SIST

SLOVENSKI STANDARD
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Aeronavtika - Titan in titanove zlitine - Preskusne metode - Kemiļ na analiza za doloļ evanje vodika

Aerospace series - Titanium and titanium alloys - Test method - Chemical analysis for the determination of hydrogen content

Luft- und Raumfahrt - Titan und Titanlegierungen - Prüfverfahren - Chemische Analyse zur Bestimmung des Wasserstoffgehaltes

Série aérospatiale - Titane et alliages de titane - Méthode d'essai - Analyse chimique pour détermination de la teneur en hydrogène

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Série aérospatiale - Titane et alliages de titane - Méthode d'essai - Analyse chimique pour détermination de la teneur en hydrogène

Luft- und Raumfahrt - Titan und Titanlegierungen - Versuchsmethode - Chemische Analyse zur Bestimmung des Wasserstoffanteils

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Foreword

This document (EN 3976:2006) has been prepared by the Aerospace and Defence Industries Association of Europe - Standardization (ASD-STAN).

After enquiries and votes carried out in accordance with the rules of this Association, this Standard has received the approval of the National Associations and the Official Services of the member countries of ASD, prior to its presentation to CEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2007, and conflicting national standards shall be withdrawn at the latest by June 2007.

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Introduction

This standard is part of the series of EN metallic material standards for aerospace applications. The general organization of this series is described in EN 4258.

1 Scope

This standard specifies the requirements for chemical analysis using Inert Gas Fusion Thermal Conductivity Method for the determination of the hydrogen content of titanium and titanium alloys for aerospace applications.

The method applies to hydrogen contents ranging from several micrograms per gram to several hundreds of micrograms per gram.

It shall be applied when referred to in the EN technical specification or material standard unless otherwise specified on the drawing, order or inspection schedule.

NOTE The absolute method not used in routine inspection is solid state hot extraction under vacuum followed by measurement of volume and pressure. Due to its complexity, it is only summarized in Annex A.

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 Guide 30:1992, *Terms and definitions used in connection with reference materials.*

ISO Guide 31, *Reference materials — Contents of certificates and labels.*

ISO Guide 35, *Reference materials — General and statistical principles for certification.*

EN 2003-10, *Aerospace series — Titanium and titanium alloys — Test methods — Part 10: Sampling for determination of hydrogen content.* [1)]

EN 4258, *Aerospace series — Metallic materials — General organization of standardization — Links between types of EN standards and their use.*

EN 4259, *Aerospace series — Metallic materials — Definition of general terms.* [1)]

1) Published as ASD Prestandard at the date of publication of this standard.

3 Terms and definitions

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

3.1
general terms
definition, see EN 4259

3.2
certified reference material (C.R.M.)
definition, see ISO Guide 30

4 Health and safety

Resources, test pieces, test samples, test materials, test equipment and test procedures shall comply with the current health and safety regulations/laws of the countries where the test is to be carried out.

Where materials and/or reagents which may be hazardous to health are specified, appropriate precautions in conformity with local regulations and/or laws shall be taken.

5 Principle

5.1 General

The test consists of thermally decomposing the hydrogenated compounds (mostly as metallic hydrides) present in the titanium or titanium alloys.

The degassed hydrogen is sent, in a stream of supporting inert gas, into the detection system (thermal conductivity cell) which allows a quantitative measurement of hydrogen content (comparative).

Two extraction modes are considered, with respect to the use (or not) of a melting flux. (Table 1).

Table 1

Method	Extraction Mode	Temperature	Supporting Gas
1	With flux	1 800 °C	Nitrogen or Argon
2	Without flux	2 100 °C	Argon

5.2 Method 1

The role of melting flux is to liquefy the mixture (thus facilitating the gas extraction) and to avoid an eventual formation of nitrides. Fusion is performed in a graphite crucible at a temperature of around 1 800 °C:

— either in a nitrogen supporting gas which allows the nitrogen in the sample to be disregarded with the carbon monoxide which, with the nitrogen, has a very small difference in conductivity. (The excess of carbon and the temperature at which melting is performed prevents any carbon dioxide from forming; it is necessary, however, to foresee a way of trapping small quantities of this gas eventually present by using, for example, a molecular sieve);

— or in an argon supporting gas which prevents nitrides forming and results in a better sensitivity since argon has a greater difference in conductivity compared to hydrogen. A separation of nitrogen and carbon oxides is necessary as well as, in the case of using a high frequency furnace, an adapted frequency to avoid discharges by ionization.

5.3 Method 2

Fusion is performed in a graphite crucible at a temperature of around 2 100 °C and shall be carried out in an argon supporting gas (nitrogen in this case would cause formation of nitrides).

6 Testing requirements

6.1 Resources

6.1.1 Equipment

There are numerous models of apparatus but they all generally include the following elements:

— Device for introducing samples;

— Heating device (induction furnace or resistance furnace);

— Sweeping system using supporting gas;

— System for separating gases (if necessary);

— Thermal conductivity cell.

Where a high frequency furnace is used with an argon supporting gas, the frequency shall be adapted to avoid discharges by ionization.

6.1.2 Materials/Reagents

6.1.2.1 Certified Reference Materials (titanium base alloys with certified hydrogen content)

According to ISO Guides 31 and 35 these C.R.Ms. should be produced by a certifying body, traceable to S.I. Units by demonstrated methods and accompanied by a certificate.

The certified hydrogen content value shall be accompanied by an uncertainty at a stated level of confidence.

6.1.2.2 Reagents

During the analysis only reagents of recognised analytical quality shall be used.

— Nitric acid
ρ_{20} = 1,38 g/ml

— Hydrofluoric acid
Aqueous solution at 40 %, ρ_{20} = 1,13 g/ml

— 95 % or 96 % (v/v) ethanol

— Melting flux tin granules

— Absorbing reagents, separating columns as specified by the manufacturer of the equipment

— Carbon tetrachloride

— Diethyl oxide

— Acetone

6.1.2.3 Compressed gases

6.1.2.3.1 Supporting gases

— Nitrogen of a purity > 99,995 % (N + rare gases) with H2 < 0,1 ppm (v/v)

— Argon of a purity > 99,995 % with N < 0,5 ppm (v/v) and H2 < 0,01 ppm (v/v)

6.1.2.3.2 Calibration gases

— Hydrogen of a purity > 99,9999 %

— Helium of a purity > 99,9999 %

6.1.2.3.3 Gas for pneumatic devices

Compressed air "for laboratory use".

6.1.3 Qualification of personnel

Testing to the requirements of this test method shall only be undertaken and/or supervised by personnel who have demonstrated their competence by a suitable education and appropriate training and experience.

6.2 Test samples/test pieces

6.2.1 Location of samples

In the case of semi-finished products: as per EN 2003-10.

In the case of finished products: as per special agreement and availability.

6.2.2 Sampling method and preparation of test portion

Sampling is carried out by machining, in dry conditions, with no lubricant, using tools cleaned with solvents (sawing with a blade, the protecting paint of which has previously been removed by cleaning with solvents and abrasive paper, drilling, pelleting, excluding hot cropping) in conditions whereby any heating of the metal is avoided as far as possible.

NOTE Titanium is easily hydrogenated by the humidity in the air at temperatures above 300 °C.

Sampling in the form of chips shall only be envisaged in the case of absolute necessity (due to the risks of heating the sample for analysis and the difficulties in introducing it in the analyser).

Whatever its surface appearance, the sample for analysis shall be thoroughly cleaned with solvents to eliminate any potential pollutants such as paint, grease, etc.

The procedure is as follows:

— clean at ambient temperature with a solvent such as carbon tetrachloride, diethyl oxide (ether), acetone ...,

— if trichlorethylene is used, it shall be followed by one of the above solvents,

— drying in the air at about 60 °C or at ambient temperature under a slight vacuum.

Furthermore, on certain semi-finished industrial products, but never on finished products, cleaning may be completed by pickling at ambient temperature for several minutes using the following acid mixture as recommended by work carried out by the European Community Bureau of References: