall be specified by the purchaser 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.

5.8 Stress relief heat treatment prior to coating

When specified by the purchaser, 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.

5.9 Hydrogen embrittlement relief heat treatment after coating

Similar to the Ni-P coating, Ni-P-ceramic composite coatings on 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 in accordance with the procedures and classes given in ISO 9588 and ISO 2081 or as specified by the purchaser.

5.10 Heat treatment to harden the coating

When required, heat treatment shall be performed within 1 h of coating to increase the hardness of the electroless Ni-P-ceramic composite coatings. 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.

5.11 Heat treatment to improve adhesion

Unless the purchaser specifies another procedure, heat treatment to improve the adhesion shall be carried out in accordance with ISO 4527, if required.

5.12 Coefficient of friction and wear resistance

Coefficient of friction and wear of Ni-P-ceramic composite coatings shall be measured using the method as specified by the purchaser to ensure that the requirement has been met.

NOTE Coefficient of friction and wear can be affected by the amount and types of incorporated ceramic particles (see Annexes A and B) as well as heat treatment on electroless nickel composite coatings.

5.13 Chemical composition

The phosphorus content of the Ni-P-ceramic composite coating shall be as specified in ISO 4527. Similarly, the ceramics content in the coating shall be measured by one of the methods given in Annex C, unless specified by the purchaser.

5.14 Undercoats

If required, electro or electroless nickel undercoats of required thickness shall be carried out in accordance with ISO 4526 and ISO 4527, respectively.

6 Sampling

The methods 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.

Annex A
(normative)

General classification of ceramic particles for improving hardness, wear and corrosion properties of composite coatings

Table A.1 — General classification and application of common ceramic particles[a]

Types	Hardness	Coefficient of friction/wear	Corrosion	Remarks
Oxides	Al_2O_3, Cr_2O_3, TiO_2, ZrO_2-Y_2O_3, SiO_2, CeO_2	Al_2O_3, Cr_2O_3, TiO_2, ZrO_2-Y_2O_3, SiO_2, CeO_2	Al_2O_3, Cr_2O_3, TiO_2, ZrO_2-Y_2O_3, SiO_2, CeO_2	Consideration of the plating bath reactivity
Carbides	SiC, WC, TiC, B_4C, Cr_3C, Carbide cermets, TaC, ZrC	SiC, WC, TiC, B_4C, Cr_3C, Carbide cermets, TaC, ZrC	SiC, B_4C, Cr_3C, TaC, Carbide cermets,	
Nitrides	C-BN, Si_3N_4, AlN, TiN	h-BN	C-BN, h-BN, Si_3N_4, AlN, TiN	
Borides	TiB_2, ZrB_2, TaB_2, HfB_2	TiB_2, ZrB_2, TaB_2, HfB_2	ZrB_2, HfB_2	
Sulfides	—	WS_2, MoS_2	WS_2, MoS_2	
Others	Diamond	CNT, $SnSe_2$, Graphite, Graphene, RGO	Diamond, CNT, Graphene, Graphite	

[a] Different morphologies and particle size of ceramics shall be considered. A successful co-deposition of ceramic particles is dependent on various factors including catalytic inertness, particles charge, bath reactivity, particles size distribution (10 nm to 1 000 nm on average) and compatibility of the particles with the metallic matrix. If required, ceramic particles shall be pre-treated before being added into the plating bath.

Annex B
(informative)

Bath formulation and operating conditions for electroless Ni-P-ceramic composite coatings

Table B.1 — Typical bath composition and operating conditions for electroless Ni-P-ceramic composite coatings

Bath composition	Acidic Ni-P bath	Alkaline Ni-P bath
Nickel source	Nickel sulfate, nickel sulfamate, nickel chloride, nickel carbonate, nickel acetate	Nickel sulfate, nickel chloride
Reducing agent	Sodium hypophosphite	Sodium hypophosphite
Complexing agents (single or combination)	Carboxylic acids, hydroxy carboxylic acids, aminoacetic acids, amines	Salts of carboxylic acids, hydroxy carboxylic acids, pyrophosphate ions, amines, alkanolamines
Stabilizers	Bismuth ions, thiourea and/or other thio-organic compounds	Thiourea and other thio-organic compounds
Wetting agents	Non-ionic and cationic surfactants	Non-ionic and cationic surfactants
pH (shall be adjusted by caustic soda or H_2SO_4)	Approximately 3,8 to 6,5	Approximately 8 to 11
Temperature (° C)	Approximately 75 to 90	Approximately 30 to 90
Amount of ceramics loading (g/l)[a]	Approximately 1 to 10	Approximately 1 to 10

[a] The dispersion of fine ceramic particles significantly increases the surface area loading that shall lead to the decomposition of plating solution. Solution agitation is recommended to avoid particles agglomeration and sedimentation. In addition, the plating specimen shall also be rotated to make a homogeneous distribution of co-deposited ceramic particles into the deposit that shall also affect the coefficient of friction, wear and corrosion properties of the coatings.