INTERNATIONAL STANDARD

ISO 10587

First edition
2000-02-15

Metallic and other inorganic coatings — Test for residual embrittlement in both metallic-coated and uncoated externally-threaded articles and rods — Inclined wedge method

Revêtements métalliques et autres revêtements inorganiques — Essai de fragilisation résiduelle des articles et tiges filetés avec et sans revêtement métallique extérieur — Méthode de la cale biaise

Reference number
ISO 10587:2000(E)

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Printed in Switzerland

Foreword

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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.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 10587 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 2, *Test methods*.

Annex A of this International Standard is for information only.

Introduction

When atomic hydrogen enters steels and certain other 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, autocatalytic processes and in the service environment as a result of cathodic protection reactions or corrosion reactions. Hydrogen can also be introduced during fabrication, for example, during roll forming, machining and drilling, due to lubricant break-down, and during welding or brazing operations.

A variety of articles have threads as part of their structure. Examples are tools such as metal and wood working clamps, metal vices, tension clamps and taps, and hardware items such as threaded metal projectiles and bomb components, rifles, spring tension adjusters and piano stool lifts.

Industrial practice for threaded articles and rods has evolved three graduated levels of test exposure to assure reduced risk of hydrogen embrittlement (see clause 2). These levels have evolved from commercial applications having varying levels of criticality. In essence, they represent the confidence level that is required. They also represent the time that finished goods are held before they can be shipped and used. This time equates to additional cost to the manufacturer that may of necessity be added to the cost of the finished goods.

Metallic and other inorganic coatings — Test for residual embrittlement in both metallic-coated and uncoated externally-threaded articles and rods — Inclined wedge method

WARNING — Great care should be taken when applying this International Standard. The heads of embrittled articles or rods can suddenly break off and become flying projectiles capable of causing blindness or other serious injury. As this hazard can occur as long as 200 h after the test has started, shields or other apparatus should be provided to avoid such injury.

1 Scope

This International Standard specifies a method of determining, on a statistical basis, the probability of the existence of hydrogen embrittlement or degradation in:

a) a batch of barrel electroplated, autocatalytic plated, phosphated or chemically processed threaded articles;

b) a batch of rack plated threaded articles or rods.

This International Standard is applicable to threaded articles and rod made from steel with an actual tensile strength ⩾ 1 000 MPa (corresponding hardness values: 300 HV, 303 HB or 31 HRC) or to surface-hardened threaded articles or rods. It is not applicable to fasteners.

The test method is carried out after hydrogen embrittlement relief heat treatment and may also be used for assessing differences in processing solutions, conditions and techniques.

The test method has two main functions: a) when used with a statistical sampling plan it can be used for lot acceptance or rejection; b) it can be used as a control test to determine the effectiveness of the various processing steps including pre- and post-baking treatments to reduce the mobile hydrogen in the articles or rod.

Although the test method is capable of indicating those articles that are embrittled to the extent defined in clause 2, it does not guarantee complete freedom from embrittlement.

This International Standard does not relieve the plater, processor or manufacturer from imposing and monitoring suitable process control.

NOTE 1 The use of inhibitors in acid pickling baths does not necessarily guarantee avoidance of hydrogen embrittlement.

NOTE 2 Annex A provides guidance on sources of introduction of hydrogen into threaded articles.

2 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply.

2.1
embrittled article
article, parts of which fail immediately or up to 48 h in test

2.2
grade 48 proof batch
batch that provides no failures after 48 h of test

2.3
grade 96 proof batch
batch that provides no failures after 96 h of test

2.4
grade 200 proof batch
batch that provides no failures after 200 h of test

2.5
batch
distinct portion of articles processed collectively as a single group through the same identical treatment steps at the same time on the same rack or in the same barrel

2.6
lot
group of articles processed through the same or similar steps at the same time or over a contiguous time period and from the same heat of material

NOTE 1 A lot may be broken down into a number of batches for processing purposes and then reassembled into the same lot.

NOTE 2 The degree to which articles within a single plated batch or a given lot can be embrittled can vary over a wide range. The degree of embrittlement is a function of the concentration of atomic hydrogen in the individual articles of the batch or lot, measured in parts per million (ppm), and in particular that portion of the hydrogen that is mobile or free to migrate to areas of high stress concentration.

3 Principle

The threaded articles or rods are subjected to stress by tensioning with a mating nut after insertion through a clearance hole in a hardened rectangular wedge of steel; see Figure 1. Additional hardened rectangular pieces of steel, with parallel faces, are provided as filler plates and are inserted such that the required length of the threaded article is placed under test. Other loading systems and fixtures may be used provided the same load, angle and exposure are created for the test. The upper surface of the wedge is ground at an angle to the lower surface. The mating nut is tensioned by any means capable of measuring tensile load. The torque method referred to in 4.5 is one such method. If the torque method of tightening is used, the articles are torqued to the desired value, then held for the minimum specified number of hours and then checked to determine if the initial torque has been maintained. Following this they are examined for embrittlement failures; see clause 7.

NOTE Increasing the applied torque by a small percentage as a “safety factor” is not recommended.

4 Apparatus

4.1 General

The test fixture shall consist of a hardened wedge (4.2), see Figure 1, one or more filler plates (4.3) and a hardened washer (4.4). The hole in each shall be as close as practical to the major diameter of the threaded article or rods being tested.

NOTE 1 Excess clearance space can cause the article to tilt in the hole and can result in failure at a lower torque value.

NOTE 2 A fixture with multiple holes has been found useful for multiple or repetitive testing. The fixture can be made from a rectangular piece of an air hardening grade of steel with one face ground to the appropriate wedge angle and hardened to 60 HRC.

4.2 Wedge

The wedge shall have an angle as specified in Table 1.

Table 1 — Wedge angle selection

Nominal size of threaded article	Wedge angle for articles with unthreaded lengths less than two diameters °	Wedge angle for articles with unthreaded lengths two diameters and longer °
2 mm to 6 mm ($^{1}/_{12}$ inch to ¼ inch)	6	6
6 mm to 18 mm (¼ inch to ¾ inch)	4	6
$>$ 18 mm to 38 mm ($>$ ¾ inch to 1½ inch)	0	4

4.3 Filler plate

The filler plate(s) shall be of the same steel grade and hardness as the wedge fixture and have a thickness such that, after installation and tightening, a minimum of three full threads of the test article are engaged and no more than five full threads extend beyond the nut.

4.4 Washer

The washer shall be of 38 HRC to 45 HRC.

4.5 Torque application device

If the torque method of tightening is used, a load-measuring device capable of measuring the actual tension induced in the article or rod as the item is tightened shall be used to determine the tightening torque (see 6.3).

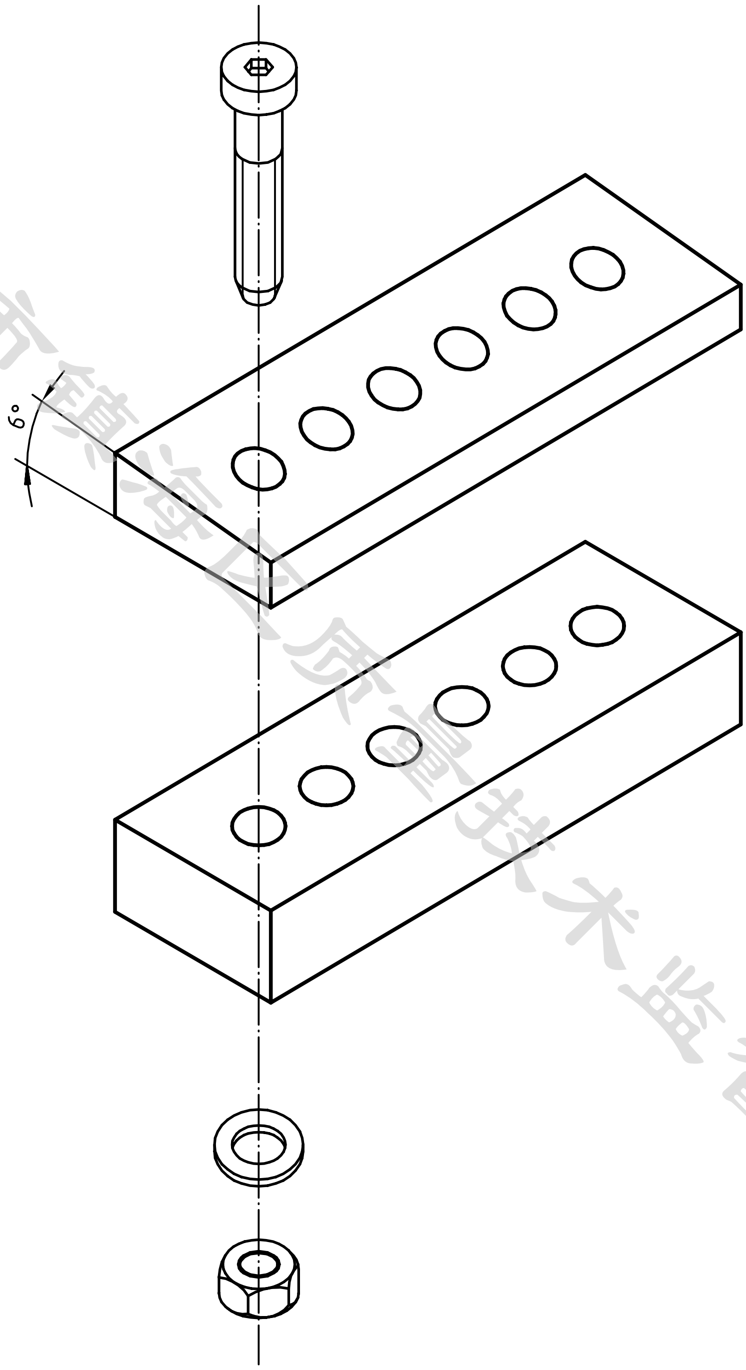

Figure 1 — Schematic diagram for testing of threaded articles and rods — Example of a 6° wedge and parallel filler plate (see Introduction for examples of threaded components)

5 Sampling

The AQL level and sampling plan used shall be that specified in the document that calls up this International Standard.

NOTE Widely used sampling plans are provided in ISO 4519 and ISO 2859.

A minimum sample size of 30 pieces shall be selected from each embrittlement relief treated batch that exceeds 500 articles plated as a single group.

6 Procedure

6.1 Test temperature

The test shall be carried out over a temperature range of 15 °C to 25 °C.

6.2 Test piece placement

Place the test pieces in the clearance holes with the heads positioned against the angle of the wedge. For pieces with square, hexagonal (or similar straight side heads) a straight side shall be placed against the angle of the wedge. For elliptical or other shaped heads, the side with the minor radius of the ellipse shall be placed against the angle of the wedge. For pieces without heads, studs or threaded rods one end shall be nutted and tested as the head. When the pieces are threaded with different pitch threads the finer thread shall be treated as the head. Nut the free end of the pieces and run them up finger tight.

NOTE No significance has been found between the start of the thread on an article in relation to the angle of the wedge.

6.3 Torque determination

Five artices from the test lot shall be selected at random. Each shall be assembled into the torque application device (4.5), mated with a nut, and the nut tightened until a load equal to (75 ± 2) % of the ultimate tensile strength of the article is induced. The torque required to induce this load shall be measured. The tightening torque shall be determined as the arithmetic mean of the five measured torques.

6.4 Torque application

Clamp the wedge with the nutted ends facing in a convenient position in a securely attached vice. Using a calibrated torque tool tighten the nuts to the desired torque and record the values. The wedge shall be removed from the vice and left undisturbed for the test period. See clause 2.

7 Evaluation

7.1 Cracks, separated heads and breakage

After the specified holding period is complete examine each article for failures such as cracks, separated heads and breakage. Use finger pressure to check each head for breakage. Cracks can be identified by examination at × 10 magnification, magnetic particle inspection or the use of a liquid dye penetrant.

7.2 Relaxed torque

Following the examination of the pieces as described in 7.1, place the wedge in a vice and, using the torque tool, carefully turn each mating nut in the "on" direction until a forward angular motion, after break loose, is noticeable. Record the torque value at break loose and compare it with the initially recorded torque. Torque relaxation greater than 10 % shall be recorded as failure.

Remove the nuts and examine the pieces for transverse cracks, which shall also be recorded as failure.

8 Test report

The test report shall include the following information:

a) the number of this International Standard, i.e. ISO 10587;

b) the batch identification and total number of articles in the batch;

c) the number of articles tested;

d) the number of broken articles, articles with visible cracks or other observed failures and the number of articles that exhibited relaxed torque;

e) the duration of the test;

f) the grade proof of the batch (see 2.2 to 2.4).

Annex A
(informative)

Sources of introduction of hydrogen into threaded articles

The preparation and metallic coating of threaded articles and rods are usually accomplished by the barrel-plating process. In this process, quantities of an article are placed within a containment vessel, called a barrel. The barrel is designed to move the group of articles, together, through each of the process steps, allowing ready ingress and egress of processing solutions and rinses. As the barrel is moved through the process steps, it is also rotated such that the individual articles are constantly cascading over one another. In some of the process steps, notably the electrocleaning and electroplating steps, an electric current is applied to the group of articles. The cascading action randomly exposes the surfaces of each individual article to the process electrodes while also maintaining electrical continuity between all the articles.

During both the electrolytic and non-electrolytic steps hydrogen is generated and exposed to the individual articles in the same random manner. Experience and experimentation have shown that despite best practice, some individual articles of the group will receive more hydrogen-charging exposure than others of the group due to the randomness of the barrel-plating process.

Examination and analysis of barrel-plated articles have shown that when hydrogen charging of such articles does occur, that it follows the normal distribution or bell-shaped curve. A very few of the articles absorb no hydrogen, the vast majority absorb a small amount of hydrogen and a very few articles absorb more hydrogen. Heat treatment, which can vary in time and temperature, can render the normally mobile hydrogen immobile thus rendering the individual articles free of hydrogen embrittlement. However, a number of variables exist within processes that, despite best practice, increase hydrogen charging on the articles. Platers cannot eliminate or easily control such random hydrogen charging. Therefore, testing representative quantities of the finished articles, selected using a statistical sampling plan, is necessary. It is not always possible to guarantee that lots of threaded articles produced by such processes are completely free of hydrogen embrittlement; the procedure can only guarantee that representative quantities of the lot have been tested and have shown no hydrogen embrittlement failures for the specified period of test.

Bibliography

[1] ISO 2859-1, *Sampling procedures for inspection by attributes — Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection*.

[2] ISO 2859-2, *Sampling procedures for inspection by attributes — Part 2: Sampling plans indexed by limiting quality (LQ) for isolated lot inspection*.

[3] ISO 4519, *Electrodeposited metallic coatings and related finishes — Sampling procedures for inspection by attributes.*

[4] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

ICS 25.220.40

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