INTERNATIONAL STANDARD

ISO 11114-4

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Transportable gas cylinders — Compatibility of cylinder and valve materials with gas contents —

Part 4: Test methods for selecting metallic materials resistant to hydrogen embrittlement

Bouteilles à gaz transportables — Compatibilité des matériaux des bouteilles et des robinets avec les contenus gazeux —

Partie 4: Méthodes d'essai pour le choix de matériaux métalliques résistants à la fragilisation par l'hydrogène

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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.

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

ISO 11114-4 was prepared by Technical Committee ISO/TC 58, *Gas cylinders*.

ISO 11114 consists of the following parts, under the general title *Transportable gas cylinders — Compatibility of cylinder and valve materials with gas contents*:

— *Part 1: Metallic materials*

— *Part 2: Non-metallic materials*

— *Part 3: Autogenous ignition test in oxygen atmosphere*

— *Part 4: Test methods for selecting metallic materials resistant to hydrogen embrittlement*

Introduction

It is widely recognised that compressed hydrogen and some hydrogen bearing gases can have an embrittling effect on metallic materials, especially steels. This embrittling effect has resulted in the failure of hydrogen cylinders (including some bursts) that has led gas cylinder users and manufacturers to adopt specific measures.

The adoption of these measures has eliminated all known failures of hydrogen cylinders from this embrittlement phenomenon.

The basic recommendation was to minimise the stresses in the cylinder wall (see ISO 11114-1) and eliminate harmful defects.

This tensile strength limit of 950 MPa was developed for quenched and tempered gas cylinders of 34 Cr Mo 4 type steels using steelmaking practices, chemistry and manufacturing techniques typical of those used during the early 1980s and successfully used for filling pressures up to 300 bar. This practice has been in widescale use up to the current time. Other higher pressures, although at lower tensile strength limits, have also been used.

In recent years, improvements in steelmaking, e.g. by reducing the sulphur and phosphorus contents, have indicated the possibility of increasing the tensile strength limit of 950 MPa for embrittling gas service, as a result of improvements in the fracture toughness of the material.

Experimental work has shown that the relevant parameters affecting hydrogen embrittlement are the following:

a) microstructure resulting from the combination of the chemistry and the heat treatment;

b) mechanical properties of the material;

c) applied wall stress;

d) internal surface imperfections resulting in local stress concentrations;

e) characteristics of the gas contained (composition, quality, pressure, etc.).

When developing this part of ISO 11114 only the material aspects, a) and b) above, were considered. Other essential features, c) and d), are covered by the relevant parts of ISO 9809.

This part of ISO 11114 specifies test methods to identify steels which, when combined with the cylinder manufacturing requirements specified in ISO 9809 (all parts), will result in cylinders suitable for use in embrittling gas service.

However, some low alloy steels other than 34 Cr Mo 4 may require tensile strength lower than 950 MPa to be suitable for the manufacture of gas cylinders for embrittling gas service.

These tests have been developed following an extensive world-wide programme which incorporated laboratory and full scale tests.

Transportable gas cylinders — Compatibility of cylinder and valve materials with gas contents —

Part 4: Test methods for selecting metallic materials resistant to hydrogen embrittlement

1 Scope

This part of ISO 11114 specifies test methods and the evaluation of results from these tests in order to qualify steels suitable for use in the manufacture of gas cylinders (up to 3 000 l) for hydrogen and other embrittling gases.

This part of ISO 11114 only applies to seamless steel gas cylinders.

The requirements of this part of ISO 11114 are not applicable if at least one of the following conditions for the intended gas service is fulfilled [1]:

— the working pressure of the filled embrittling gas is less than 20 % of the test pressure of the cylinder;

— the partial pressure of the filled embrittling gas of a gas mixture is less than 5 MPa (50 bar) in the case of hydrogen and other embrittling gases, with the exception of hydrogen sulphide and methyl mercaptan in which cases the partial pressure shall not exceed 0,25 MPa (2,5 bar).

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 4287, *Geometrical product specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

ISO 7539-1:1987, *Corrosion of metals and alloys — Stress corrosion testing — Part 1: General guidance on testing procedures*

ISO 7539-6:2003, *Corrosion of metals and alloys — Stress corrosion testing — Part 6: Preparation and use of pre-cracked specimens for tests under constant load or constant displacement*

ISO 9809-1, *Gas cylinders — Refillable seamless steel gas cylinders — Design, construction and testing — Part 1: Quenched and tempered steel cylinders with tensile strength less than 1 100 MPa*

ISO 9809-2, *Gas cylinders — Refillable seamless steel gas cylinders — Design, construction and testing — Part 2: Quenched and tempered steel cylinders with tensile strength greater than or equal to 1 100 MPa*

1) In such cases the cylinders may be designed as for ordinary (non embrittling) gases.

ISO 9809-3, *Gas cylinders — Refillable seamless steel gas cylinders — Design, construction and testing — Part 3: Normalized steel cylinders*

ISO 11114-1:1997, *Transportable gas cylinders — Compatibility of cylinder and valve materials with gas contents — Part 1: Metallic materials*

ISO 11120, *Gas cylinders — Refillable seamless steel tubes for compressed gas transport, of water capacity between 150 l and 3 000 l — Design, construction and testing*

3 Terms, definitions and symbols

3.1 Terms and definitions

For the purposes of this document, the following terms and definitions apply. Some of the definitions used are based upon those in ISO 7539-1:1987.

3.1.1
embrittling gases
gases listed in ISO 11114-1:1997, Table A.3, group 2 and Table A.12, group 11

3.1.2
hydrogen rupture pressure, P_{H2}
maximum pressure recorded during the hydrogen rupture pressure test

3.1.3
helium rupture pressure, P_{He}
maximum pressure recorded during the helium rupture pressure test

3.1.4
hydrogen embrittlement index
maximum value of the ratio P_{He}/P_{H2} as a function of the pressure rise rate

3.1.5
environmentally-assisted cracking
synergistic effect on a metal caused by the simultaneous action of a particular environment and a nominally static tensile stress, which results in the formation of cracking

3.1.6
threshold stress
stress above which a crack will initiate and grow, for the specified test conditions

3.1.7
plane strain stress intensity factor, K_1
function of applied load, crack length and specimen geometry having dimensions of stress × length

NOTE K_1 uniquely defines the elastic stress field intensification at the tip of a crack subjected to opening mode displacements.

3.1.8
threshold stress intensity factor for susceptibility to environmentally-assisted cracking, K_{1H}
stress intensity factor above which an environmentally-assisted crack will initiate and grow, for the specified test conditions under conditions of high constraint to plastic deformation, i.e. under essentially plane strain conditions

3.1.9
HAC
hydrogen assisted cracking

