

Metallic coatings — Electrodeposited coatings of nickel

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee STI/33, Electrodeposited and related coatings, which has the responsibility to:

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**Metallic coatings — Electrodeposited
coatings of nickel**

Revêtements métalliques — Dépôts électrolytiques de nickel



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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 3.

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

International Standard ISO 1458 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 3, *Electrodeposited coatings and related finishes*.

This third edition cancels and replaces the second edition (ISO 1458:1988), which has been technically revised.

Annexes B, C and D form a normative part of this International Standard. Annex A is for information only.

Introduction

Decorative, electrodeposited nickel coatings with and without copper undercoats and without chromium top-coats are suitable for applications in which tarnishing is prevented by rubbing or handling in service or by the use of topcoats other than chromium. They are also suitable for those applications where tarnishing is of no importance. Corrosion resistance depends on the type and thickness of the coatings. In general, multilayer nickel coatings provide better protection than single-layer nickel coatings of equal thickness.

Metallic coatings — Electrodeposited coatings of nickel

1 Scope

This International Standard specifies requirements for nickel electrodeposited coatings that are applied to iron and steel, to zinc alloys, to copper and copper alloys, and to aluminium and aluminium alloys to provide an attractive appearance and corrosion resistance. In addition, it specifies requirements for copper plus nickel coatings applied to iron and steel, and to zinc alloys. Coating designations that differ in thickness and type are specified and guidance is given in selecting the coating appropriate to the service conditions to which the coated product will be exposed.

This International Standard does not specify the surface condition required by the basis metal prior to the coating process, and is not applicable to coatings on sheet strip or wire in the non-fabricated form nor to threaded fasteners or coil springs.

Requirements for nickel plus chromium and copper plus nickel plus chromium coatings for metallic substrates are given in ISO 1456. Requirements for similar coatings for plastic materials are given in ISO 4525. ISO 4526 and ISO 6158 specify requirements for coatings of nickel and chromium, respectively, for engineering purposes.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

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

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

ISO 2080, *Surface treatment, metallic and other inorganic coatings — Vocabulary*

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

ISO 2361, *Electrodeposited nickel coatings on magnetic and non-magnetic substrates — Measurement of coating thickness — Magnetic method*

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

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

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

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

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

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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 16348, *Metallic and other inorganic coatings — Definitions and conventions concerning appearance*

3 Terms and definitions

For the purposes of this International Standard, the definitions given in ISO 2064, ISO 2079, ISO 2080 and ISO 16348 apply.

4 Information to be supplied by the purchaser to the electroplater

4.1 Essential information

When ordering articles to be electroplated in accordance with this International Standard, the purchaser shall provide the following information in writing, in, e.g., the contract or purchase order, or on engineering drawings:

- a) the designation (see clause 6);
- b) the appearance required, e.g., bright, dull or satin (see 6.3 and 7.1). Alternatively, samples showing the required finish or range of finish shall be supplied or approved by the purchaser, and used for comparison purposes (see 7.1);
- c) an indication of the significant surfaces to be made on drawings of the parts, or by the provision of suitably marked specimens;
- d) the type of corrosion test to be used (see 7.5 and Table 6);
- e) the type of adhesion test to be used (see 7.4);
- f) the extent to which defects are able to be tolerated on non-significant surfaces (see 7.1);
- g) the positions on the significant surface for rack or contact marks, where such marks are unavoidable (see 7.1);
- h) sampling methods and acceptance levels (see clause 8);
- i) the tensile strength of the steel and any requirement for pre- or post-treatments of iron or steel to reduce the risk of hydrogen embrittlement, as well as hydrogen embrittlement test methods (see 7.8 and 7.9).

4.2 Additional information

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

- a) any requirements for STEP testing (see 7.6);
- b) thickness requirements on those areas that cannot be reached by a ball 20 mm in diameter (see 7.2);
- c) whether or not a copper undercoat is required [see 6.1 c) and 6.2].

5 Service condition number

The service condition number is used by the purchaser to specify the degree of protection required, as related to the severity of the conditions to which a product is to be subjected, in accordance with the following scale:

- 3 - severe
- 2 - moderate
- 1 - mild

Typical service conditions for which the various service condition numbers are appropriate are listed in annex A.

6 Designation

6.1 General

The coating designation is a means of specifying the basis metal and the types and thicknesses of coatings appropriate for each service condition (see Table 1 to Table 4 for various substrates) and comprises the following:

- a) the term, "Electroplated coating", the number of this International Standard, i.e. ISO 1458, followed by a hyphen;
- b) the chemical symbol for the basis metal (or for the principal metal if an alloy) followed by a stroke, as follows:
 - Fe/ for iron and steel;
 - Zn/ for zinc alloys;
 - Cu/ for copper and copper alloys;
 - Al/ for aluminium and aluminium alloys.
- c) the chemical symbol for copper (Cu), if copper or brass containing more than 50 % copper is used as an undercoat;
- d) a number indicating the minimum local thickness, in micrometres, of the copper coating where applicable;
- e) a letter indicating the type of copper, where applicable (see 6.2);
- f) the chemical symbol for nickel (Ni);
- g) a number indicating the minimum local thickness of the nickel coating in micrometres;
- h) a letter designating the type of nickel coating (see 6.3);
- i) if a topcoat is to be applied over the nickel, its chemical symbol and a number indicating its minimum local thickness shall be appended to the coating designation. If the topcoat is an electrodeposited alloy, the chemical symbols of the principal alloy constituents shall be used. If the topcoat is a precious metal, e.g., gold or silver, the chemical symbol shall be followed by a number in parenthesis giving the minimum content of the precious metal in the topcoat, expressed as mass percent to one decimal point.

See the examples of designations given in 6.4

Table 1 — Nickel and copper plus nickel coatings on steel

Service condition number	Partial designation
3	Fe/Ni30b Fe/Cu20a Ni25b Fe/Ni30p Fe/Cu20a Ni25p Fe/Ni30s Fe/Cu20a Ni25s Fe/Ni25d Fe/Cu20a Ni20d
2	Fe/Ni20b Fe/Cu15a Ni20b Fe/Ni20p Fe/Cu15a Ni20p Fe/Ni20s Fe/Cu15a Ni20s Fe/Ni15d Fe/Cu15a Ni15d
1	Fe/Ni10b Fe/Cu10a Ni10b Fe/Ni10s Fe/Cu10a Ni10s
NOTE An initial copper coating, 5 µm to 10 µm thick, is normally applied to iron and steel from a copper cyanide solution before electroplating with ductile acid copper to prevent immersion deposition and poorly adherent deposits. The initial copper coating (copper strike) may not be substituted for any portion of the ductile acid copper specified in Table 1.	

Table 2 — Nickel and copper plus nickel coatings on zinc alloys

Service condition number	Partial designation
3	Zn/Ni25b Zn/Cu15a Ni20b
	Zn/Ni25s Zn/Cu15a Ni20b
	Zn/Ni20d Zn/Cu15a Ni15d
2	Zn/Ni15b Zn/Cu10a Ni15b
	Zn/Ni15s Zn/Cu10a Ni15b
1	Zn/Ni10b Zn/Cu10a Ni10b
	Zn/Ni10s Zn/Cu10a Ni10b

NOTE Zinc alloys must first be electroplated with copper to ensure adhesion of the subsequent nickel coatings. The initial layer of copper is usually electrodeposited from a copper cyanide solution but cyanide-free alkaline copper solutions are also being used. The minimum thickness of the initial copper layer is 8 μm to 10 μm . For articles of complex shape, the minimum copper thickness may need to be increased to about 15 μm to ensure adequate coverage on low-current density areas outside the significant surfaces. Ductile, levelling copper electrodeposited from acid solutions is usually applied over the initial cyanide copper deposit when the specified copper thickness is greater than 10 μm .

Table 3 — Nickel coatings on copper and copper alloys

Service condition number	Partial designation
3	Cu/Ni20b Cu/Ni20p Cu/Ni20s Cu/Ni20d
	Cu/Ni10b Cu/Ni10s Cu/Ni10p
	Cu/Ni5b Cu/Ni5s

Table 4 — Nickel coatings on aluminium and aluminium alloys

Service condition number	Partial designation
3	Al/Ni30b
	Al/Ni30s
	Al/Ni30p
	Al/Ni25d
2	Al/Ni25b
	Al/Ni25s
	Al/Ni25p
	Al/Ni20d
1	Al/Ni10b

NOTE For aluminium and aluminium alloys, immersion deposits of zinc or tin, and electrodeposited copper and other undercoats are used as part of the preparation for electroplating to ensure adhesion, prior to application of the designated nickel coatings given in Table 4.

6.2 Type of copper coating

The type of copper is indicated by the following symbol:

- a for ductile, levelling copper electrodeposited from acid-type solutions.

6.3 Types of nickel coatings

The type of nickel coating shall be designated by the following symbols:

- b for nickel electrodeposited in the fully bright condition;
- p for dull or semi-bright nickel which has been mechanically polished;
- s for dull, semi-bright or satin nickel that shall not have been mechanically polished;
- d for double- or triple-layer coatings, the requirements for which are given in Table 5.

6.4 Example of a designation

A coating on steel comprising 20 μm (minimum) ductile, levelling copper (Cu20a) plus 30 μm (minimum) bright nickel (Ni30b) is designated as follows:

Electroplated coating ISO 1458 - Fe/Cu20a Ni30b

The above coating electroplated with 2 μm minimum gold having a minimum gold content of 98 % by mass [Au(98,0)2] is designated as follows:

Electroplated coating ISO 1458 - Fe/Cu20a Ni30b Au(98,0)2

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

7 Requirements

7.1 Appearance

Over the significant surface, there shall be no clearly visible plating defects such as blisters, pits, roughness, cracks, non-plated areas, stains or discolorations. The extent to which defects may occur on non-significant surfaces shall be specified by the purchaser. Where rack marks on the significant surface are unavoidable, their position on the surface shall be specified by the purchaser. The appearance shall be uniform and of an agreed colour and approved samples of artefacts shall be used for comparison purposes [see 4.1 b)].

7.2 Local thickness

The thickness of the coating specified in the designation shall be the minimum local thickness. The minimum local thickness of an electrodeposited 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.

The thickness of the electrodeposited coating shall be measured by one of the methods given in annex B.

7.3 Double- and triple-layer nickel coatings

The requirements for double- and triple-layer nickel coatings are summarized in Table 5.

Table 5 — Requirements for double- and triple-layer nickel coatings

Layer (type of nickel coating)	Specific elongation ^a %	Sulfur content % mass fraction ^b	Thickness as a percentage of total nickel thickness ^c	
			Double-layer	Triple-layer
Bottom (s)	> 8	< 0,005	≥ 60	50 to 70
Middle (high sulfur layer)	—	> 0,15		≤ 10
Top (b)	—	> 0,04 and < 0,15	10 to 40	≥ 30

^a The test method for determination of specific elongation (or ductility) is specified in annex C.

^b The sulfur contents are specified in order to indicate the type of nickel plating solution that is to be used. No simple method exists for determining the sulfur content of a nickel deposit on a coated article. However, an accurate determination is possible on a specially prepared test specimen (see annex D).

^c It will usually be possible to identify the type and to determine the ratios of nickel layers by microscopical examination of a polished and etched section of an article prepared in accordance with ISO 1463, or by means of a STEP test.

7.4 Adhesion

The coating shall be sufficiently adherent to the basis metal, and the separate layers of a multilayer coating shall be sufficiently adherent to each other, to pass the file test or the thermal shock test specified in ISO 2819. There shall not be any detachment of the coating from the substrate, or any separation between layers of the coating.

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

7.5 Corrosion resistance in CASS and acetic acid salt spray

Coated articles shall be subjected to one of the corrosion tests specified in Table 6 for the stated time appropriate for the particular service condition number. The corrosion tests are described in ISO 9227. The particular test to be used shall be specified by the purchaser. In order to assure that the coatings render a useful service, the duration of the corrosion test shall be approved by the purchaser, taking into account the requirements of the particular application and its intended service. The duration and results of these tests, however, may bear little relationship to the service

life of the coated article. After the articles have been subjected to the appropriate corrosion test, they shall be examined and rated in accordance with ISO 10289. The rating after corrosion testing shall be 9 or 10.

Nickel and copper plus nickel coatings without chromium topcoats are not widely used and, as a result, there is limited information about their performance in accelerated tests and in actual service.

Table 6 — Corrosion tests appropriate for each service condition

Service condition number	Duration of corrosion test <i>h</i> ^{a, b}	
	CASS test (ISO 9227)	Acetic acid salt spray test (ISO 9227)
3	16	96
2	8	48
1	4	8

^a The duration of each corrosion test has not been established experimentally and the times indicated are provided as guidance only.

^b The salt spray corrosion tests specified in ISO 9227 provide a means of controlling the continuity and quality of the coating. However, the duration of these tests bears little relationship to the service life of the finished article, especially in connection with the nickel coatings covered in this International Standard.

7.6 STEP test requirements

When specified by the purchaser, the electrochemical potential differences between individual nickel layers shall be measured for multilayer coatings.

NOTE 1 Although universally accepted STEP values have not been established, some agreement exists for required ranges; e.g., the STEP potential difference between the semi-bright and bright nickel layer is within the range of 100 mV to 200 mV, and the semi-bright nickel layer is always more noble (cathodic) to the bright nickel layer.

NOTE 2 In triple-layer nickel coatings, the STEP potential difference between the special high-activity nickel layer and the bright nickel layer is within the range of 15 mV to 35 mV, and the high-activity layer is always more active (anodic) than the bright nickel layer.

7.7 Ductility

The specific elongation or ductility of the semi-bright nickel layer in multilayer nickel coatings, as well as for copper undercoats, shall be that specified in Table 5 when tested in accordance with the method specified in annex C.

7.8 Stress relief heat treatments before coating

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

Steels with oxide or scale shall be cleaned before application of the coatings. For high-strength steels, non-electrolytic alkaline and anodic alkaline cleaners, as well as mechanical cleaning procedures, are preferred to avoid the risk of producing hydrogen embrittlement during cleaning operations.

7.9 Hydrogen embrittlement relief treatment

Steel parts having an ultimate tensile strength equal to or greater than 1 000 MPa (31 HRC) or above, as well as surface hardened parts, shall receive hydrogen embrittlement relief heat treatment according to the procedures and classes described in ISO 9588 or as specified by the purchaser.

The effectiveness of the hydrogen 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.

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

NOTE The coatings described in this International Standard are rarely, if ever, applied to steel parts having a tensile strength greater than 1 000 MPa, and rarely, if ever, heat treated. If they are applied to steels that are susceptible to hydrogen embrittlement and heating after electroplating is required, the purchaser should be aware that heating may discolour and embrittle nickel coatings that contain sulfur.

8 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
(informative)

Examples of service conditions for which the various service condition numbers are appropriate

A.1 Service condition number 3

Service outdoors where occasional or frequent wetting by rain or dew may occur.

A.2 Service condition number 2

Service indoors where condensation may occur.

A.3 Service condition number 1

Service indoors in warm, dry atmospheres.

Annex B (normative)

Thickness test methods

B.1 General

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

B.2 Destructive

B.2.1 Microscopical method

Use the method specified in ISO 1463 with, if required, the nitric acid/glacial acetic acid etchant specified therein or, for coatings of copper plus nickel, a solution of 1 part by volume of nitric acid (density = 1,40 g/ml) to 5 parts by volume of glacial acetic acid.

NOTE The use of these etchants enables the thickness of the different layers in double- and triple-layer coatings to be distinguished and hence measured.

B.2.2 Coulometric method

The coulometric method specified in ISO 2177 may be used to measure the total thickness of the nickel and the thickness of the copper underlayer, 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 individual layers in multilayer coatings.

B.2.4 STEP Test

The thickness of the individual nickel layers in double- and triple-layer coatings can be measured by a STEP test.

NOTE In cases of dispute, the coulometric method may be used for measuring the thickness of the chromium coating and for nickel coatings of thickness less than 10 μm , and the microscope method shall be used for measuring the thickness of nickel coatings and undercoats 10 μm and above.

B.3 Non-destructive

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

Use the method specified in ISO 2361.

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

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

Use the method specified in ISO 3543.

BS ISO 1458:2002

NOTE This method determines the total coating thickness, including that of a copper undercoat, if present. The thickness of this undercoat can, however, be distinguished from that of the outer coating by using this method in conjunction with that specified in ISO 2177, for nickel and chromium coatings, or in conjunction with that specified in ISO 2361, for nickel coatings.

B.3.3 X-ray spectrometry

Use the method specified in ISO 3497.

Annex C (normative)

Ductility test

C.1 Scope

This annex specifies a method for determining the specific elongation or ductility of nickel electrodeposited on a test piece and provides a means of assessing the ductility of the coating.

NOTE The test is used to check that the type of nickel deposit complies with the coating requirements specified in Table 5, and may be used in assessing the ductility of other coatings.

C.2 Principle

The test is based on bending a test piece that has been electroplated with nickel around a mandrel of specified diameter to produce a minimum elongation of 8 %, followed by examining the coating visually for signs of cracking.

C.3 Apparatus

C.3.1 Mandrel, diameter 11,5 mm \pm 0,1 mm.

C.4 Preparation of test piece

Prepare a coated test piece 150 mm long, 1,0 mm wide and 1,0 mm \pm 0,1 mm thick as follows.

Polish a sheet of the appropriate basis metal, similar to that of the articles being coated except that the sheet may be of soft brass if the basis metal is zinc alloy. Use a sheet that is sufficiently large in order to allow the test strip to be cut from it after trimming off a border at least 25 mm wide all round.

Electroplate the polished side of the sheet with nickel to a thickness of 25 μ m under the same conditions and in the same bath as are used with the corresponding articles.

Cut the test piece from the coated sheet with a guillotine or flat shear. Round or chamfer the longer edges of the test strip, at least on the plated side, by careful filing or grinding.

C.5 Procedure

Bend the test piece with the coated side in tension, by steadily applied pressure, through 180° over the mandrel (C.3.1) until the two ends of the test piece are parallel. Ensure that contact between the test piece and the mandrel is maintained during bending. Examine the convex side of the bent test piece visually for cracks.

C.6 Expression of results

The coating is deemed to comply with the minimum requirement of an elongation of 8 % provided that after testing there are no cracks passing completely across the convex surface.

Annex D (normative)

Determination of the sulfur content of electrodeposited nickel coatings

D.1 Determination by combustion and iodate titrimetry

The sulfur content of electrodeposited nickel shall be determined, when required, by combustion of a test portion of the nickel in a stream of oxygen in an induction furnace. The sulfur dioxide that is evolved is absorbed in acidified potassium iodide/starch solution. The solution is then titrated with potassium iodate solution that has been freshly standardized against steels of known sulfur content to compensate for day-to-day variations in sulfur dioxide recovery. Compensation is made for the blank to allow for the effects of crucibles and accelerators.

This method is applicable to electrodeposited nickel having sulfur contents, expressed as S, in the range of 0,005 % mass fraction to 0,5 % mass fraction.

NOTE Commercial instruments are available that utilize infra-red and thermal conductivity detection methods to measure the sulfur dioxide produced by combustion and that have computer facilities that permit direct read-out of sulfur content.

D.2 Determination by sulfide formation and iodate titrimetry

Alternatively, the sulfur content of electrodeposited nickel shall be determined by converting the sulfur in the nickel to hydrogen sulfide by treatment with hydrochloric acid containing dissolved hexachloroplatinic acid, as an accelerator for dissolution. The hydrogen sulfide that is evolved is reacted with ammoniacal zinc sulfate. The zinc sulfide that is formed is titrated with standard volumetric potassium iodate solution. Results are based on potassium iodate as the primary standard.

Bibliography

- [1] ISO 1456, *Metallic coatings — Electrodeposited coatings of nickel plus chromium and of copper plus nickel plus chromium*
- [2] ISO 4525, *Metallic coatings — Electroplated coatings of nickel plus chromium on plastics materials*
- [3] ISO 4526, *Metallic coatings — Electroplated coatings of nickel and nickel alloys for engineering purposes*
- [4] ISO 6158, *Metallic coatings — Electrodeposited coatings of chromium for engineering purposes*
- [5] 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*

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