



SLOVENSKI STANDARD
oSIST prEN ISO 17781:2015
01-marec-2015

Petrokemična industrija ter industrija za predelavo nafte in zemeljskega plina - Preskusne metode za kontrolo kakovosti mikrostrukture avstenitno-feritnega (dupleksnega) nerjavnega jekla (ISO/DIS 17781:2015)

Petroleum, petrochemical and natural gas industries - Test methods for quality control of microstructure of austenitic/ferritic (duplex) stainless steel (ISO/DIS 17781:2015)

Erdöl-, petrochemische und Erdgasindustrie - Werkstofftestanforderungen für nichtrostenden Duplexstahl (ISO/DIS 17781:2015)

Industries du pétrole, de la pétrochimie et du gaz naturel - Méthodes d'essai de contrôle de la qualité de la microstructure des aciers inoxydables (duplex) austénitiques/ferritiques (ISO/DIS 17781:2015)

Ta slovenski standard je istoveten z: prEN ISO 17781

ICS:

75.180.01 Oprema za industrijo nafte in zemeljskega plina na splošno
Equipment for petroleum and natural gas industries in general

oSIST prEN ISO 17781:2015

en

DRAFT INTERNATIONAL STANDARD

ISO/DIS 17781

ISO/TC 67

Secretariat: NEN

Voting begins on:
2014-11-10Voting terminates on:
2015-02-10

Petroleum, petrochemical and natural gas industries — Test methods for quality control of microstructure of austenitic/ferritic (duplex) stainless steel

Industries du pétrole, de la pétrochimie et du gaz naturel — Exigences de test pour les matériaux en acier duplex inoxydable

ICS: 75.180.01

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Reference number
ISO/DIS 17781:2014(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 2.

The main task of technical committees is to prepare International Standards. 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.

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ISO 17781 was prepared by Technical Committee ISO/TC 67, *Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries*.

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Introduction

Duplex stainless steels have a two phase microstructure consisting of austenite and ferrite. Ideally these phases are present in equal proportions although in commercial alloys the ferrite phase volume fraction may vary between 35 % and 60 % for products in the solution annealed condition. Duplex stainless steels have roughly twice the strength of austenitic stainless steels with the same PREN value. Their resistance to localised corrosion (i.e. pitting and crevice corrosion) is similar, but the resistance of the duplex grades to chloride stress corrosion cracking is generally higher. They are characterized by high chromium (19 % to 33 %) and low nickel contents compared to austenitic stainless steels.

Duplex stainless steels are more prone than austenitic steels to precipitation of nitrides and intermetallic phases causing embrittlement and reduced corrosion resistance. The formation of intermetallic phases such as Sigma and Chi occurs in the temperature range 590 °C to 950 °C (1 100 °F to 1 750 °F) and reformation of ferrite occurs in the range 340 °C to 520 °C (650 °F to 975 °F). Exposure at these temperatures should therefore be avoided. In normal heat treatment and welding operations the risk of embrittlement is low. However, certain risks exist and consequently the thermal treatments required for manufacture and fabrication, as well as the operation conditions, must take the reaction kinetics of phase formation into account to ensure that desired corrosion resistance and mechanical properties are obtained ^{[4], [5]}.

The mechanical properties, corrosion performance and hydrogen embrittlement properties of duplex stainless steels require the control of the microstructure through application of appropriate manufacturing routes. The microstructure of fabricated equipments is sensitive to the thermal-mechanical history associated with hot working, solution annealing and with subsequent fabrication and welding.

Quality control testing should be specified to verify that components in these steel stainless grades are manufactured, heat treated and fabricated within acceptable parameters and possess the required properties for the relevant stainless steel grades.

This standard defines quality control testing methods, test conditions for the characterization of microstructure in relation to relevant properties of ferritic/austenitic stainless steels and specifies corresponding recommended acceptance criteria. The aim is to facilitate common testing requirements among the end users enabling the manufacturers to apply the same test procedures for their clients.

This standard may be used to supplement other product related standards and specifications. As such the standard should be applicable to a range of industrial users.

Petroleum, petrochemical and natural gas industries — Test methods for quality control of microstructure of austenitic/ferritic (duplex) stainless steels

1 Scope

This ISO standard provides quality control testing methods, conditions and specifies acceptance criteria to be used for the characterization of microstructure in relation to relevant properties in wrought, cast, hot isostatically pressed and welded austenitic/ferritic (duplex) stainless steel components in the solution annealed condition and fabrication welds. The standard considers “lean”, “standard”, “super” and “hyper” duplex grades.

The standard is based upon experience with duplex stainless steels in offshore oil and gas industry applications including topside and subsea hydrocarbon service, sea water service as well as structural use.

2 Normative references

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

ISO 148-1, *Metallic materials — Charpy pendulum impact test — Part 1: Test method*

ISO 8249, *Welding — Determination of ferrite number (FN) in austenitic and duplex ferritic-austenitic Cr-Ni stainless steel weld metals*

ISO 17025, *General requirements for the competence of testing and calibration laboratories*

ASTM A370, *Standard test methods and definitions for mechanical testing of steel products*

ASTM A1084, *Standard test method for detecting detrimental phases in lean duplex austenitic/ferritic stainless steels*

ASTM E562, *Standard test method for determining volume fraction by systematic manual point count*

ASTM E1245, *Standard practice for determining the inclusion or second-phase constituent content of metals by automatic image analysis*

ASTM G48, *Standard test methods for pitting and crevice corrosion resistance of stainless steels and related alloys by use of ferric chloride solution*

AWS A4.2 *Standard procedures for calibrating magnetic instruments to measure the delta ferrite content of austenitic and duplex austenitic-ferritic stainless steel weld metal*

3 Terms, definitions and abbreviated terms

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

3.1 Terms and definitions

3.1.1

type 20Cr duplex Group A (lean duplex)

ferritic/austenitic stainless steel alloys with $24 \leq \text{PREN} < 28$

3.1.2

type 20Cr duplex Group B (lean duplex)

ferritic/austenitic stainless steel alloys with $28 \leq \text{PREN} \leq 30$

3.1.3

type 22Cr duplex (standard duplex)

ferritic/austenitic stainless steel alloys with $30 < \text{PREN} < 40$ and $\text{Cr} \geq 19,5 \%$ (mass fraction)

3.1.4

type 25Cr duplex (super duplex)

ferritic/austenitic stainless steel alloys with $40 \leq \text{PREN} < 48$

3.1.5

type 27Cr duplex (hyper duplex)

ferritic/austenitic stainless steel alloys with $48 \leq \text{PREN} \leq 55$

3.1.6

pitting resistance equivalent number

PREN

index, developed to reflect and predict the pitting resistance of a stainless steel, based upon the proportions of Cr, Mo, W and N in the chemical composition of the alloy

Note 1 to entry: For the purposes of this part of this International Standard, all PREN limits specified in this International Standard shall be considered absolute limits, as defined in ASTM E29. With the absolute method, an observed value or a calculated value is not to be rounded, but is to be compared directly with the specified limiting value. Conformance or non-conformance with the specification is based on this comparison.

Note 2 to entry: The PREN calculation is based on actual composition, not nominal composition. Nominal composition is used for general classification only.

$$\text{PREN} = W_{\text{Cr}} + 3,3 \left(W_{\text{Mo}} + 0,5 W_{\text{W}} \right) + 16 W_{\text{N}}$$

where

W_{Cr} is the mass fraction of chromium in the alloy, expressed as a percentage of the total composition;

W_{Mo} is the mass fraction of molybdenum in the alloy, expressed as a percentage of the total composition;

W_{W} is the mass fraction of tungsten in the alloy, expressed as a percentage of the total composition;

W_{N} is the mass fraction of nitrogen in the alloy, expressed as a percentage of the total composition.

Note 3 to entry: There are several variations of the PREN. All were developed to reflect and predict the pitting resistance of Fe/Ni/Cr/Mo corrosion resistant stainless steels in the presence of dissolved chlorides and oxygen. e.g. in sea water. Though useful, these indices are not directly indicative of corrosion resistance in H₂S-containing oil field environments.

3.2 Abbreviated terms

HAZ	heat affected zone
HIP	hot isostatically-pressed
PREN	pitting resistance equivalent number
RT	room temperature
UNS	unified numbering system

4 Test methods

Compliance with the chemical and mechanical requirements for the applicable product specification does not necessarily indicate the absence of detrimental phases in the product or weldments made in duplex stainless steel grades. Therefore additional testing is specified to verify that the manufacturing process and the final quality heat treatment process are carried out under conditions that develop the required material properties and expected formability and weldability of the product.

NOTE The presence of intermetallic phases such as Sigma and Chi laves, and nitride and carbide precipitates are readily detected in all tests listed above when present in a certain amount. The test methods, conditions and acceptance criteria established and presented within this standard are based upon experience with the use of type 22 and 25Cr duplex grades in the oil and gas industry over the last 20 – 30 years.

Laboratory test methods for evaluation of the microstructure of duplex SS are as follows:

- a) microstructural examination;
 - check for presence of intermetallic phases and precipitates;
 - ferrite content counting.
- b) Charpy V-notch impact toughness testing;
- c) Ferric chloride corrosion test; ASTM G48 Method A or ASTM A1084 (type 20Cr duplex).

There are several parameters of the material microstructure that can influence the test results and the use of all three test methods as far as practical is necessary to demonstrate acceptable quality.

NOTE Preparation of metallographic specimens and subsequent etching procedures, Charpy V-notch impact toughness testing and corrosion testing require the use of hazardous equipment, consumables and chemicals. Personnel should be adequately trained. Facilities and procedures shall follow the relevant safety guidelines in place at the laboratory.

4.1 Microstructural examination

4.1.1 General

Microstructural examinations should be carried out at an appropriately equipped test house, by trained and experienced technicians and in accordance with established procedures in line with requirements of ISO 17025 and the requirements specified by this ISO standard.

4.1.2 Preparation of specimen

Duplex stainless steels can be prepared according to standard metallographic methods (grinding and polishing; manual or automated). The surface to be examined shall be free from any artefacts that may interfere with the interpretation of the microstructure.

NOTE Guidance to metallographic preparation of stainless steels may be found in ASTM E3.

With mechanical polishing, a 1 µm diamond final polish or equivalent should be considered a minimum. Preferably, a final oxide polish (e.g. colloidal silica/alumina) should be used.