

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 1457

ELECTROPLATED COATINGS OF COPPER PLUS NICKEL
PLUS CHROMIUM ON STEEL (OR IRON)

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BRIEF HISTORY

The ISO Recommendation R 1457, *Electroplated coatings of copper plus nickel plus chromium on steel (or iron)*, was drawn up by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings*, the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1457 which was circulated to all the ISO Member Bodies for enquiry in May 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Israel	Spain
Czechoslovakia	Italy	Sweden
Finland	Netherlands	Switzerland
France	New Zealand	Thailand
Germany	Norway	Turkey
Hungary	Poland	U.A.R.
India	Portugal	United Kingdom
Iran	South Africa, Rep. of	U.S.A.

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in April 1970, to accept it as an ISO RECOMMENDATION.

FOREWORD

This ISO Recommendation covers a range of coatings of copper plus nickel plus chromium on steel (or iron) for various service conditions. In this ISO Recommendation the surface condition of the basis metal prior to plating is not specified and agreement should be reached between the plater and the purchaser regarding the degree of roughness of the basis metal that is acceptable.

The purchaser should specify the service condition number denoting the severity of the conditions the coating is to withstand. It is not necessary to state the classification number of the coating, defined in terms of the nature, thickness and properties of the deposits it comprises, but the purchaser may include this if he wishes.

For copper, nickel and chromium deposits, the minimum thickness requirements apply only to those portions of the significant surface that can be touched by a ball 20 mm in diameter. If the design of the article is such that it cannot at all points be touched by a 20 mm ball, the minimum thickness permitted on specified areas should be agreed between the purchaser and the supplier.

The duration of the laboratory corrosion tests is varied according to the service condition number. Acceptance or rejection of an article subjected to corrosion tests is governed by the extent of basis metal corrosion. Surface deterioration of the coating itself will occur on some types of coating, and it is recommended that the extent to which this can be tolerated should be subject to agreement between supplier and purchaser.

IT IS ESSENTIAL THAT THE PURCHASER STATE THE BASIS METAL AND THE SERVICE CONDITION NUMBER; IN ADDITION HE MAY ALSO STATE THE CLASSIFICATION NUMBER.

MERELY TO ASK FOR PLATING TO BE CARRIED OUT IN ACCORDANCE WITH ISO RECOMMENDATION R 1457 WITHOUT THIS ADDITIONAL INFORMATION IS INSUFFICIENT.

ELECTROPLATED COATINGS OF COPPER PLUS NICKEL PLUS CHROMIUM ON STEEL (OR IRON)

1. SCOPE

This ISO Recommendation defines the characteristic properties as well as the classification and the symbols of electroplated coatings of copper plus nickel plus chromium on steel (or iron). Furthermore, it describes, in annexes, the test methods for these coatings.

2. FIELD OF APPLICATION

This ISO Recommendation applies to electroplated coatings defined in section 1, *except* for the following :

- coatings applied to machine screw threads (with tolerance);
- coatings applied to sheet, strip or wire in the unfabricated form, or to coil springs;
- coatings applied for other than protective and decorative purposes.

3. DEFINITION

For the purposes of this ISO Recommendation the following definition applies :

Significant surface. The part of the surface which is essential to the appearance or serviceability of the article and which is to be covered by the coating.

When necessary, the significant surface should be the subject of agreement, and should be indicated on drawings, or by provision of suitably marked samples.

4. APPEARANCE

Over the significant surface, the plated article should be free from clearly visible plating defects such as blisters, pits, roughness, cracks or unplated areas, and should not be stained or discoloured. The extent to which blistering can be tolerated on non-significant surfaces should be the subject of agreement between supplier and purchaser. On articles where a contact mark is inevitable, its position should also be the subject of agreement between the supplier and the purchaser.

The article should be clean and free from damage. The purchaser should state the appearance required, for example bright, dull or satin. Alternatively, a sample showing the required finish should be supplied or approved by the purchaser.

5. HEAT TREATMENT FOR STEEL

When required by the purchaser, heat treatment as described below should be performed on certain steels to reduce the risk of damage by hydrogen embrittlement.

5.1 Stress relief before plating

- 5.1.1 Severely cold-worked steel parts, or parts made from steel of tensile strength of about 1000 MN/m^2 (100 kgf/mm^2) or greater which have been ground or subjected to severe machining after tempering, should normally be stress relieved. As a guide, they may be maintained, preferably, at the highest temperature within the limit imposed by the tempering temperature for 30 minutes, or maintained at 190 to 210°C for not less than 1 hour.
- 5.1.2 Some steels which have been carburized, flame-hardened or induction-hardened and subsequently ground would be impaired by the treatment given as guidance in clause 5.1.1, and should instead be stress relieved at a lower temperature; for example at 170°C for not less than 1 hour.

5.2 Heat treatment after plating

- 5.2.1 Components subject to fatigue or sustained loading stresses in service and made from severely cold-worked steels or from steels of tensile strength of about 1000 MN/m^2 (100 kgf/mm^2) or greater should be heat treated after plating. Guidance is given in Annex L.
- 5.2.2 Where the proposed temperature of heat treatment would be harmful, as for some surface-hardened parts, a lower temperature for a longer time may be required.

6. COATING REQUIREMENTS

6.1 Manner of specifying requirements

When ordering the plating of articles in accordance with this ISO Recommendation, the purchaser should state, in addition to the number of the ISO Recommendation, the service condition number denoting the severity of the conditions the coatings are required to withstand (see clause 6.2). He need not, but he may if he wishes, also state the classification number of the particular coating required (see clause 6.3). If the service condition number is quoted and not the classification number, the supplier is free to supply any of the classes of coating corresponding to the service condition number, but he should inform the purchaser of the classification number of the coating supplied.

6.2 Grading of service conditions

The service condition number indicates the severity of the service conditions in accordance with the following scale :

- 4 — exceptionally severe
- 3 — severe
- 2 — moderate
- 1 — mild

6.3 Classification of coatings

The classification number comprises the following elements :

- the chemical symbol, Fe, for the basis metal (steel or iron), followed by an oblique stroke;
- the chemical symbol for copper, Cu;
- a number indicating the minimum thickness (in micrometres) of the copper coating;
- the chemical symbol for nickel, Ni;
- a number indicating the minimum thickness (in micrometres) of the nickel coating;
- a letter designating the type of nickel deposit;
- the chemical symbol for chromium, Cr;
- a letter designating the type of chromium deposit and its minimum thickness.

6.3.1 *Copper deposit.* The number following the chemical symbol Cu indicates, in micrometres, the minimum thickness of the copper or brass (at least 50 % copper) deposit, measured by the method given in Annex B, at points on the significant surface agreed between purchaser and supplier or at any point on the significant surface that can be touched by a ball 20 mm in diameter.

6.3.2 *Nickel deposit.* The number following the chemical symbol Ni indicates, in micrometres, the minimum thickness of the nickel deposit, measured by the method given in Annex B, at points on the significant surface agreed between the purchaser and the supplier or at any point on the significant surface that can be touched by a ball 20 mm in diameter.

The type of nickel* is designated by the following symbols :

- b — for nickel deposited in the fully bright condition;
- p — for dull or semi-bright nickel requiring polishing to give full brightness, containing less than 0.005 % sulphur**, and having an elongation greater than 8 % when tested by the method given in Annex A;
- d — for a double-layer or triple-layer nickel coating, of which the bottom layer contains less than 0.005 % sulphur** and has an elongation greater than 8 % when tested by the method given in Annex A and the top layer contains more than 0.04 % sulphur**; the thickness of the bottom layer in double-layer coatings should be not less than 60 % of the total nickel thickness and in triple-layer coatings should be not less than 50 % of the total nickel thickness, the thickness of the top layer in either case being not less than 20 % of the total nickel thickness. If there are three layers, the intermediate layer should contain more sulphur than the top layer and should not exceed 10 % of the total nickel thickness.

6.3.3 *Chromium deposit.* The thickness of the chromium deposit should be measured by the method given in Annex C at points on the significant surface agreed between the purchaser and the supplier or at any point on the significant surface that can be touched by a ball 20 mm in diameter.

The type of chromium is designated by the following symbols placed after the chemical symbol Cr :

- r — for regular (i.e. conventional) chromium, having a minimum thickness of 0.3 μm ;
- f — for chromium free from cracks, when checked by the method given in Annex D, section D.2 (except for cracks extending from the edges of the article to 25 % of the width of the article or 10 mm whichever is the lesser), and having a minimum thickness of 0.8 μm ;
- m — for micro-cracked chromium, having more than 250 cracks per centimetre in any direction forming a closed network over the whole of the significant surface when determined by one of the methods given in Annex D, and having a minimum thickness of 0.3 μm . (With some processes a substantially greater thickness, about 0.8 μm , will be required to achieve the necessary crack pattern.)

Example of complete classification number. A coating comprising 20 μm (minimum) copper plus 25 μm (minimum) bright nickel plus 0.3 μm (minimum) micro-cracked chromium has the classification number :

Fe/Cu20 Ni25b Cr m

* It will usually be possible to identify the type of nickel by microscopical examination of a polished and etched section of an article prepared according to Annex B.

** The sulphur 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 sulphur content of a nickel deposit on a coated article. An accurate determination is possible on a specially prepared test specimen.

6.4 Coatings appropriate to each service condition number

Table 1 shows the coating classification numbers appropriate for each service condition number.

TABLE 1 – Coatings on steel (or iron)

Service condition number	Classification number		
4	Fe/Cu20	Ni25b	Cr m
	Fe/Cu20	Ni30p	Cr r
	Fe/Cu20	Ni25p	Cr m
	Fe/Cu20	Ni30d	Cr r
	Fe/Cu20	Ni30d	Cr f
	Fe/Cu20	Ni25d	Cr m
3	Fe/Cu20	Ni30b	Cr r
	Fe/Cu20	Ni20b	Cr m
	Fe/Cu15	Ni25p	Cr r
	Fe/Cu15	Ni20p	Cr m
	Fe/Cu15	Ni25d	Cr r
	Fe/Cu15	Ni25d	Cr f
	Fe/Cu15	Ni20d	Cr m
2*	Fe/Cu20	Ni10b	Cr r
1*	Fe/Cu10	Ni5b	Cr r

* p or d nickel and f or m chromium may be substituted for b nickel and r chromium for service condition numbers 2 and 1.

6.5 Adhesion

Adhesion of the coating should be tested by one of the methods given in Annexes E and F. The coating should continue to adhere to the basis metal when subjected to the test selected.

6.6 Corrosion resistance

Coated articles should be subjected for the stated time to one of the corrosion tests shown in Table 4 appropriate for the particular service condition number. The tests are described in detail in Annexes G, H and J.

The corrosion tests indicated in Table 2 are a means of controlling the continuity and quality of the coatings and the duration of the tests does not necessarily have a fixed relationship with the service life of the finished article.

TABLE 2 — Tests appropriate for each service condition number

Service condition number	Duration (in hours) of corrosion test		
	CASS (see Annex G)	Corrodkote (see Annex H)	Acetic salt (see Annex J)
4	24	2 × 16	144
3	16	16	96
2	—	—	24
1	—	—	8

After the article has been subjected to the treatment described in the relevant test method, as give in Annex G, H or J, it should be examined by the procedure described in Annex K to determine whether it passes or fails the test.

Surface deterioration of the coating itself is expected to occur during tests on some types of coating; the extent to which surface deterioration can be tolerated should be subject to agreement between the supplier and the purchaser.

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ANNEX A

DUCTILITY TEST*

A.1 PREPARATION OF TEST PIECE

Prepare a plated test strip 150 mm long, 10 mm wide and 1 mm thick by the following method.

Polish a sheet of steel that is sufficiently large to allow the test strip to be cut from it after trimming off a border at least 25 mm wide all round. Plate the sheet on one side with nickel to a thickness of 25 μ m under the same conditions and in the same bath as the corresponding articles.

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

A.2 PROCEDURE

Bend the test strip with the plated side in tension, by steadily applied pressure, through 180° over a mandrel of diameter 11.5 mm until the two ends of the test strip are parallel. Ensure that contact between the test strip and the mandrel is maintained during bending.

A.3 ASSESSMENT

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. Small cracks at the edges do not signify failure.

* This test is used to check that the type of nickel deposit complies with the appropriate definition given in clause 6.3.2.

ANNEX B

DETERMINATION OF THICKNESS OF NICKEL AND COPPER OR BRASS MICROSCOPICAL METHOD

Use the method described in ISO Recommendation R 1463, *Measurement of metal and oxide coating thicknesses by microscopical examination of cross-sections*.

If etching is necessary, the following etchants are suitable :

- (1) equal parts by volume of nitric acid ($d = 1.42$) and glacial acetic acid;
 - (2) equal parts by volume of solutions of sodium cyanide (100 g/l) and sodium or ammonium persulphate (100 g/l).
- WARNING : Toxic fumes are evolved when solutions of these chemicals are mixed.

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ANNEX C

DETERMINATION OF CHROMIUM THICKNESS COULOMETRIC METHOD

C.1 PRINCIPLE

The method is based on the measurement of the quantity of electricity required to dissolve anodically an electrodeposited coating over a known area.

C.2 TEST SOLUTION

Orthophosphoric acid. Dilute 62 ml of orthophosphoric acid ($d = 1.75$) to 1 litre.

C.3 PROCEDURE

Clean the area to be tested with a cloth, wetted with an organic solvent for grease removal if necessary.

Press an electrolytic cell, fitted with a flexible sealing ring and incorporating an annular cathode, onto the coating so that a circle of known area is exposed to the test solution. Introduce the test solution into the cell; insert and operate the stirrer if appropriate to the instrument used and the thickness of the deposit.

Make the electrical connections with the specimen anodic. Continue electrolysis until dissolution of the chromium coating is complete, as indicated by a sudden change in anode potential, and record the quantity of electricity consumed.

Examine the specimen and ensure that chromium removal is complete over the area of the cell.

Calculate the thickness of the chromium coating from the following formula assuming 100 % current efficiency :

$$\text{thickness in micrometres} = \frac{Q}{A} \times 0.126$$

where

Q is the quantity of electricity consumed, in coulombs;

A is the area tested, in square centimetres.

ANNEX D

DETERMINATION OF CRACKS AND CRACK PATTERN IN CHROMIUM COATINGS

D.1 VISUAL EXAMINATION

Examine the surface for cracks in reflected light under an optical microscope at a magnification of 100X. For very fine crack patterns and accurate counting a higher magnification may be desirable. Use a micrometer eyepiece or similar device for indicating the distance over which cracks are counted.

According to the magnification used take a measured length such that at least 40 cracks are counted.

D.2 COPPER DEPOSITION

D.2.1 Principle

Electrodeposition of copper from an acid sulphate solution at low current density or low voltage occurs only on the underlying nickel that is exposed through cracks and other discontinuities.

This method provides a rapid means of visual inspection for uniformity of the crack pattern but, if counting of cracks is required, the optical aid described in section D.1 should be employed.

D.2.2 Procedure

The test is best applied immediately on completion of the plating process. If there is any delay, degrease the specimen thoroughly by chemical means, avoiding the use of any electrolytic treatment.

Deposit copper from a bath containing approximately 200 g/l of crystalline copper sulphate and 20 g/l of sulphuric acid at an average current density of 30 A/m². Use an immersion time of approximately 1 minute at room temperature.

NOTE. – Specimen and anode should be connected to the current supply before immersion.

In cases when the test is applied several days after chromium deposition, immerse the specimen in 10 to 20 g/l nitric acid solution for 4 minutes at approximately 95 °C before the copper deposition stage, to help reveal the crack pattern.

ANNEX E

FILE TEST FOR ADHESION

Saw off a piece of a plated article, hold it in a vice and apply a coarse file to the cut edge in such a manner as to try to raise the deposit. File in the direction from the basis metal to the coating at an angle of approximately 45° to the coated surface.

ANNEX F

QUENCHING TEST FOR ADHESION

Heat a plated article for 1 hour in an oven at $300 \pm 10^\circ\text{C}$. Then quench the article in water at room temperature.

CAUTION. This test may have an adverse effect on the mechanical properties of the article tested.

ANNEX G

CASS TEST

(Copper accelerated acetic acid salt spray test)

G.1 SPRAYING LIQUID

Dissolve 50 ± 5 g of sodium chloride* (NaCl) and 0.26 ± 0.02 g of cupric chloride (CuCl_2) in water containing less than 100 p.p.m. of total solids or having a conductivity of less than 0.002 S/m and dilute to 1 litre.

Add glacial acetic acid (CH_3COOH) to adjust the pH to 3.2 ± 0.1 .

G.2 TEST CONDITIONS

Operating temperature inside cabinet : 50 ± 1 °C.

Spray collection rate : 1.5 ± 0.5 ml/h over 8000 mm² horizontal area, averaged over at least 16 hours.

Collected spray : solution containing 50 ± 10 g sodium chloride per litre and having a pH of 3.2 ± 0.1 .

G.3 SUGGESTED APPARATUS

A fog cabinet or room should be made of or lined with a material resistant to the corrosive medium employed in this test (glass, rubber or certain plastics materials are suitable), and should be provided with specimen supports meeting the same requirements.

Provision should be made for a supply of clean air of controlled pressure and humidity, one or more spray nozzles, and means for heating the apparatus and maintaining the test temperature.

Baffles inside the cabinet should prevent direct impingement of the spray on the specimens. The use of adjustable baffles will be found of assistance in obtaining a uniform collection rate throughout the cabinet.

The air used to provide the spray should first be humidified by passing it through a saturator tower containing water** thermostatically maintained at the correct temperature. The temperature of the water in this tower will need to be several degrees higher than the test temperature, but should be adjusted according to the pressure used and the type of nozzle, so that both the collection rate and the concentration of collected fog are kept within the specified limits. It is desirable to maintain the level of the water in the saturator tower at a set height, and this is most conveniently done by an automatic level control.

It is also important to maintain a constant head between the nozzle or nozzles and the test solution in the supply tank. If this cannot be done manually throughout the cycle, an automatic level control should be fitted.

G.4 PROCEDURE

Immediately before testing, clean the specimens with a slurry of light magnesium oxide on a swab; rinse the specimens in clean running water so that they are free from water breaks.

* In a dry condition the sodium chloride (NaCl) should contain not more than 0.1 % of sodium iodide (NaI) and not more than 0.3 % of total impurities.

** The water used should contain less than 100 p.p.m. of solids, or have a conductivity of less than 0.002 S/m.

Place the specimens in the heated cabinet so that as far as possible their significant surfaces are at an angle of 15 to 30° to the vertical. Do not allow test solution from one specimen to drip onto any other. Place at least two collector vessels in the zone where the specimens are located, but take care that only fog, and no liquid falling from specimens or parts of the cabinet and its fittings, is collected.

Restore the cabinet and saturator tower to the required temperature and commence the test. Do not recirculate the spraying liquid.

After the test is completed, rinse the specimens in clean, running water to remove any deposits of salt, and dry. For each specimen make an assessment of the frequency of corrosion spots, based on the number of spots showing evidence of basis metal corrosion.

To examine any deterioration in appearance, swab the surface using a mild abrasive to remove all corrosion products, rinse in clean water, and dry.

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