
**Metallic and other inorganic coatings —
Post-coating treatments of iron or steel to
reduce the risk of hydrogen embrittlement**

*Revêtements métalliques et autres revêtements inorganiques —
Traitements après revêtement sur fer ou acier pour diminuer le risque
de fragilisation par l'hydrogène*

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Foreword

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International Standard ISO 9588 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 3, *Electrodeposited coatings and related finishes*.

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Introduction

When atomic hydrogen enters steels and certain other metals, for example aluminium and titanium alloys, it can cause loss of ductility or load-carrying ability or cracking (usually as submicroscopic cracks), or catastrophic brittle failures at applied stresses well below the yield strength or even the normal design strength for the alloys. This phenomenon often occurs in alloys that show no significant loss in ductility, when measured by conventional tensile tests, and is frequently referred to as hydrogen induced delayed brittle failure, hydrogen stress cracking or hydrogen embrittlement. The hydrogen can be introduced during, cleaning, pickling, phosphating, electroplating and autocatalytic processes, and in service, as a result of cathodic protection or corrosion reactions. Hydrogen can also be introduced during fabrication, for example during roll forming, machining, and drilling, due to the breakdown of unsuitable lubricants, as well as during welding or brazing operations.

The susceptibility to hydrogen embrittlement resulting from the absorption of atomic hydrogen and/or the tensile stresses induced during fabrication and subsequent surface finishing processes can be reduced by heat treatment. The time-temperature relationship of the heat treatment is dependent on the composition and structure of steels, as well as on the specific coatings being applied and the nature of the coating procedures. For most high strength steels, the effectiveness of the heat treatment falls off rapidly with reduction of time and temperature.

This International Standard is intended for use by purchasers in specifying requirements to the electroplater, supplier or processor and should be indicated on the part drawing or purchase order.

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1 Scope

This International Standard establishes procedures for reducing susceptibility or degree of susceptibility to hydrogen embrittlement that can arise in surface finishing processes.

The heat treatment procedures established in this International Standard have been shown to be effective in reducing the susceptibility to hydrogen embrittlement. These heat-treatment procedures are used after surface finishing, but prior to any secondary conversion coating operation.

Stress-relief heat treatment procedures applied after fabrication, but prior to surface finishing, are specified in ISO 9587.

This International Standard does not apply to fasteners.

NOTE The heat treatment does not guarantee complete freedom from the adverse effects of hydrogen embrittlement.

2 Normative references

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

ISO 2080, *Electroplating and related processes — Vocabulary*.

ISO 9587:1998, *Metallic and other inorganic coatings — Pretreatment of iron or steel to reduce the risk of hydrogen embrittlement*.

3 Terms and definitions

For the purposes of this International Standard the terms and definitions given in ISO 2080 and the following apply.

3.1

embrittlement relief heat treatment

thermal process carried out over a temperature range and for a duration of time such that no alteration of metallurgical structures, such as recrystallization, of the basis metal occurs, but at which embrittlement relief of the plated articles is achieved

4 Requirements

Heat treatment shall be performed on coated metals in order to reduce the risk of hydrogen embrittlement. In all cases, the heat treatment shall be deemed to commence at the time at which the whole of each article attains the specified temperature.

Articles made from steel with actual tensile strengths greater than or equal to 1 000 MPa (with corresponding hardness values of 300 HV 10, 303 HB, or 31 HRC) and surface-hardened parts shall require heat treatment unless class ER-0 is specified. Preparation involving cathodic treatments in alkaline or acid solutions shall be avoided. Additionally, the selection of electroplating solutions with high cathodic efficiencies is recommended for steel components with tensile strengths greater than 1 400 MPa (with corresponding hardness values of 425 HV 10, 401 HB, or 43 HRC).

Table 1 lists the embrittlement-relief heat-treatment classes from which the purchaser may specify the treatment required, to the electroplater, supplier or processor either on the part drawing or on the purchase order. When no embrittlement-relief treatment class is specified by the purchaser, then class ER-1 shall be applied.

NOTE 1 The treatment class selected is based upon experience with the part or similar parts, and the specific alloy used, or with empirical test data. Some parts, because of factors such as alloy composition and structure, trap-population density, size, mass or design parameters might perform satisfactorily with no embrittlement-relief treatment. Therefore, the class ER-0 treatment is provided for parts that the purchaser wishes to exempt from treatment.

NOTE 2 Class ER-1, one of the longest treatments, is the default when the purchaser does not specify a class. The electroplater, supplier or processor is not normally in possession of the necessary information, such as design considerations, induced stresses from manufacturing operations, etc., that have to be considered in selecting the correct stress relief treatment. It is in the purchaser's interest that their part designer, manufacturing engineer or other technically qualified individual specify the treatment class on the part drawing or purchase order in order to avoid the extra cost of the default treatment.

NOTE 3 The use of inhibitors in acid pickling baths does not necessarily guarantee that hydrogen embrittlement is minimized.

5 Embrittlement relief treatment classes

5.1 With the exception of surface-hardened parts, the heat treatment conditions shall be selected on the basis of actual tensile strength. When only the minimum tensile strength is specified or if the tensile strength is not known, the heat treatment condition shall be selected by relating known or measured hardness values to equivalent tensile strengths. The tensile strength, or equivalent derived from known or measured hardness values, shall be supplied by the purchaser.

Steels that have been wholly or partly surface-hardened shall be considered as being in the category appropriate to the hardness of the surface-hardened layer.

5.2 If the purchaser requires any tests to be performed to verify adequate embrittlement relief treatment, then the test method and the sampling plan to be used shall be specified.

6 Heat treatment after processing

6.1 The heat treatment shall commence as soon as possible, preferably within 1 h but not later than 3 h after surface finishing and before commencement of any grinding or other mechanical operation. For cadmium, tin, zinc, their alloys or any other coating receiving a chromate treatment, heat treatment shall be carried out before chromate treatment, with the exception of electrodeposited zinc-cobalt alloys that should be passivated prior to hydrogen embrittlement relief heat treatment.

NOTE 1 Chromate coatings undergo change at temperatures above 66 °C. The coating changes from an amorphous structure to a crystalline structure and no longer exhibits 'self-healing' properties. Although the crystallized chromate coating will provide satisfactory corrosion protection under most natural environments, the chromate coating will no longer pass accelerated corrosion tests.

NOTE 2 The time period referred to in clause 6 is the time between the end of the plating operation and the loading of the article concerned into the heat treatment processor.

6.2 For high strength steels, the conditions given in Table 1 and Figure 1 shall apply. For steels of actual tensile strength less than 1 000 MPa, heat treatment after plating is not essential.

6.3 If threads or sharp notches exist or the articles are greater than 25 mm thick, then for articles electroplated with cadmium or zinc, heat treatment shall be carried out immediately after electroplating for a minimum period of 24 h.

6.4 The minimum duration of heat treatment for steels of actual tensile strength above 1 800 MPa may be selected in accordance with Figure 1, i. e.,

$$t = 0,02R_m - 12$$

where

t is the minimum duration, in hours;

R_m is the actual tensile strength, expressed in megapascals.

6.5 Electroplated, autocatalytic or phosphate coated steel articles having surface-hardened areas and through-hardened or bearing steels, that would suffer an unacceptable reduction in hardness by treatment in accordance with Table 1 and Figure 1 shall be heat treated at a lower temperature, but at not less than 130 °C for a minimum period of 8 h. This heat treatment is applicable for articles made of steel with actual tensile strength below 1 400 MPa. For articles electroplated with cadmium, tin, zinc or their alloys, the minimum period shall be 16 h for those with a tensile strength below 1 400 MPa, and 22 h for articles with a tensile strength in the range 1 400 MPa to 1 800 MPa.

NOTE Lower temperature treatment may adversely affect the fatigue strength of the article.

6.6 Treatment at 440 °C to 480 °C reduces the hardness of chromium deposits. It shall not be applied to steels that can be adversely affected by heat treatment at this temperature. For such steels, the lower temperature range, i.e. 190 °C to 220 °C, shall be applied. For tempered steels, the article shall not be heat treated above a temperature that shall be 50 °C below the tempering temperature.

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Table 1 — Classes of embrittlement-relief heat treatment for high strength steels
(see clauses 4, 5 and 6 for details)

| Class | Steels of tensile strength R_m MPa | Temperature °C | Minimum time (see clause 4) h |
|--------------------|---|-------------------|-------------------------------------|
| ER-0 | Not applicable (see clause 4, note 1) | | |
| ER-1 | $1\ 701 \leq R_m \leq 1\ 800$ | 190 to 220 | 22 |
| ER-2 | $1\ 601 \leq R_m \leq 1\ 700$ | 190 to 220 | 20 |
| ER-3 | $1\ 501 \leq R_m \leq 1\ 600$ | 190 to 220 | 18 |
| ER-4 | $1\ 401 \leq R_m \leq 1\ 500$ | 190 to 220 | 16 |
| ER-5 | $1\ 301 \leq R_m \leq 1\ 400$ | 190 to 220 | 14 |
| ER-6 | $1\ 201 \leq R_m \leq 1\ 300$ | 190 to 220 | 12 |
| ER-7 ^a | $R_m \geq 1\ 525$ | 177 to 205 | 12 |
| ER-8 | $1\ 101 \leq R_m \leq 1\ 200$ | 190 to 220 | 10 |
| ER-9 | $1\ 000 \leq R_m \leq 1\ 100$ | 190 to 220 | 8 |
| ER-10 ^a | $1\ 250 \leq R_m \leq 1\ 525$ | 177 to 205 | 8 |
| ER-11 ^a | $1\ 450 \leq R_m \leq 1\ 800$ | 190 to 220 | 6 |
| ER-12 ^a | $1\ 000 \leq R_m \leq 1\ 500$ | 177 to 205 | 4 |
| ER-13 | $1\ 000 \leq R_m \leq 1\ 800$ unpeened items and for engineering chromium electroplated articles | 440 to 480 | 1 |
| ER-14 ^a | Surface hardened articles $R_m < 1\ 401$ | 130 to 160 | 8 |
| ER-15 ^a | Surface hardened articles $1\ 401 \leq R_m \leq 1\ 800$ electroplated with cadmium, tin, zinc or their alloys | 130 to 160 | 8 |
| ER-16 | Surface hardened articles $R_m < 1\ 400$ electroplated with cadmium, tin, zinc or their alloys | 130 to 160 | 16 |
| ER-17 | Parts > 25 mm thick and articles with threads or sharp notches | 190 to 220 | 24 |

^a Traditional treatments referred to in some national standards.