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Metallic coatings — Physical vapour-deposited coatings of cadmium on iron and steel — Specification and test methods

Revêtements métalliques — Revêtements de cadmium appliqués par vide sur fer et acier — Spécification et méthodes d'essai



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ISO 22778:2006(E)

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 22778 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 3, *Electrodeposited coatings and related finishes*.

Introduction

Cadmium coatings are applied by physical vapour-deposition techniques to iron, steel and other substrates to enhance appearance and improve resistance to corrosion. Unlike electroplating, physical vapour deposition does not introduce hydrogen into the basis material. Hence, vapour-deposited cadmium coatings are especially beneficial for protecting high-strength steels that are susceptible to hydrogen embrittlement. Hydrogen, however, can be introduced into the substrate during fabrication, cleaning, pickling and other treatments, and care must still be exercised to prevent the introduction of hydrogen prior to vapour deposition.

Cadmium is toxic and health, safety and environmental concerns are now eliminating its non-essential uses. There remain, nevertheless, critical applications, often aerospace-related, where the unique properties of cadmium coatings, for example, their corrosion resistance, intrinsic lubricity, ductility, electrical conductivity and low contact resistance, make continued use necessary. One benefit of depositing cadmium by physical deposition techniques is that the process is conducted in a sealed vacuum chamber and exposure to cadmium is at an absolute minimum.

The corrosion resistance of physical vapour-deposited cadmium coatings may be further enhanced by the use of chromate conversion coatings and other supplementary treatments.

Metallic coatings — Physical vapour-deposited coatings of cadmium on iron and steel — Specification and test methods

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

1 Scope

This International Standard specifies requirements for high-purity cadmium coatings produced by physical vapour deposition on iron, steel, and other substrates. It provides test methods to determine that the requirements have been met, and a method for designating the cadmium and chromate conversion coatings and other supplementary treatments.

Cadmium coatings are not suitable for production items that will reach a temperature of 225 °C or higher in service, or that will come into contact with other parts that will reach that temperature.

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, 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, Electroplating and related processes — Vocabulary

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

ISO 2360, Non-conductive coatings on non-magnetic electrically conductive basis materials — Measurement of coating thickness — Amplitude-sensitive eddy-current method

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

ISO 2859 (all parts), Sampling procedures for inspection by attributes

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 3892, Conversion coatings on metallic materials — Determination of coating mass per unit area — Gravimetric methods

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- ISO 4518, Metallic coatings Measurement of coating thickness Profilometric method
- ISO 4519, Electrodeposited metallic coatings and related finishes Sampling procedures for inspection by attributes
- ISO 4520, Chromate conversion coatings on electroplated zinc and cadmium coatings
- 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 12686, Metallic and other inorganic coatings Automated controlled shot-peening of metallic articles prior to nickel, autocatalytic nickel or chromium plating, or as a final finish
- ISO 16348, Metallic and other inorganic coatings Definitions and conventions concerning appearance
- EN 12508, Corrosion protection of metal and alloys Surface treatment, metallic, and other inorganic coatings — Vocabulary
- IEC 60454, Specifications for pressure-sensitive adhesive tapes for electrical purposes Part 2: Methods of test

Terms and definitions

For the purposes of this document, the terms and definitions in ISO 2064, ISO 2080, ISO 2859, ISO 4519 and EN 12508 apply.

Information to be supplied by the purchaser to the processor

When ordering articles for physical vapour deposition of cadmium in accordance with this International Standard, the purchaser shall provide the following information in writing in the contract or purchase order, or in the engineering drawing:

- the designation (see Clause 5);
- the specification and metallurgical condition of the basis metal, any process-temperature limitations (see 6.1.2) and shot-peening requirements [see 6.1.3 c)];
- the significant surface, including the coating of holes, recesses and presence of rack marks (see 6.1.1 and 6.3.1);
- details of any sensitive material and whether ultra high-purity argon is required (see 6.1.2);
- the requirement for any consolidation, for example, by glass bead peening (see 6.4.1); e)
- the requirement for coating thickness (see 5.4 and Table 1) and a chromate conversion coating (see 5.5 f) and 6.4.2);
- the requirement for a supplementary treatment, such as paint, and details of the specification involving the organic finish (see 5.5 and 6.4.3);

- h) the requirement for coating adhesion (see 6.3.3 and Annex C).
- i) the requirement for special test specimens and for the type of test method, for example, destructive or non-destructive (see 6.6);
- j) sampling and inspection requirements (see Clause 7 and Annex D).

5 Designation

The designation shall appear on engineering drawings, in the purchase order, the contract or in the detailed product specification. The designation specifies the basis material, the requirements for stress relief before vapour deposition, the nominal composition and thickness of the vapour-deposited cadmium coating, the type of chromate conversion coating and other supplementary treatment, and the heat treatment to reduce susceptibility to hydrogen embrittlement.

5.1 General

The designation shall comprise the following:

- a) the term: vapour-deposited coating;
- b) the number of this International Standard, ISO 22778;
- c) a hyphen;
- d) the chemical symbol of the basis metal;
- e) a stroke (/); symbols for the cadmium coating, as well as any coatings that may be applied prior to and after deposition, separated by strokes for each stage in the coating sequence in the order of application. The coating designation shall include the thickness of the coating, in micrometres.

5.2 Basis metal

The basis metal shall be designated by its chemical symbol, or its principal constituent, if it is an alloy. For example:

- Fe for iron and steel;
- Zn for zinc alloys;
- Cu for copper and copper alloys;
- Al for aluminium and aluminium alloys.

The specific alloy may be identified by its standard designation (for example, its UNS number, or its national or regional equivalent) placed between the symbols, <>, for example, Fe<G434000>. See Reference [3] in the Bibliography.

5.3 Pre-process stress-relief heat treatment

Stress-relief heat treatment prior to coating may be required for some basis materials. Brackets shall be placed around the letters SR, the temperature in degrees Celsius, and the time in hours. The temperature shall be in parentheses after the letters SR; for example [SR(210)1].

5.4 Type and thickness of cadmium

The cadmium coating shall be designated by its chemical symbol, Cd, followed by a number giving the minimum local thickness of the coating in micrometres. For example, Cd5, designates a cadmium coating that is $5 \, \mu m$ thick. The minimum thickness of the vapour-deposited cadmium coatings shall be selected in accordance with Table 1.

Allowances for the thickness of the coating should be made by adjusting the dimensional tolerances during the manufacture of threaded items, and those with close tolerances.

Table 1 — Minimum thickness requirements for physical vapour-deposited cadmium coatings

Application	Minimum local thickness
	μm
Steels, normal requirements	15
Threaded articles of less than 20 mm nominal diameter, as follows:	
< 3 mm	4
3 to < 6 mm	5
6 to < 10 mm	6
10 mm and over	8
Articles with bore dimensions, as follows:	
< 3 mm	5
3 mm and over	8
Other small articles	8

5.5 Chromate conversion coatings and other supplementary treatments

The type of chromate conversion coating and other supplementary treatments shall be designated by the symbols given in Tables 2 and 3.

Table 2 — Symbols for chromate conversion coatings

Code	Туре	Typical appearance	Coating mass per unit area	
	Name		g/m ²	
Αa	Clear	Transparent, clear to bluish	≤ 0,5	
С	Iridescent	Yellow iridescent	< 0,5 to < 1,5	
D	Opaque	Olive-green	> 1,5	
F	Black	Black	\leqslant 0,5 to \leqslant 1,0	
Chromium in hexavalent form may or may not be present.				

Table 3 — Supplementary treatments other than conversion coatings

Code	Type of treatment	
T1	Application of paints, varnishes, powder coatings or similar coating materials	
T2	Application of organic or inorganic sealants.	
Т3	Dyeing	
T4	Application of grease or oil, or other lubricants	
T5	Application of wax	

NOTE The function of chromate conversion coatings and other supplementary treatments is to retard or prevent the formation of white corrosion products on surfaces exposed to corrosive atmospheres, and to delay the appearance of red corrosion of cadmium coatings on steel. The iridescent yellow to olive drab chromate films are satisfactory for the application of subsequent paint coatings, but bleached or leached chromate films are not recommended as a supplementary finish with vapour-deposited cadmium coatings.

Chemical conversion coatings that do not contain hexavalent chromium are commercially available. Some contain trivalent chromium; others are chromium-free. Substitutes shall meet the corrosion requirements given in Table 4.

5.6 Post-coating heat treatment

Heat treatment to reduce the susceptibility of high-strength steels to hydrogen embrittlement may be required in some circumstances (see Clause 8). Brackets shall be placed around the letters ER, the temperature in degrees Celsius, and the time in hours. The temperature shall be in parentheses after the letters ER; for example [ER(400)12].

5.7 Examples of coating designations

Example of a physical vapour-deposited cadmium coating 8 µm thick on steel (Fe), with a supplementary chromate conversion coating that is iridescent yellow (C):

Physical vapour-deposited coating ISO 22778 - Fe/Cd8/C

Example of a coating $5 \, \mu m$ thick on steel, with a supplementary chromate conversion coating that is transparent or colourless (A) and that shall receive a subsequent organic sealant (T2):

Physical vapour-deposited coating ISO 22778 - Fe/Cd5/A/T2

Example of a coating on high-strength steel that is to be stress relieved at 150 °C for 2 h before applying a cadmium coating 5 µm thick, with a supplementary black chromate conversion (F):

Physical vapour-deposited coating ISO 22778 - Fe/[SR(150)2]/Cd5/F

6 Requirements

6.1 General

6.1.1 Basis material

This International Standard does not specify requirements for the condition, finish and surface roughness of the basis metal prior to physical vapour deposition of cadmium. Surfaces to be coated, however, shall be smooth and free from oxides, tool marks, pitting, intergranular attack, or other defects.

6.1.2 Equipment and materials

The equipment, process and materials used for the physical vapour deposition of cadmium shall be capable of producing a uniform coating, covering completely all significant surfaces, including roots of threads, recesses, sharp corners, holes and other areas specified in the drawing, contract or purchase

NOTE Suitable rotating and revolving racks and fixtures may be used to ensure that all significant surfaces are cleaned and coated to the specified thickness limits.

- High-purity argon (at least 99,995 % pure) shall be used during substrate conditioning and coating, as required. For sensitive material, ultra-high-purity argon (at least 99,998 % pure) shall be used.
- The equipment shall be capable of producing a vacuum of 7×10^{-4} Pa, or better, in the coating chamber.

Where water-cooled surfaces are present in the chamber, including chamber walls, then a means of heating the circulating water is to be used, to ensure that the chamber surfaces are warm enough to prevent condensation when open to the atmosphere.

- The cadmium metal used as a source material shall contain not less than 99,95 % cadmium by mass; the mass fraction of mercury shall not exceed 0,004 %, and total impurities shall not exceed 0,05 %. The composition of cadmium cannot be determined by using chemical methods, but shall be obtained by difference using spectrographic means for determining the impurities.
- The equipment and process shall be such that any temperature rise in the items will have no adverse effects on the properties of the basis metal or the coating, or on the adhesion between the basis metal and the coating [see 4 b)].

Annex A contains additional information about equipment, materials and processing parameters.

6.1.3 Surface preparation

- Steels of tensile strength 1 000 MPa and above shall be cleaned before the application of the coating, preferably by using non-electrolytic alkaline and anodic alkaline cleaners to avoid the risk of creating hydrogen embrittlement problems as a result of the cleaning procedure. For other materials, appropriate cleaning procedures, or procedures as specified in the drawing, contract or purchase order, shall be followed.
- Abrasives used on any metal or alloy shall not have been used on any dissimilar metals or alloys. Separate abrasive cleaning media shall be used for different alloy families, for example, iron and its alloys, aluminium and its alloys. After abrasive cleaning, residues shall be removed from the surfaces of articles prior to subsequent processing.
- Shot peening of the basis metal or substrate, if required, shall be in accordance with ISO 12686, and shall be performed before final cleaning and the application of the coating [see 4 b)].
- Only items of a similar basis metal shall be processed together.

Stress-relief heat treatment before coating 6.2

Steel parts that have an ultimate tensile strength of 1 000 MPa and above and that contain tensile stresses caused by machining, grinding or cold-forming operations shall be given a stress-relief heat treatment prior to cleaning and metal deposition. The stress-relief heat treatment shall be as specified in ISO 9587, or as specified by the purchaser.

6.3 Coating requirements

6.3.1 Appearance

The coating shall be present on all significant surfaces and shall be smooth, continuous, uniform, adherent, fine-grained and free from stains, burns, blisters, pits, nodules, cracks, porosity, uncoated areas, edge build-up or other defects. The cadmium coating shall show no indication of contamination or improper operation of equipment, such as powered or darkened areas. The coating shall extend into holes and recesses to a distance as specified in the drawing, contract or purchase order [see 4 c)].

Masking material is to be completely removed from non-significant surfaces, and the masked areas shall be clean and free from any defect and cadmium coating. The line of demarcation between the coated and non-coated areas shall be clear-cut. Lifting of the deposit at the edge shall not be acceptable.

Approved samples of artefacts shall be used for comparison purposes, to control the appearance of the final finish (see ISO 16348).

NOTE Unavoidable rack marks may be permitted if specified in the drawing, contract or purchase order, or with the prior agreement of the purchaser.

6.3.2 Thickness

The coating thickness specified in the designation shall be the minimum local thickness. The minimum local thickness of the coating shall be measured on any point on the significant surface that can be touched by a ball 20 mm in diameter. Thickness shall comply with the requirements of Table 1.

The minimum local thickness of the vapour-deposited cadmium coating shall be measured by one of the following methods: ISO 1463, ISO 2177, ISO 2360, ISO 3497, ISO 3543, ISO 3882, ISO 3892, ISO 4518 or ISO 9220. The referee methods are specified in ISO 1463, ISO 3497 or ISO 9220. For the determination of the average thickness (ISO 3892) of the cadmium coating, see Annex B.

6.3.3 Adhesion

The coating shall adhere to the basis metal without chipping, flaking, or other coating damage and shall conform to the minimum requirements given in Annex C [see 4 j)].

6.3.4 Coating composition

The coating shall contain not less than 99,9 % cadmium by mass fraction. The referee method for chemical analysis of the coating shall be the mass spectrometry method.

6.4 Supplementary treatments

Prior to the application of supplementary coatings, the cadmium-coated surface may require solvent degreasing. No other form of cleaning shall be allowed.

6.4.1 Treatment with glass beads

If required, the physical vapour-deposited coating may be peened with glass beads [see 4 e)].

NOTE Peening improves the compactness of the coating, enhances corrosion resistance and is a qualitative test for the adhesion of the coating to the substrate. Peening may be carried out by using glass beads $50 \, \mu m$ to $100 \, \mu m$ in diameter, at a pressure of 2.8×10^5 Pa to produce a satin finish on the surface. Thicker coatings deposited at relatively high pressure often develop a coarse-grained structure which can be converted to a fine-grained one by glass-bead peening.

6.4.2 Chromate conversion coating

Unless otherwise specified, chromate conversion coating shall be applied in accordance with ISO 4520. The chromate films shall be adherent, continuous and free from any flaws and defects.

6.4.3 Organic coatings

If specified, physical vapour-deposited items shall be given an organic protective coating in accordance with the requirements of the drawings, contract, or purchase order [see 4 g)].

Corrosion testing in neutral salt spray

When tested in accordance with the neutral salt spray test specified in ISO 9227, the articles shall show no evidence of corrosion of the basis metal when exposed for the durations given in Table 4.

Table 4 — Duration of salt spray testing for physical vapour deposited cadmium coatings with organic finishes

Minimum local thickness of	Test duration, h			
coating µm	Without chromate conversion coating ^a	With chromate conversion coating ^a		
15	408	504		
8	312	408		
^a See the last paragraph of 5.5 for the use of non-chromate type conversion coatings.				

For applications where chromated surfaces are not to be subsequently painted, accelerated tests for corrosion resistance shall, also, be carried out in accordance with ISO 9227. The test shall commence 24 h to 36 h after chromating, and no white corrosion product of the cadmium shall be visible on the chromated articles after 96 h exposure to the test environment, when visually examined with normal or corrected vision.

The duration and results of artificial-atmosphere corrosion tests may bear little relationship to the service life of the coated article and, therefore, the results obtained should not be regarded as a direct guide to the corrosion resistance of the tested coatings in all environments where these coatings may be used.

6.6 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 it is not practical to submit the coated articles to destructive tests because the articles are few in number or too expensive [see 4 k)].

If special test specimens are to be used, their number, shape and size shall be specified in the drawing, contract or order.

If separate test specimens are to be used to represent the coated articles in a test, the specimens shall be of the same nature, same surface condition, of the same material and in the same metallurgical condition as the articles they represent, and shall be placed in the same production lot of, and be processed along with, the coated article they represent.

A test specimen used to represent an article in a coating thickness test shall be introduced into the process at the point where the coating or coatings are applied, and it shall be carried through all steps that might influence the coating thickness.

When both destructive and non-destructive tests exist for the measurement of a characteristic, the purchaser shall specify which is to be used and which test is to be considered as destructive or non-destructive because a test may destroy the coating but it may be in a non-critical area.

NOTE When a test specimen is used as a coated article in a thickness test, the specimen will not necessarily have the same thickness distribution as the article, unless the specimen and the article are of the same general size and shape. Therefore, before coated articles may be accepted on the basis of thickness tests performed on the representative test specimens, the relationship between the thickness on the specimen and the thickness on the article is to be established. The criterion of acceptance will be the thickness on the specimen that corresponds to the designated thickness on the article.

7 Sampling

A random sample shall be selected from the inspection lot in accordance with ISO 2859 and ISO 4519. The articles shall be inspected for conformance to the requirements of Annex D [see 4 j)].

8 Rejection

If the deposited cadmium coating does not meet the thickness or adhesion requirements, then the items may be coated a second time in accordance with the requirements of this International Standard.

If it is necessary to strip the coating, the method of removal shall not damage the substrate.

If the coating is stripped by a wet process, then steel items shall be treated in accordance with ISO 9588 immediately after stripping. Items of steel of tensile strength 1 000 MPa and above shall not be chemically stripped more than once, or coated more than twice. For parts treated in accordance with ISO 9588 after stripping with a wet process, the effectiveness of the embrittlement-relief heat treatment may be determined by test methods specified by the purchaser, or by test methods described in appropriate International Standards. For example, ISO 10587 describes a test method for testing threaded articles for residual hydrogen embrittlement-relief heat treatment, and ISO 15724 for measuring the relative diffusible hydrogen concentration in steels.

Annex A

(informative)

Additional information concerning equipment, substrate preparation and process parameters

A.1 General

- a) All cold-working processes, for example, forming, straightening, grinding and machining, where required, are to be completed before any preparation, stress relieving and coating, unless otherwise specified in the drawing, contract or purchase order.
- b) All heat treatment operations, including those designed to reduce the susceptibility to hydrogen embrittlement (see 6.2 and Clause 8) as a consequence of cleaning operations, are to be completed prior to the deposition of cadmium.
- c) Due allowance should be made for surface preparation (especially cleaning) and the application of a uniform coating thickness, in order to obtain the specified surface finish and dimensional tolerance specified in the drawing, contract or purchase order.
- All surfaces are to be free from contaminants, such as soap, drawing compounds, oil, grease, machining fluid, polishing compounds, discolouration, oxide scale, or other foreign matter, so as to produce a chemically clean surface. Drying by means of chlorinated solvents is not to be allowed.
- e) Careful control should be exercised in the cleaning of partially closed fabrications and lapped joints to reduce the risk of residue retention. Care should also be taken during the abrasive cleaning of thin sections, to avoid distortion.
- Areas which are not required to be coated are to be kept free from cadmium by masking prior to the application of the coating. Aluminium or corrosion-resisting steel masking, foil or plugs are to be used to cover areas where the coating is not required. Organic masking materials that are unstable at high temperatures are not be used.
- Contamination of cleaned items, for example, by handling with bare skin, is to be avoided by the use of clean, dry cotton gloves.

NOTE Difficulty may be experienced in coating small holes and recesses to a specified depth.

A.2 Equipment

The equipment used should consist of a coating chamber with associated pumps, controls and power supplies containing the following:

- A means of producing energetic bombardment of the substrate prior to coating and at a suitable intensity, throughout deposition of the growing film.
- A means of producing a coating flux of cadmium, such as an evaporative or sputtering source. According to the technique used, it may be necessary to include a shutter, which may be interposed between the cadmium source and the articles to be coated, except during coating.

A shutter may be necessary to reduce radiant substrate heating from the cadmium source.

- A means of holding and manipulating the articles to ensure specified coating uniformity.
- A means of ensuring that the substrate temperature of the articles is controlled within the specified limits.

NOTE 2 The substrate temperature is controlled by suitable modification of the process parameters.

A.3 Substrate conditioning

After cleaning and drying, as necessary, articles are to be transferred, as soon as possible, into the coating chamber using suitable gloves.

The coating chamber is then pumped to the vacuum specified in 6.1.2 c).

To ensure adequate coating adhesion, articles are then subjected to substrate conditioning to remove absorbed and chemically bound contaminants. Substrate conditioning may be achieved by intimate contact with a glow-discharge plasma (sputter cleaning), bombardment with energetic ions, radicals or atoms, or by reactive plasma cleaning or by other suitable methods. Certain conditioning treatments may require the coating chamber to be back filled with argon or another gas of sufficiently high purity. In addition, manipulation of the items to ensure complete exposure may be required.

On completion of the specified substrate conditioning, transition to the coating deposition stage of the process should be smooth and uninterrupted.

NOTE The chamber is not to be vented to the atmosphere after substrate conditioning prior to coating. Should this occur, the substrate conditioning stage is to be repeated. In some circumstances, it may be necessary to unload and reclean the articles before restarting the process.

A.4 Cadmium coating

The coating process is to be carried out in the evacuated coating chamber in which the articles have been conditioned. At the conclusion of the substrate conditioning, a smooth transition is effected by introducing the cadmium coating flux, for example, by opening the shutter and adjusting the process conditions.

It is essential that a sufficiently energetic ion, or radical, or atom bombardment of the growing film occurs throughout the deposition process according to the technique used, for example by glow discharge. Where necessary, the articles are rotated and revolved to ensure uniform coating deposition.

The cadmium source shutter is then closed, the cadmium source switched off and the coated articles and sources allowed to cool before air is admitted to the coating chamber. The articles are then removed from the coating chamber and the masking material is removed from the articles prior to examination.

The substrate conditioning techniques and the coating conditions will vary with the nature, size, and shape of the items to be coated. These variable factors are to be determined experimentally for each type of article, to ensure that the requirements for thickness and adhesion of the coating are met.

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Annex B

(normative)

Determination of the average thickness of cadmium coatings

Weigh the clean article and then immerse it in an aqueous solution containing 10 % to 30 % ammonium nitrate at room temperature, with occasional stirring, until the cadmium coating is dissolved (usually about 10 min). Remove the item, wash, dry and reweigh.

The average thickness of the cadmium coating, S, in micrometres, may be calculated from the following formula:

$$S = \frac{(m_1 - m_2) \times 10^4}{A \times 8,64}$$

where

is the mass of the article plus the coating, in grams; m_1

is the mass of the article after the coating has been removed, in grams; m_2

is the area of the coating, in square centimetres; A

8,64 is the specific gravity of cadmium.

Annex C (normative)

Adhesion test methods

C.1 General

Adhesion shall meet the requirements specified in ISO 2819.

C.2 Additional adhesion test methods

Adhesion of the coating to the basis metal can also be determined by the following additional methods.

- a) By scoring the surface of the coated article to expose the basis metal and examining it at a minimum of $4 \times \text{magnification}$ (normal or corrected vision) for evidence of non-adhesion.
- b) A clean surface of the item or test panel shall be scored at 2 mm intervals in a cross-hatched pattern. A strip, for example, 100 mm long by 25 mm wide, of pressure-sensitive tape (see IEC 60454), having a peel force for adhesion to steel of at least 3.5 N/10 mm, shall be applied firmly to the prepared surface. After 5 min, it shall be removed by peeling back at 180° as rapidly as possible. The test shall be carried out at 20 °C ± 5 °C. There shall be no sign of separation of the coating from the surface.
- c) Articles shall be clamped in a vice and the projecting part bent back and forth until rupture occurs. If the edge of the ruptured coating can be peeled back, or if the separation between the coating and the basis metal can be seen at the point of rupture when examined, adhesion is unsatisfactory.
- d) A small area of the plated surface shall be rubbed rapidly and firmly with a suitable tool for about 15 s at a pressure sufficient to burnish the coating at each stroke, but not to cut the deposit. The coating shall not become blistered or otherwise detached from the basis metal. A suitable tool is a steel rod of 6 mm diameter with a smooth hemispherical end.
- e) Articles may be shot peened in accordance with ISO 12686. There shall be no separation of the coating from the basis metal.

Annex D (normative)

Sampling

D.1 General

The articles in the sample shall be inspected for conformance to the requirements of this specification and the lot shall be classified as conforming to, or not conforming to, each requirement in accordance with the criteria of the sampling plans given in D.2.

D.2 Sampling plans

- a) A number of samples shall be randomly selected and subjected to the test for coating composition. Each batch or lot of the coating material shall be tested for purity (see 6.1.2).
- b) A sample comprising 1 % from each batch of coated articles, but not less than two items for each test, prepared and processed together, shall be selected for coating thickness and coating adhesion tests, as specified in 6.3.2, 6.3.3 and Annex C. The items shall be selected randomly and from all positions on the rack or fixture, and the number of articles for each test shall be approximately equal.
- c) In exceptional circumstances, such as the coating of single large items, the sampling procedure specified above may be modified. Special test specimens coated along with the articles shall be used, due consideration being given to their shape, size and material (see 6.6). The treatment of the test specimen shall be representative of that applied to the articles being coated.
- d) If any coated item fails a test (or tests), a further 1 % sample shall be selected in accordance with item b) and the whole of this sample shall be subjected to the relevant test (or tests). If any part of this sample fails, the whole batch shall be rejected.
- e) A sample comprising not less than two items, from each class of coating, shall be selected and subjected to salt spray testing (see 6.5).

Bibliography

- [1] 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
- [2] ISO 15724, Metallic and other inorganic coatings Electrochemical measurement of diffusible hydrogen in steels Barnacle electrode method
- [3] ASTM DS-56G, *Metals and Alloys in the Unified Numbering System*, 8th edition, published by American Society for Testing and Materials, West Conshohocken, PA, 1998



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