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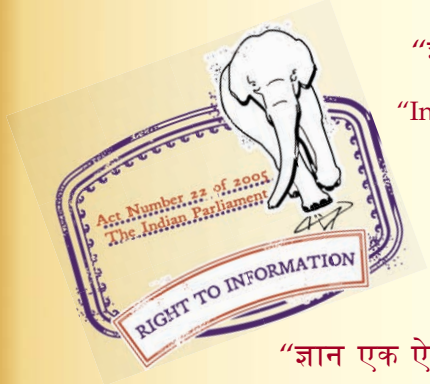
IS 10461-2 (1994): Resistance to Intergranular Corrosion of Austenitic Stainless Steels - Method for Determination, Part 2: Corrosion Test in a Sulphuric Acid/Copper Sulphate Medium in the Presence of Copper Turnings (Monypenny Strauss Test) [MTD 24: Corrosion Protection]



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"Knowledge is such a treasure which cannot be stolen"

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भारतीय मानक

आस्टेनाइटी स्टेनलैस इस्पात की अन्तकणिका संक्षारण के प्रतिरोध की निर्धारण विधि

भाग 2 तांबा छीलन की उपस्थिति में गन्धक का अम्ल/तांबा सल्फेट माध्यम में संक्षारण परिक्षण (मोनीपैनी सृटास परिक्षण)

(पहला पुनरीक्षण)

Indian Standard

RESISTANCE TO INTERGRANULAR CORROSION OF AUSTENITIC STAINLESS STEELS — METHOD FOR DETERMINATION

PART 2 CORROSION TEST IN A SULPHURIC ACID/COPPER SULPHATE MEDIUM IN THE PRESENCE OF COPPER TURNINGS (MONYPENNY STRAUSS TEST)

(First Revision)

UDC 669·14·018·8-194·56 : 620·193

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110 002

May 1994

Price Group 2

Corrosion Protection Sectional Committee, MTD 24

FOREWORD

This Indian Standard (Part 2) (First Revision) was adopted by Bureau of Indian Standards, after the draft finalized by the Corrosion Protection Sectional Committee, had been approved by Metallurgical Engineering Division Council.

This standard was first published in 1985 based on ISO 3651/II 'Austenitic stainless steels — Determination of resistance to intergranular corrosion : Part II Corrosion test in a sulphuric acid/copper sulphate medium in the presence of copper turnings (Monypenny Strauss Test)' issued by the International Organisation for Standardization.

While reviewing this standard in the light of experience gained during these years, the committee has decided to revise this standard bringing it in line with present practices being followed in the country.

Intergranular corrosion denotes deterioration of materials by means of preferential attack along grain boundaries. For austenitic stainless steels, which may be subjected to such an attack when they are kept at a temperature between 430 to 800°C, it is associated with the precipitation of chromium rich carbides at grain boundaries and the resultant chromium depletion (to a level less than 12% wt.) adjacent to the grain boundaries. In the case of wrought nickel-chromium-molybdenum steels, and in titanium or niobium-stabilized alloys, the presence of sigma phase also causes accelerated corrosion. The heat cycle, which may provoke susceptibility to intergranular corrosion, may occur during hot-forming process like forging and rolling, as a result of incorrect solution treatment or during a welding operation.

This standard (Part 2) has been prepared to provide a uniform and rationalized basis for a method of determining the susceptibility of austenitic stainless steels to intergranular attack due to precipitation of chromium carbide and the resultant chromium depletion regions. The attack caused by sigma phase is not reflected in this test.

This standard is issued in two parts. Part 1 of this standard deals with corrosion test in nitric acid medium by measurement of loss in mass (Huey test).

In reporting the result of a test done in accordance with this standard, if the final value, observed or calculated is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

RESISTANCE TO INTERGRANULAR CORROSION OF AUSTENITIC STAINLESS STEELS — METHOD FOR DETERMINATION

PART 2 CORROSION TEST IN A SULPHURIC ACID/COPPER SULPHATE MEDIUM IN THE PRESENCE OF COPPER TURNINGS (MONYPENNY STRAUSS TEST)

(First Revision)

1 SCOPE

1.1 This standard (Part 2) describes a procedure by which the copper sulphate-sulphuric acid test is to be conducted to determine the susceptibility of austenitic stainless steel to inter-granular attack.

1.2 This method is applicable only to austenitic stainless steel supplied in the form of cast rolled or forged products and tubes, and intended to be used in mildly oxidizing media.

1.2.1 The presence or absence of intergranular corrosion in these tests is not necessarily a measure of the performance of the material in other corrosion media. The tests do not provide a basis for predicting the resistance to other forms of corrosion, such as general corrosion, pitting or stress corrosion cracking.

2 REFERENCE

The Indian Standard IS 245 : 1988 'Trichloroethylene, technical (*third revision*)' is a necessary adjunct to this standard.

3 PURPOSE OF THE TESTS

3.1 The test indicates susceptibility to intergranular attack associated with the precipitation of chromium rich carbides at the grain boundaries and chromium depleted regions adjacent to the grain boundaries. The test does not detect susceptibility associated with sigma or other phases.

3.2 These tests may be used to evaluate any of the following three factors:

a) The effectiveness of the final heat-treatment when the material is tested in the as delivered condition without further test heat-treatment sensitisation,

b) The effectiveness of the added stabilizing elements, or low carbon content when the material is tested in the solution heat-treated and subsequently sensitized condition, and

c) The influence of welding and associated post fabrication heat-treatments.

4 HEAT TREATMENT FOR SENSITIZATION

In order to verify the effectiveness of the added stabilizing elements or low carbon content for resisting intergranular corrosion, it is necessary to carry out a heat treatment for sensitization. This treatment is usually obtained by maintaining a test piece for one hour at a temperature of 650-675°C followed by rapid cooling to room temperature in air or water unless other specific heating or cooling rates are stipulated. The most commonly used sensitizing heat treatment is 1 hour at 675°C and water quenching or air cooling.

5 CORROSION TEST

5.1 Principle

A test piece, prepared as specified in **5.4.2**, is immersed in a boiling sulphuric acid/copper sulphate solution for a specified time. The test piece is then subjected to a bend test. The convex surface of the test piece is examined after bending in order to reveal any cracks. When a test is made on a seamless tube, the test piece being subjected to the corrosion test is cut to give a test piece suitable for bending, and the surface of the tube to be put in tension during the bend test is to specified on the order. In the case of certain tubes (for example, small diameter tubes), a flattening test is used instead of the bend test.

5.2 Corrosive Solution

The sulphuric acid/copper sulphate solution shall be prepared as follows, using analytical quality reagents :

Dissolve 100 g of copper sulphate pentahydrate ($CuSO_4$, $5H_2O$) in 700 ml of dis-

tilled water. Then add 184 g (100 ml) of sulphuric acid (r. sp, gr 1.84) and make up to 1 000 ml with distilled water.

The corrosive solution may only be used once.

5.3 Apparatus

5.3.1 *Conical Flask*

Capacity one litre fitted with a four ball rising condenser.

5.3.2 *Support for the Test Piece*

Generally of glass, if necessary.

5.3.3 *Heating Device*

To keep the solution boiling.

5.4 Test Piece

5.4.1 *Dimensions*

The size of the test specimen and the area from which it is to be taken shall be mutually agreed between the purchaser and the supplier. The final size and shape of the test specimen will depend upon the available test apparatus but Table 1 may be used as a guide for suitable specimen sizes.

5.4.1.1 Rest pieces for welded products shall be the subject of agreement between the interested parties.

5.4.1.2 Specimens obtained by shearing should have the sheared edges machined or ground off prior to testing, without overheating. A squared edge is desirable.

5.4.2 *Surface Preparation*

Depending on the purpose of the test, the test piece, either with or without sensitization treatment, shall be prepared as specified in either **5.4.2.1** or **5.4.2.2.** Unless otherwise stated on the order, the method of preparation shall be left to the manufacturer.

Table 1 Size of Test Specimen
(*Clause* 5.4.1)

Sl No. (1)	Type of Material (2)	Size of Test Specimen (3)
i)	Wrought wire or rod:	
a)	Up to and including 6 mm diameter	Full diameter × minimum 75 long
b)	Over 6 mm diameter	Cylindrical segment of 6 mm (thick) × 25 mm wide (*Max*) × 75 to 125 mm
ii)	Flat rolled products (plate, sheet and strip):	
a)	Up to and including 5 mm thick	Full thickness × 10 to 25 mm wide × 75 mm (*Min*) long
b)	Over 5 mm thick	5 to 12.5 mm thick × 10 to 25 mm wide × 75 mm (*Min*) long
iii)	Pipes and tubes:	
a)	Up to 37.5 mm diameter	Full ring, 25 mm wide
b)	Over 37.5 mm diameter	A circumferential segment 75 mm (*Min*): in length, cut from a 25 mm wide ring

NOTES:

1 When bending cylindrical segment specimens, the curved surface shall be on the outside of the bend.

2 For flat-rolled products, which are not tested in full thickness, one of the original surfaces shall be retained in the test specimen and it shall be on the outside of the bend. Cold-rolled strip or sheets may be tested in the thickness supplied.

3 Ring sections of tubes should not be flattened or otherwise mechanically worked before they are put in the rest solution.

4 Specimens from welded tubes over 37.5 mm diameter, shall be taken with the weld in the axis of the bend.

5.4.2.1. *Mechnical Properties*

The test piece shall be descaled mechanically by polishing on all surfaces with grade 120 abrasive paper or cloth.

5.4.2.2 *Chemical preparation*

The test piece shall be descaled, without any previous mechanical treatment, in a solution of 50 volumes of hydrochloric acid (sp. gr. 1.19), 5 volumes of nitric acid (sp. gr, 1.40) and the 50 volumes of the water at 50 to 60°C.

5.4.2.3 *Degreasing*

The test piece shall then be degreased either in trichloroethylene (*see* IS 245 : 1970) or any other suitable solvent before being placed in the corrosive solution.

5.5 Procedure

5.5.1 *Immersion in Corrosive Solution*

Carry out the test in the presence of metallic copper (50 g of copper filings per litre of solution, introduced, at the beginning of the test), using a volume of corrosive solution of at least 10 ml/cm^2 of surface area of the test piece. Place the test piece on the copper filings, and ensure good galvanic contact over the entire surface by surrounding the specimens with copper filings. Bring to the boil and continue boiling for 24-72 hours. In case of dispute the duration of the test shall be subject of an agreement between the interested parties. For critical applications for example, chemical industries, fertilizer plants, power plants (Nuclear and Thermal), etc, test duration of 72 hours is recommended.

5.5.2 *Bend Test*

In the case of cylindrical and flat test pieces, subject the test piece to a bend test through 180° on a mandrel, the diameter of which is equal to the thickness of the test piece, except for cast products in which case the mandrel diameter is twice the thickness of the test piece.

5.5.2.1 In cases of material having low ductility, such as severely cold worked material, if a 180° bend proves impractical, then determine the maximum angle of bend without causing cracks in such a material by bending an untested specimen of the same configuration as the specimen to be tested.

5.5.3 *Flattening Test*

In the case of tubes tested in full section, a ring 40 mm long shall be flattened cold between parallel flat platens without showing crack or flaw until the distance between the platens under load is not greater than the value, H, in millimeters, given by the following formula:

$$H = \frac{1.09Dt}{0.09D + t}$$

where

D = outside diameter of the tube, in millimetres; and

t = specified wall thickness, in millimetres.

5.5.3.1 In case of welded tubes, the weld shall be at the point of maximum flattening.

5.6 Evaluation

5.6.1 The surface of test piece (convex side) shall be examined with the naked eye or using a magnifying glass (magnification not greater than 10 ×) in order to detect cracking. In the case of sheet with a thickness greater than 4 mm, the convex surface of the test shall be the retained rolled surface.

5.6.2 Freedom from fissures or cracks indicate the absence of intergranular corrosion and constitutes a satisfactory result. The presence of fissures or cracks at positions away from the edges of the test piece indicates the presence of intergranular attack and constitutes a test failure. For critical applications, when the test is carried out for 72 hours, presence of cracks greater than 25 microns should be taken as test failure. Appearance of deformation lines, wrinkles on the surface without accompanying cracks or fissures should be disregarded.

5.6.3 In the case of doubtful results, a 180° bend test shall be carried out on a second test piece prepared in a similar way but without having been submitted to the corrosion test. Alternatively a dye penetrant test may be carried out to distinguish between crack and wrinkles. The comparison of the two test pieces enables it to establishes whether the cracks observed on the corroded test pieces are due to intergranular corrosion. At the same time, if cracks are observed which cannot be definitely identified as intergranular, the longitudinal section of the test pieces may be examined metallographically, at a magnification of 100 × to 250 ×.

Bureau of Indian Standards

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