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Zinc diffusion coatings on ferrous products — Sherardizing — Specification

Revêtements par diffusion de zinc sur les produits ferreux — Shérardisation — Spécification

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information.

The committee responsible for this document is ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 4, *Hot dip coatings (galvanized, etc.)*.

Introduction

Sherardizing is a thermal diffusion coating process in which ferrous articles are heated in the presence of a sherardizing mixture consisting of zinc dust with or without an inert material.

The process is commonly performed in closed, slowly rotating or fixed containers at temperatures ranging from around 300 °C to 500 °C. The normal processing temperature is below the melting point of zinc (419 °C).

During the process, zinc reacts with the surface to form inter-metallic layers on ferrous articles.

A coating thickness of 10 µm to 75 µm (and higher if required) can be achieved. The coating thickness is accurately controlled by the amount of zinc dust, processing time and temperature. The coating closely follows the contours of the base material and uniform coating thicknesses are produced on articles, including those of irregular shape.

After sherardizing, the container load is cooled down. A screening process separates the sherardized articles from the unused sherardizing mixture. The articles, with the zinc-iron inter-metallic layers, are eventually post-treated (by phosphating, chromating or another suitable passivation process) resulting in a clean and passivated surface.

It is common to use articles coated with zinc-iron inter-metallic layers as a primer or base-coat for duplex-systems.

For additional information about the sherardizing process and the application possibilities of sherardized articles, see Reference [12] and Reference [13].

Sherardizing (thermal diffusion coating) is also known as the following:

— diffusion zinc plating (Germany);

— thermal diffusion coating (Russia);

— thermal diffusion galvanizing (Ukraine);

— vapour galvanizing (UK);

— zinc diffusion coating (USA);

— zinc inter-metallic coating (Russia);

— zinc thermo diffusion galvanizing (Israel).

In China, Europe and the USA, the common name for the thermal diffusion coating process is sherardizing.

Zinc diffusion coatings on ferrous products — Sherardizing — Specification

1 Scope

This International Standard specifies minimum thickness requirements for six classes of zinc diffusion layers applied to ferrous products by the sherardizing process for the purpose of protection against corrosion and wear.

This International Standard does not specify any requirements for the surface condition (finish or roughness) of the basis material before sherardizing.

Post-treatments (conversion coatings), after-treatments or organic over-coatings (Duplex) of sherardized articles are not in the scope of this International Standard.

NOTE 1 For general information about post-treatments, see Annex C and Annex D.

This International Standard does not apply to sherardized products (e.g. fasteners, tubes) for which specific standards exist and which might include additional requirements or requirements which are different from those of this International Standard.

NOTE 2 Individual product standards can incorporate this International Standard for the coating by quoting its number, or can incorporate it with modification specific to the product.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1460, *Metallic coatings — Hot dip galvanized coatings on ferrous materials — Gravimetric determination of the mass per unit area*

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

ISO 2178, *Non-magnetic coatings on magnetic substrates — Measurement of coating thickness — Magnetic method*

ISO 2808, *Paints and varnishes — Determination of film thickness*

ISO 10474, *Steel and steel products — Inspection documents*

3 Terms and definitions

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

3.1
sherardizing process
zinc diffusion coating process in which articles are heated in close contact with a *sherardizing mixture* (3.3), commonly performed in a closed slowly rotating container or a fixed (non-rotating) container, to form *sherardized layers* (3.2)

3.2
sherardized layer
zinc diffusion layer
zinc diffusion coating consisting of zinc-iron alloy layers obtained by sherardizing

Note 1 to entry: The "sherardized layer" is referred to in this International Standard as "coating".

Note 2 to entry: The sherardized layer may subsequently be post-treated by phosphating, chromating or another suitable passivation process (guidance for these post-treatments are given in Annex C and Annex D).

3.3
sherardizing mixture
mixture consisting of mainly zinc dust, with or without other process-supporting ingredients

Note 1 to entry: Zinc dust is also known as zinc powder.

3.4
mass of the zinc diffusion layer
total mass of zinc/iron alloys per unit area of surface

Note 1 to entry: The mass of the zinc diffusion layer is expressed in grams per square metre (g/m^2).

3.5
thickness of the zinc diffusion layer
total zinc diffusion layer thickness consisting of zinc/iron alloys

Note 1 to entry: The thickness of the zinc diffusion layer is expressed in micrometre (μm).

3.6
significant surface
part of the article covered or to be covered by the *zinc diffusion layer* (3.2) and for which these coating is essential for serviceability and/or appearance and where the layer will meet all the specified requirements

3.7
control sample
article, or group of articles, from a lot which is selected for testing

3.8
reference area
area within which a specified number of single measurements is required to be made

3.9
local thickness of the zinc diffusion layer
mean value of zinc diffusion layer thickness obtained from the specific number of measurements within a *reference area* (3.8) for a magnetic or electro-magnetic test or the single value of a gravimetric test

Note 1 to entry: Guidance for the methods of measurement of the zinc diffusion layer thickness is given in 6.2.2 and Annex B.

3.10
mean thickness of the zinc diffusion layer
average value of the local zinc diffusion layer thicknesses of different *reference areas* (3.8)

Note 1 to entry: In case there is only one *reference area* (3.8), the mean zinc diffusion layer thickness is the same as the local zinc diffusion layer thickness.

3.11
inspection lot
one or more articles of the same type and size comprising either a single order, a single delivery load or the number of articles identified as a lot by the sherardizer

3.12
acceptance inspection
inspection of an *inspection lot* (3.11) at the sherardizer's works (unless otherwise specified)

4 General requirements

4.1 Surface condition base material

The surface of the base material should be clean before sherardizing.

Surface contamination that cannot be removed by grit blasting should be removed prior to the mechanical pre-treatment process. The responsibility of removing the contamination should be agreed upon between the sherardizer and purchaser.

The surface condition of the base material, the mass of the parts and the sherardizing conditions can affect the appearance, the thickness, surface roughness and the physical and mechanical properties of the coating. This International Standard does not define any requirements regarding these properties.

NOTE Guidance on these parameters can be found in ISO 14713-3:2009, Clause 4.

4.2 Information to be supplied by the purchaser

Information shall be supplied by the purchaser in accordance with A.1 and A.2.

5 Acceptance inspection and sampling

Acceptance inspection shall be undertaken before the products leave the sherardizer's custody, unless otherwise specified at the time of ordering by the purchaser.

Acceptance inspection involves the assessment of appearance of the coated article and testing the thickness. The results of other tests of the coated article are normally not provided. Other tests will only be carried out by agreement between purchaser and sherardizer made at the time of ordering.

A control sample for thickness testing shall be taken randomly from each inspection lot selected for testing. The minimum number of articles to form the control sample shall be taken in accordance with Table 1.

Alternatively, sampling procedures selected from ISO 2859-1, ISO 2859-2, and ISO 2859-3 can be used.

Table 1 — Control sample size related to batch size

Number of articles in the batch	Minimum number of articles in the control sample
1 to 3	All
4 to 500	3
501 to 1 200	5
1 201 to 3 200	8
3 201 to 10 000	13
Above 10 000	20

6 Coating properties

6.1 Appearance

The surface of the coating has a grey (matt or lustrous) appearance and might show scratches resulting from normal contact with other articles, during processing or storage. Such scratches are superficial and not detrimental to the corrosion resistance of the sherardized articles.

The coating shows a certain surface roughness which is characteristic for the zinc-iron alloy type of coating.

NOTE It is noted that "grey (matt or lustrous) appearance" and "surface roughness" are relative terms. These appearance properties can be influenced by the composition of and surface conditions of the base material. It is not possible to establish a definition of appearance and finish of the coating covering all requirements in practice.

Areas without a zinc diffusion layer shall not be allowed, unless otherwise specified at the time of ordering by the purchaser and agreed upon with the sherardizer.

Rejected articles, regarding these areas, shall be re-sherardized and resubmitted for inspection in agreement with the purchaser.

The development of (grey-) white corrosion products (white staining), the formation of mainly basic zinc oxide during storage in humid conditions after sherardizing, shall not be cause for rejection.

It is not permitted for the sherardized articles to have an orange-brown colouring at the time of delivery, unless otherwise specified at the time of ordering by the purchaser and agreed upon with the sherardizer.

Rejected articles, regarding this discolouring, shall be re-sherardized and resubmitted for inspection in agreement with the purchaser.

6.2 Thickness

6.2.1 General

Coatings applied by sherardizing are designed to protect ferrous products against corrosion and wear. The service life of such coatings in a given environment is approximately proportional to the coating thickness (see ISO 14713-1).

6.2.2 Test methods

Thickness measurements shall only be made on clean, washed surfaces. In case cleaning and/or passivation is not part of the coating process, samples for thickness testing shall be cleaned separately.

The local coating thickness shall be determined in accordance with the magnetic method given in ISO 2178 or the electro-magnetic method given in ISO 2808, also described in ISO 3882.

These two methods are generally most appropriate for routine quality control within works.

The gravimetric method according to ISO 1460 can be used to determine the coating mass. The coating thickness can be calculated by dividing the coating mass per unit area by the nominal coating density of the coating 7,2 g/cm^3 (see B.2).

If a sufficient number of measurements is made within the reference area (see 6.2.3), effectively the same coating thickness will be determined by the magnetic method, the electro-magnetic method as the gravimetric method.

For articles with a complex shape, in which no suitable reference area can be determined for measuring the thickness by the magnetic method, the electro-magnetic method, the gravimetric method shall be used.

The microscopic cross-section method according to ISO 1463 is less appropriate for routine use because it is destructive and relates only to a single line. However, this method can be used for special quality control operations for articles with a complex shape, agreed upon by the purchaser and the sherardizer at the time of ordering.

NOTE Annex B provides additional information and guidance on the measurement of thickness.

6.2.3 Reference areas

The number and position of reference areas and their sizes for the magnetic, electro-magnetic, micrometre method or gravimetric test shall be chosen with regard to the shape and size of the article(s) in order to obtain a result as representative as possible of mean coating thickness or mass per unit area as applicable.

The reference areas shall be within the significant surfaces, unless otherwise agreed upon between purchaser and sherardizer.

For articles with a significant surface area greater than or equal to 10 cm^2, there shall be at least one reference area on each article in the control sample. Control sample sizes are given in Table 1.

For articles with a significant surface area of less than 10 cm^2, there shall be enough articles grouped together to provide at least 10 cm^2 significant surface for one reference area. Hence, the total number of articles tested shall equal the number of articles required to provide one reference area multiplied by the total number of articles in a control sample, in accordance with Table 1 (or the total number of articles sherardized, if that is less).

Alternatively, sampling procedures selected from ISO 2859-1, ISO 2859-2, and ISO 2859-3 can be used.

6.2.4 Magnetic method or electro-magnetic method

A minimum of five magnetic or electro-magnetic test readings shall be taken within each reference area. Because the area over which each measurement is made in this method is very small, individual figures can be lower (typically up to 15 %) than the values for the local thickness. This is irrelevant as only the average value over the whole of each reference area is required to be equal to, or greater than, the local coating thickness minimum value.

When more than five articles have to be taken to make up a reference area of at least 10 cm^2, a single magnetic measurement shall be taken on each article if a suitable area of significant surface exists. If such a suitable area does not exist, the gravimetric method according to ISO 1460 shall be used.

6.2.5 Gravimetric method

The mass of the coating per unit area, expressed in g/cm^2, shall be determined according to ISO 1460. This mass value can be converted to a thickness value in µm by dividing the nominal density of the coating 7,2 g/cm^3.

NOTE See B.2

6.2.6 Thickness requirements

The thickness requirements for six classes are given in Table 2.

Table 2 — Coating thickness

Class of coating	Local coating thickness minimum µm	Local coating mass minimum g/m^2
Class 10	10	72
Class 15	15	108
Class 30	30	216
Class 45	45	324
Class 60	60	432
Class 75	75	540
NOTE For typical application fields of the coating with different coating thicknesses, see C.7.		

6.3 Acceptance criteria

When tested in accordance with 6.2.2, for the appropriate number of reference areas given in 6.2.3, the local coating thickness shall be not less than the values given in Table 2.

If the local coating thickness on a control sample does not conform to Table 2, twice the original number of articles (or all the articles, if that is the lower number) shall be taken from the batch and tested. If this larger control sample conforms to Table 2, the batch shall be deemed to conform. Failure of the larger control sample to meet the thickness requirements shall constitute grounds for rejection of the batch. If the batch is rejected, individual articles may be submitted for re-testing.

6.4 Additional clearances for threaded components

Although sherardizing gives a uniform coating without any significant changes in the profile of threads, there shall be adequate clearance between external and internal threads before sherardizing. The purchaser shall discuss the additional thread clearance requirement with the sherardizer, see A.2 e).

Guidance on the adequate clearances can be found in ISO 14713-3.

7 Ceritificate of compliance

When required by the purchaser, the sherardizer shall provide a certificate of compliance with the requirements of this International Standard according to ISO 10474. A request for such a certificate shall be made at the time of ordering, see A.2 i).

Annex A
(normative)

Information to be supplied by the purchaser to the sherardizer

A.1 Essential information

The purchaser shall provide the following information on all relevant documents:

a) number of this International Standard, i.e. ISO 17668;

b) class of coating or, alternatively, the minimum coating thickness required (see Table 2).

A.2 Additional information

Where the following information is required for particular purposes by the sherardizer, it shall be specified by the purchaser at the time of ordering:

a) type of material to be sherardized;

b) any likely effects on the metallurgical properties of the basis material caused by heating temperatures of up to 500 °C, and if the parts are heat-treated;

c) surface area and weight of the articles to be sherardized, in case different articles are part of the same order the surface area and weight from each group of articles of the same kind shall be provided;

d) identification of significant surfaces and reference areas, for example, by drawings or by the provision of suitably marked samples;

e) thread clearances, depending on the class of coating thickness specified, to be stated on the product drawing or on the order document;

f) any special pre-treatment requirements;

g) any post-treatments, after-treatments or over-coating to be applied to the sherardized coating (see C.7);

h) any requirements for inspection;

i) whether a certificate of compliance is required in accordance with ISO 10474.

Annex B
(informative)

Determination of thickness

B.1 General

The most general non-destructive methods of determining coating thickness are the magnetic method according to ISO 2178 and the electro-magnetic method according to ISO 2808. These two methods are commonly used for in-works quality control procedures.

Destructive methods include the gravimetric method according to ISO 1460, determination of mass per area converted to thickness (see B.2) and microscopic cross-section method (see B.3).

Careful consideration should be made of the relationship between local and average thickness where the magnetic method or the electro-magnetic method is used and the results are compared with those obtained by the gravimetric method. If a sufficient number of measurements is made within a reference area, effectively the same coating thickness will be determined by the magnetic method, electro-magnetic method as by the gravimetric method.

Another non-destructive method, which can be used for routine quality control purposes, is measuring the thickness of an article, before and after applying the coating, with a precision micrometre device (gauge). The local coating thickness is half of the increase in thickness of the article after applying the coating. This method, however, is not standardized. This method is less precise, but the accuracy increases with the coating thickness. The method is useful in case the magnetic method or the electro-magnetic method cannot be applied, such upon agreement between purchaser and sherardizer. This method can successfully be used for small products such as rivets and washers.

B.2 Calculation of thickness from mass per unit area

The gravimetric method according to ISO 1460 provides a result for the coating mass per unit area expressed in g/m^2. This value can be converted to local thickness in micrometre by dividing by the nominal density of the coating 7,2 g/cm^3.

The mean thickness L is given by Formula (B.1):

$$L = \Delta M \Big/ \left(\rho \cdot A \right) \tag{B.1}$$

where

L is mean thickness of the coating, in µm;

ΔM is the weight difference between a coated and an uncoated part, in g;

A is the surface area of the part, in m^2;

ρ is the nominal density of the coating, 7,2 g/cm^3.

NOTE 7,2 g/cm^3 is equal to 7,2 g/(µm × m^2)

For equivalent values of coating thickness and coating mass, see Table 2.

B.3 Microscopic cross section method

The microscopic cross section method according to ISO 1463 can be used for special quality control procedures for articles with a complex shape with recesses or with hollow parts, such as tubes, agreed upon between purchaser and sherardizer at the time of ordering. Careful attention is required to prepare the cross-section and a skilled personnel is required to analyse the results of the test procedure. The method can also be used for analysing the structure of the zinc-iron alloy layers. The microscopic cross-section method is less appropriate for routine quality control, because it is a destructive method and relates only to a single line.

Annex C
(informative)

General information

C.1 Sherardizing process

Sherardizing is a thermal diffusion coating process which forms zinc-iron diffusion layers on ferrous materials. The articles are heated in the presence of a sherardizing mixture.

The coating closely follows the contours of the base material, and uniform coatings are produced on articles, including those of complex shape.

The coating may subsequently be post-treated by phosphating, chromating or other suitable passivation processes, resulting in a clean passivated surface.

C.2 Base material

Unalloyed carbon steels, low alloy steels, heat treated steels, high tensile steels, sintered material, malleable grey and cast iron are suitable for sherardizing. The influence on mechanical properties of heat treated steel articles is considerably low.

The process does not give rise to hydrogen embrittlement. However, when pre-cleaning high tensile material (above 1 000 N/mm^2), cathodic cleaning should not be employed. Pre-treatment by anodic, mechanical or any other processes which do not affect the base material are recommended. The surface should be free from any contamination prior to processing.

It is essential that parts having soft-soldered or resin bonded joints are not sent for sherardizing as joints of this nature are affected by the process. Any such jointing should be carried out after sherardizing has taken place.

C.3 Post-treatments

Post-treatments (passivation), after-treatments or organic over-coatings (Duplex) of sherardized articles are not in the scope of this International Standard. The service life can be significantly extended by these processes (see Annex D).

The adhesion of paints and organic overcoats can be improved by a post-treatment.

C.4 After-treatments

After-treatments by oils, stains, sealants or special lubricants can subsequently be applied to the coating or to the passivated coating to enhance the corrosion resistance, to provide colour or to regulate the coefficient of friction of the coating.

Overcoats such as paints or organic coatings can be supplied to the eventual passivated zinc diffusion coating to increase corrosion resistance and/or decorative appearance.

Guidance on these after-treatments can be found in ISO 14713-3 and ISO 12944-5.

C.5 Appearance

The sherardized coating has a grey (matt or lustrous) appearance and may show scratches resulting from normal contact with other articles, such scratches are superficial and not detrimental to its corrosion resistance.

The occurrence of darker and lighter areas is no reason for rejection.

White staining, the formation of mainly basic zinc oxide during storage in humid conditions after sherardizing, is no reason for rejection.

Orange-brown discolouring at the time of delivery is not allowed, unless agreed upon between the sherardizer and the purchaser.

C.6 Adhesion

The adhesion between the coating and the basis metal generally does not need to be tested as good bonding is characteristic of the forming of zinc-iron alloy layers by the sherardizing process. The coating is made by thermal diffusion of zinc into/on the surface of the iron and steel articles forming typical zinc-iron alloy layers.

The coated articles should be able to withstand, without peeling or flaking, handling consistent with the nature and thickness of the coating and the normal use of the article.

C.7 Coating thickness

Class 10 coatings can be specified for the application as a base coat (primer) for a paint or an organic coating, Class 10 coatings have only a limited cathodic protection and are not recommended for corrosion protection without an additional after-treatment.

Class 15 coatings can be specified for normal indoor and outdoor environments.

Class 30 coatings should be specified for outdoor applications in more severe environments, or where there is a requirement for extended service life.

Class 45 coatings should be specified for use in highly corrosive and or abrasive environments, e.g. industrial or marine.

Class 60 and Class 75 coatings can be required for special applications. When thicker coatings are specified, there can be a requirement for a specific pre-treatment.

Guidance for the coating thickness of zinc coated products for use in environments with different corrosion categories is given in ISO 14713-1:2009, Table 1.

Where doubt exists concerning the coating thickness required and/or the requirement of a post treatment, the purchaser should seek the advice of the sherardizer.

C.8 Additional clearances for threaded components

Threaded components need additional clearance depending on the coating thickness. Guidance on this subject can be found in ISO 14713-3:2009, Table 1 and Reference [12].

Annex D
(informative)

Corrosion resistance of sherardized layers

D.1 General information

Coatings applied by the sherardizing process are designed to protect the base material against corrosion and abrasion.

For most applications, the sherardized steel articles are exposed to atmospheric conditions. Under these conditions, the service life of the coating is proportional to the thickness of the coating. See ISO 14713-1.

Reference should be made to ISO 9223 for determination of corrosion category for an external exposure environment. Guidance on the likely performance of the coating in a particular atmospheric exposure environment can be found in ISO 14713-1.

Salt spray tests cannot be used to accurately test zinc diffusion coated (sherardized) steel because they accelerate the wrong failure mechanism. Without a proper wet/dry cycle, the zinc coating cannot form patina layers. The absence of a patina layer allows constant attack of the zinc metal and gives a very low prediction of the coating life. See ISO 14713-1:2009, Clause 8. This test procedure can only be used for in-house production control in a comparative way.

D.2 Post treatment and passivation

The service life of the coating can be extended significantly by a post treatment of the coating by a passivation process, e.g. phosphating, chromating or other suitable passivation process developed for treating zinc-iron alloys (conversion coating).

The significance of the post-treatment (passivation) can be tested and compared by an accelerated corrosion test. However, the result of this test cannot give an absolute indication of the total coating life of the surface.

D.3 Duplex systems

The service life of painted or powder coated sherardized articles (Duplex system) can be extremely long.

For the application of duplex systems involving the use of paints, information about requirements on surface treatment, painting systems, coating thickness etc., can be found in ISO 12944-5.

For the application of duplex systems involving the use of powder coatings, recommendations on coating powders, pre-treatment, application and system performance can be found in EN 13438 and EN 15773.

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[4] EN 15773, *Industrial application of powder organic coatings to hot dip galvanized or sherardized steel articles [duplex systems] — Specifications, recommendations and guidelines*

[5] ISO 12944-5, *Paints and varnishes — Corrosion protection of steel structures by protective paint systems — Part 5: Protective paint systems*

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