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Metallic and other inorganic coatings — Electrodeposited nickel-ceramics composite coatings

Revêtements métalliques et autres revêtements inorganiques — Dépôts électrolytiques composites de nickel-céramique

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Foreword

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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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The committee responsible for this document is ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 3, *Electrodeposited coatings and related finishes*.

Introduction

Electrolytic codeposition of ceramic particles into the metal matrix is a low-cost and low-temperature method suitable for producing composite coating. Ceramics particles can be hard oxide or carbides like Al_2O_3, SiC, Si_3N_4, TiO_2, WC or diamond. Also, it may include solid lubricants like PTFE, graphite, h-BN or MoS_2. Incorporation of such particles can improve mechanical, tribological and anti-corrosion properties of electrodeposited coatings. Nickel being an engineering material, nickel-ceramics composite coating can find wide application such as cutting tools for semiconductors, heat exchangers, automobile shackle pin, glass moulds. The applications cover machine elements, tools, electronics, micro-system technology, electrochemistry, acoustics, decoration and many more. In addition, electrodeposited nickel-ceramics composite coating can be applied in automotive parts including cylinder bores, piston and piston rings to increase engine performance. Nickel-based composites exist as many different types depending upon kind of ceramic particles incorporated, which can differ significantly in their properties. To increase hardness and corrosion resistance, hard ceramic particles such as SiC, Si_3N_4 and Al_2O_3 can be used, whereas to increase wear resistance property, mainly self-lubricating solid particles such as h-BN, PTFE or MoS_2 can be used. It is therefore important to choose the right ceramic particles for a given application. Typical solution compositions, operating conditions and mechanical properties of electrodeposits from Watts and nickel sulphamate solutions are given in Annex B.

The objective of this document is to provide processing and requirements that cover all nickel-based composite system. This enables both providers as well as users to easily fabricate the composite system.

Metallic and other inorganic coatings — Electrodeposited nickel-ceramics composite coatings

1 Scope

This document specifies the requirements for electrodeposited nickel-ceramics composite coatings applied to ferrous and non-ferrous basis metals for engineering purposes. In addition, it enables both providers and users to offer a consistent methodology to fabricate electrodeposited nickel-ceramics composite system.

Nickel alloys or composites in which nickel content is less than 40 vol.% are outside the scope of this document.

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

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, *Metal and oxide coatings — Measurement of coating thickness — Microscopical method*

ISO 2177, *Metallic coatings — Measurement of coating thickness — Coulometric measurement 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 inorganic coatings — Review of methods of measurement of thickness*

ISO 4519, *Electrodeposited metallic coatings and related finishes — Sampling procedures for inspection by attributes*

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

ISO 14577 (all parts), *Metallic materials — Instrumented indentation test for hardness and materials parameters*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 2064, ISO 2080, ISO 4287 and ISO 4526 apply.

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

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

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

4 Designation

The designation shall comprise the following:

a) the term, electrodeposited composite coating;

b) the number of this document, ISO 19487;

c) a hyphen;

d) a solidus (/);

e) symbols for the nickel 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 µm.

For example, electrodeposited composite coating ISO 19487-Ni30/SiC(25) designates nickel silicon carbide composite 30 µm thick which contains 25 vol.% of SiC.

5 Requirements

5.1 Special test specimens

Special test specimens may be used to measure adhesion, thickness, porosity, corrosion, hardness and other properties when the coated articles are of a size or shape that is 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, 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 document 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.

5.2 Appearance and surface roughness

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.

Imperfections in the coating that arise from surface conditions of the basis metal and that 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.

Coatings that are to be finished by machining may have slight surface blemishes in the as-deposited condition, provided that these will be eliminated by the machining operation. For articles that are electroplated and subsequently ground to size, the grinding operation shall be performed with a sulphur-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.

For ground finishes, 0,4 µm R_a is termed a commercial finish, and 0,2 µm R_a is termed a good commercial finish.

5.3 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.

The minimum local thickness of coating shall be measured by one of the methods given in Annex A. The minimum local thickness is typically 5 µm to 200 µm, depending on the specific engineering application.

Although there is no technical limitation to the thickness of coating 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 coating thickness, especially as nickel thickness is increased. The electroplating operation may have to be interrupted at intermediate stages of processing 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 re-activated to ensure adhesion of subsequent nickel deposits. Auxiliary anodes may have to be employed to achieve uniformity of deposit thickness.

5.4 Hardness

When hardness is specified, it shall be measured by the method given in ISO 14577 (all parts).

5.5 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.

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.

5.6 Porosity

Electroplated ferrous articles or special representative test specimens shall be subjected to the hot water porosity test, or the modified ferroxyl test. After testing, the articles shall be rated in accordance with ISO 10289. The part is considered to have failed if the number of pores per part or per unit area exceeds that specified by or agreed between the purchaser and the electroplater.

5.7 Stress relief heat treatments

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.

Steels with oxide or scale have to 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 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 (over 1 450 MPa).

5.8 Hydrogen embrittlement relief

Steel parts having an ultimate tensile strength equal to or greater than 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.

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. For example, ISO 10587 describes a method of testing threaded articles for residual hydrogen embrittlement.

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

Nickel electrodeposits that contain small amounts of sulphur discolour and become embrittled when heated above 200 °C. Therefore, special care is to be taken while choosing surfactants in order to minimize the sulphur codeposition in the nickel. The exact temperature of embrittlement depends on the sulphur content of the nickel, and the time at elevated temperature.

5.9 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 ISO 12686.

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 tensively stressed, and is recommended for parts that are subject to repeated applications of complex load patterns in service. Other factors that affect fatigue life include thickness 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.

5.10 Internal stress

The internal stress of electrodeposited nickel and nickel-ceramics composite coatings varies over wide ranges. In general, high tensile stresses in excess of 100 MPa may result in processing difficulties. Electrodeposits from Watts solutions usually have higher internal stresses than deposits from nickel sulphamate solutions. Organic additives that reduce internal stress are available, but must be used with caution because they increase the sulphur content of the electrodeposited coating. Nickel-ceramics composite coatings containing sulphur are likely to be embrittled when heated above 200 °C during processing or in service. Levelling agents tend to increase the internal stress of the deposit in the tensile direction.

5.11 Ductility

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

6 Sampling

The method of sampling shall be selected from the procedures specified in ISO 4519. The acceptance levels shall be specified by the purchaser.

Annex A
(normative)

Test methods for determining thickness

A.1 General

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

A.2 Destructive

A.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.

A.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.

A.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.

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

A.3 Non-destructive

A.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.

A.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 aluminium substrate. This method determines the total coating thickness, including that of a copper undercoat, if present.

A.3.3 X-Ray spectrometry

Use the method specified in ISO 3497.

Annex B
(informative)

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

Table B.1 — Typical composition and operating conditions of Watts and nickel sulphamate solutions

	Watts nickel	Nickel sulphamate
Electrolyte composition, g/l		
Nickel sulphate, $NiSO_4 \cdot 6H_2O$	120 to 400	
Nickel sulphamate, Ni $(SO_3NH_2)_2$		200 to 800
Nickel chloride, $NiCl_2 \cdot 6H_2O$	30 to 85	0 to 10
Boric acid, H_3BO_3	30 to 55	15 to 60
Ceramic particles	2-20	2-20
Ceramic particles size	20 nm to 5 µm	20 nm to 5 µm
Surfactants[a]	<0,3	<0,3
Temperature, °C	40 to 65	40 to 50
Agitation	Air or mechanical	Air or mechanical
Operating conditions		
Cathode current density, A/dm^2	3 to 8	0,5 to 30
Anodes	Nickel	Nickel
pH	3,5 to 4,5	4,0 to 5,0
Typical mechanical properties		
Tensile strength, MPa	345 to 500	415 to 700
Elongation, %	10 to 30	5 to 30
Vickers hardness, 100 g load	>350	>450
Internal stress, MPa	150 (tensile) to 100 (compressive)	0 to 70 (tensile)

[a] Surfactants can be of anionic or cationic types or combination of both. Surfactants can improve the codeposition of ceramic particles by modifying their zeta potential values and also improve suspension stability.

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