INTERNATIONAL STANDARD

ISO 11408

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Chemical conversion coatings — Black oxide coating on iron and steel — Specification and test methods

Couches de conversion chimique — Finition noire de la fonte et de l'acier — Spécifications et méthodes d'essai

Reference number
ISO 11408:1999(E)

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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.

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.

International Standard ISO 11408 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, subcommittee SC 8, *Chemical conversion coatings*.

Annexes A and B of this International Standard are for information only.

Chemical conversion coatings — Black oxide coating on iron and steel — Specification and test methods

1 Scope

This International Standard specifies requirements for black oxide coatings on iron and steel (including cast and wrought iron, carbon steel, low alloyed steel and stainless steel). Black oxide coatings can be used to diminish friction between sliding or bearing surfaces or for decorative purposes or to reduce light reflection. Such coatings, with or without supplementary preservative treatment, may be used where a black surface is required. Only very limited corrosion protection is obtained under mildly corrosive conditions even with preservative treatment.

This International Standard does not specify requirements for the condition, finish or surface roughness of the substrate prior to black oxidizing.

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 listed 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 2064:1996, *Metallic and other inorganic coatings — Definitions and conventions concerning the measurement of thickness*.

ISO 4519, *Metallic and other inorganic coatings — Sampling procedures for inspection by attributes*.

ISO 9227, *Corrosion tests in artificial atmospheres — Salt spray tests*.

ISO 9587, *Metallic and other inorganic coatings — Pre-treatments of iron and steel to reduce the risk of hydrogen embrittlement*.

ISO 9588, *Metallic and other inorganic coatings — Post-coating treatments of iron and steel for reducing the risk of hydrogen embrittlement*.

3 Term and definition

For the purposes of this International Standard, the following term and definition apply.

3.1
significant surface
part of the article covered or to be covered by the coating and for which the coating is essential for serviceability and/or appearance and where the coating has to meet all of the specified requirements

[ISO 2064:1996, definition 3.1]

4 Information to be supplied by the purchaser to the supplier

4.1 Essential information

When ordering articles to be black oxide coated in accordance with this International Standard, the purchaser shall provide the supplier with the following information:

a) the number of this International Standard, i.e. ISO 11408;

b) the significant surface of the article to be coated, indicated, for example, on a drawing or by the provision of suitably marked samples;

c) the nature and surface condition (see clause 5) of the substrate;

d) the sampling procedure to be adopted (see clause 8);

e) the appearance of the coating, e.g. by the provision of a suitably marked sample (see 7.1).

4.2 Additional information

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

a) whether the neutral surface reaction has to be tested (see B.1);

b) whether porosity and continuity (gross defect) of the black oxide coating has to be tested (see B.2);

c) any requirements for supplementary preservative treatment (e.g. oil, wax or lacquer films) and humidity testing after such treatment;

d) any requirements for heat treatment before and/or after black oxidizing (see clause 6);

e) any requirements for oxalic acid resistance testing (see 7.2);

f) any requirements for wear resistance and its measurement;

g) any requirements for coefficient of friction and its measurement;

h) any requirements for resistance to neutral salt spray (see 7.3);

i) any requirements for adhesion with thicker black oxide coatings.

5 Substrate

The surface roughness of the coating will be dependent on the initial roughness of the substrate and this shall therefore not be cause for rejection of the black oxide coating.

6 Heat treatment of steels

6.1 General

Heat treatment may be required for certain steel grades to reduce the risk of cracking by hydrogen embrittlement or caustic brittleness.

CAUTION — High strength steel, of tensile strength $R_m \geqslant 1\,000$ MPa, may be subjected to caustic embrittlement that could lead to spontaneous cracking under internal or applied stress during the black oxidizing process.

6.2 Heat treatment before black oxidizing

Pre-treatment shall be carried out in accordance with ISO 9587. The heat treatment shall be carried out before the commencement of any preparation or cleaning treatment using aqueous solutions.

6.3 Heat treatment after black oxidizing

Post-coating treatment shall be carried out in accordance with ISO 9588. Surface-hardened parts shall be heat treated at a temperature between 190 °C and 220 °C for not less than 2 h.

7 Requirements

7.1 Appearance and surface quality

The coating shall be free of spots of red oxide and shall not be an overall reddish-brown colour. When a part is wiped with a clean Whatman 40 filter paper (or other filter paper of equivalent quality) prior to application of any supplementary preservative treatment, it shall produce no reddish-brown or green smut.

NOTE Non-uniformity of colour and haze is permissible on parts that are locally hardened, welded, cemented, riveted or submitted to other kinds of mechanical treatment.

7.2 Oxalic acid test resistance

7.2.1 When tested in accordance with 7.2.2 prior to the application of any supplementary preservative treatment, the black oxide coating on a part shall conform to Figure 3.

7.2.2 Dissolve 50 g oxalic acid in 1 l of distilled or de-ionized water. Place three drops (~ 0,2 ml) of the solution on a flat spot of the black oxide coated surface at ambient temperature. The reaction should occur after 30 s and within 8 min. After 8 min rinse and dry the surface and compare it to Figures 1 to 3.

7.3 Resistance of austenitic stainless steels to neutral salt spray

When tested using the salt spray test (NSS test) in accordance with ISO 9227, the significant surface, without any supplementary preservative coating, shall withstand 96 h without displaying traces of corrosion (rust).

8 Sampling

A random sample of the size required by ISO 4519 shall be selected from the inspection lot. The articles in the sample shall be inspected for conformance to this International Standard and the lot shall be classified as conforming or not conforming to each requirement according to the criteria of the sampling plans in ISO 4519.

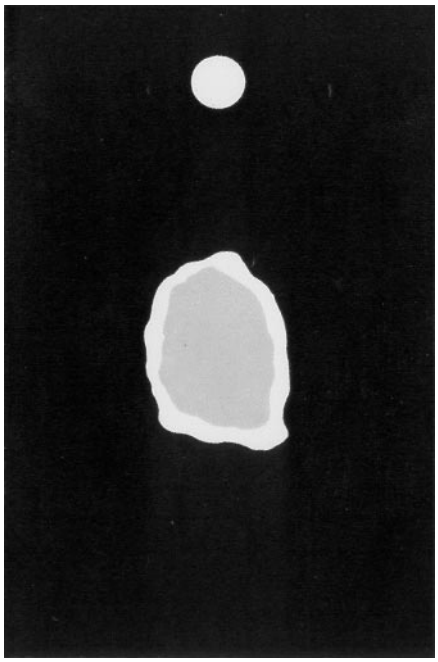

Figure 1 — Poor quality coating

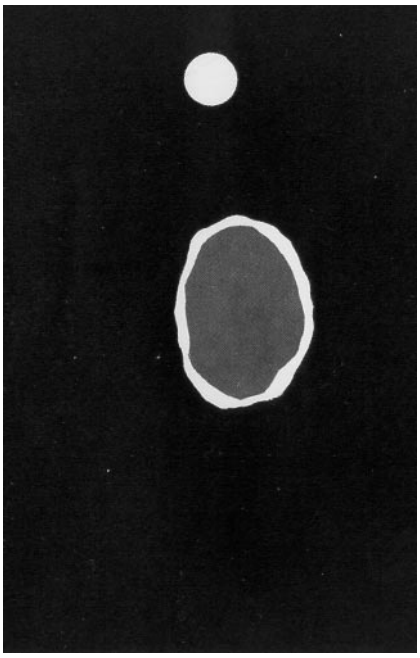

Figure 2 — Borderline quality coating

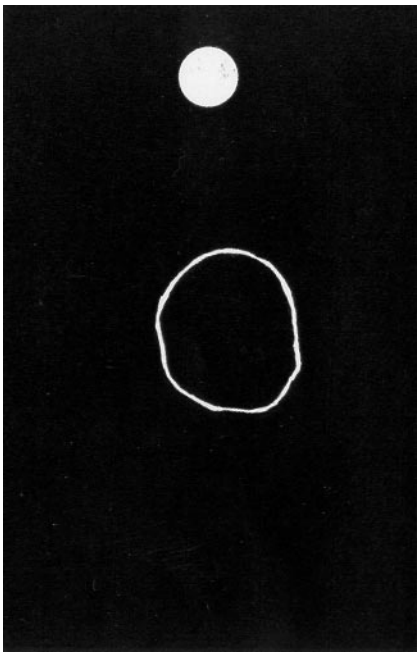

Figure 3 — Good quality coating

Annex A
(informative)

Processing procedures for iron and stainless steels

Table A.1 — Typical processing procedures for steels and stainless steels

Iron and steel grades	Process and suitable chemicals	Processing temperature °C	Immersion time min
Carbon steels, low alloy steels, wrought irons, cast irons	Alkaline oxidizing: NaOH, $NaNO_3$, water	Processing tank[a] boiling at 130 to 150	15 to 60
Martensitic stainless steel tempered at less than 480 °C	Alkaline-chromate: NaOH, $NaNO_3$, $Na_2Cr_2O_7$	115 to 125	30 to 45
Martensitic stainless steel tempered at 480 °C or higher	Fused salt; oxidizing: $Na_2Cr_2O_7$ and/or $K_2Cr_2O_7$	Molten salt bath 400 to 455	30
Stainless[b] steels (ferritic and austenitic)	Alkaline oxidizing		

[a] Subsequent tanks, if used, are made from the same chemicals at increased concentrations relative to the previous tank.

[b] Less than 8 % nickel.

Table A.2 — Different grades of stainless steels

Stainless steel grades	Nickel content %	Hardenability
Ferritic	–	Non-hardenable
Martensitic	⩽ 2,5	Hardenable
Ferritic-austenitic	4,5 to 7	Non-hardenable
Austenitic	7 to 26	Non-hardenable

Annex B
(informative)

Methods of test for neutral surface reaction and for porosity and continuity

B.1 Neutral surface reaction

The test shall be carried out after the last rinsing and drying, but before the oiling. Phenolphthalein test solution or phenolphthalein indicator paper shall be used as follows.

a) Prepare phenolphthalein test solution by dissolving 2 g of phenolphthalein in 100 ml of ethanol and keep the solution in a glass dropping bottle. Place 1 to 2 drops of the test solution on the surface to be tested (joints, cavities).

b) Moisten the phenolphthalein indicator paper with distilled water and place this paper on the surface to be tested.

Residual alkalis shall be deemed to be present if the phenolphthalein drops or indicator paper (as appropriate) turn a rose colour.

B.2 Porosity and continuity of the black oxide coating

The test shall be conducted by immersion (a) or using droplets (b).

Ensure that each test uses a fresh test solution prepared from laboratory reagent grade $CuSO_4$ at a mass fraction of 3 %.

a) Immerse the specimen in the test solution (in a glass or plastic bottle) at a temperature of 15 °C to 20 °C. After 30 s remove the specimen, rinse it using cold water and dry it using filter paper.

b) Place three drops of the test solution on the surface to be tested. After 30 s remove the drops using filter paper.

Examine the surface under normal corrected vision. Red points or spots shall be deemed to indicate the presence of pores or that the coating is damaged.

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