



HYDROGEN CYLINDERS AND TRANSPORT VESSELS

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EUROPEAN INDUSTRIAL GASES ASSOCIATION



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KEYWORDS

- CYLINDER
- HYDROGEN
- PRESSURE VESSEL
- STORAGE
- VESSEL

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1 Introduction

1.1 History

The origin of commercial hydrogen distribution, in Europe, goes back to the beginning of this century. It coincides with the introduction of flame welding as a major joining technique, where hydrogen originally served as a fuel gas. Hydrogen was pressurised and transported in steel cylinders or small pressure vessels, using cars or trucks for the distribution of the gas to the various customers.

This system of handling hydrogen in pressurised containers has not changed much during the last century, but the ever increasing demand for hydrogen, in all European countries, has led to the development and use of larger and more sophisticated transport equipment. This now includes individual bundles of steel cylinders, large vessels/tubes mounted onto railway trucks and a variety of trailer trucks for road transportation. Some companies use large cylinders mounted on semi-trailers, others use standard gas cylinders.

When compared to the long history of hydrogen transportation, the use of trailer trucks started in the early 1960s. The leading European hydrogen distributors now run several hundred thousand cylinders and transport vessels which supply 2,5 to $300 \times 10^6 \text{ m}^3$, annually, to their customers.

Most of the containers are seamless cylinders and, vessels/tubes made from quenched and tempered chrome-molybdenum alloy steels. In recent years, in order to improve the transport efficiency a variety of composite cylinders have been introduced. Generally, the working pressures are in the range of 200 to 300 bar.

Hydrogen in Europe is produced by steam reforming or as a by-product of chlorine production. After compression and purification the gas is delivered to the customer. Some companies also use their transport equipment as storage vessels at the customer's site, and it is exchanged as required, other companies re-charge static storage vessels instead.

For some years, this system of hydrogen distribution had been considered safe and reliable. However, in the late 1970s, records of reported failures and accidents with hydrogen transport equipment, in Europe, showed a sudden increase in frequency. In most cases the failure just resulted in leakage without any further damage, since the hydrogen gas did not ignite. However, in a few cases, the vessels exploded causing damage. It is interesting to note that the accidents in Europe occurred almost at the same time as problems with hydrogen storage vessels in the United States [4]. At that time the phenomenon of hydrogen environmental embrittlement was virtually unknown. No consideration had been given to the fact that structural steels might suffer from hydrogen embrittlement when in contact with the pure gas. The only information was some old studies where the problem had been treated as an academic curiosity. This situation changed soon after the first incidents with hydrogen vessels were experienced. In the United States research was undertaken to study the embrittlement mechanism and to identify the basic factors influencing the susceptibility of a material or engineering structure to Hydrogen.

In Europe, research activities [5] started a few years later. The French were the first to realise that. Hydrogen cylinders, as opposed to cylinders for other compressed gases, had to meet specific requirements. Based on the experience, which had been gained with similar steels in oil-field applications, the performance of the material in hydrogen was improved by limiting the maximum ultimate tensile strength and by closely controlling the alloy composition and heat treatment.

1.2 Present situation

The main factors that influence hydrogen embrittlement of a pressure vessel are shown in figure 1.

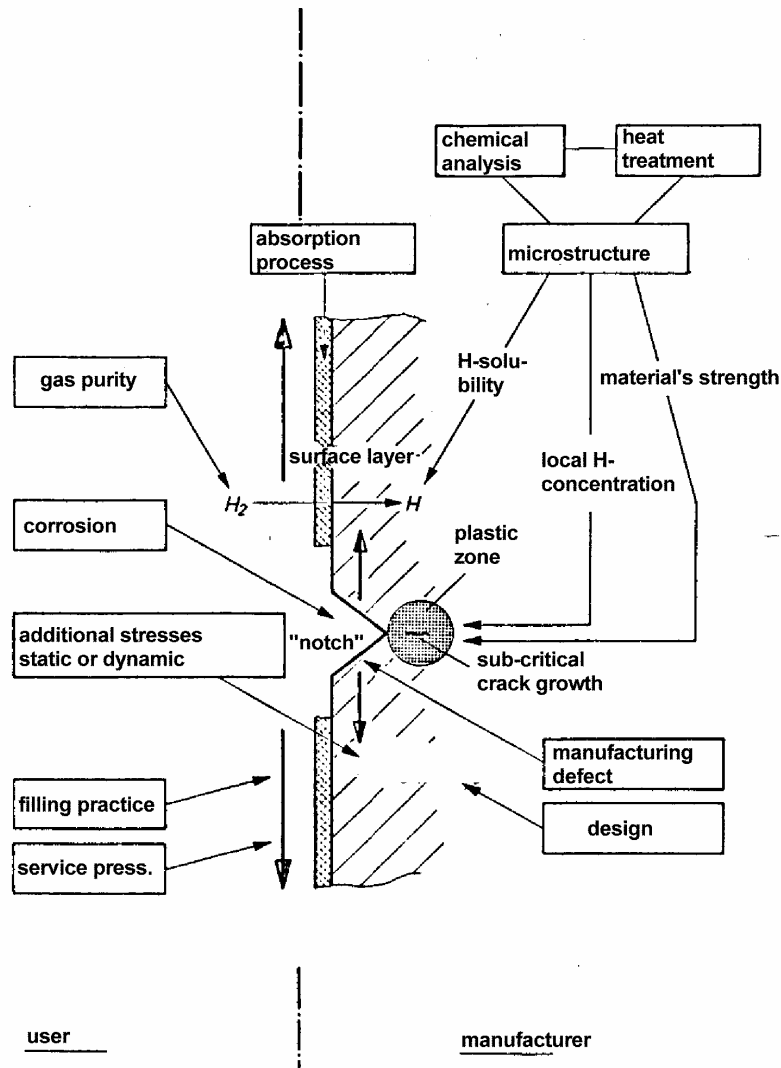


Figure 1: Hydrogen induced cracking (factors of influence)

The material and design aspects, which the cylinder manufacturer has to consider, are shown schematically on the right hand side of the figure. The basic requirements are:

- a steel of limited tensile strength with a suitable micro-structure and freedom from harmful inclusions
- a cylinder which is free from harmful surface defects

According to our present knowledge, probable cause for the early failure of hydrogen cylinders is fatigue, accelerated by hydrogen embrittlement. These failures are much more likely to occur in cylinders used in bundles or on trailers, which undergo by far the most pressure cycles; trailer cylinders/tubes may be filled twice a day and in some cases even more often.

Hydrogen accelerated fatigue cracks initiate at critical surface defects. Whether a surface defect has to be considered critical, of course, not only depends on its geometry but also on the locally acting stresses. This crack initiation process is favoured by the slow strain-rate that the cylinder wall is subjected to during filling with gas and pressurisation by hydrogen. The crack initiation process is also affected by the existing microstructure. With a more stable microstructure the generation of fatigue cracks is more difficult.

The growth of the resulting fatigue crack is accelerated in the presence of high pressure, high purity hydrogen. This has been demonstrated experimentally, with specimens taken from hydrogen cylinders [6]. Since failures occur by brittle fracture, from surface defects, investigation procedures based on fracture mechanics offer a useful method of assessment [7].

Failure of the cylinder or vessel/tube is imminent when the growing crack attains the conditions for crack growth to proceed under static load. This is controlled by the static threshold stress intensity K_{th} , which is a material property in the presence of hydrogen, but depends on the tensile strength and the microstructure of the steel. In figure 2, the results obtained by Loginow [8] are compared with findings from TI Chesterfield using cylinder material.

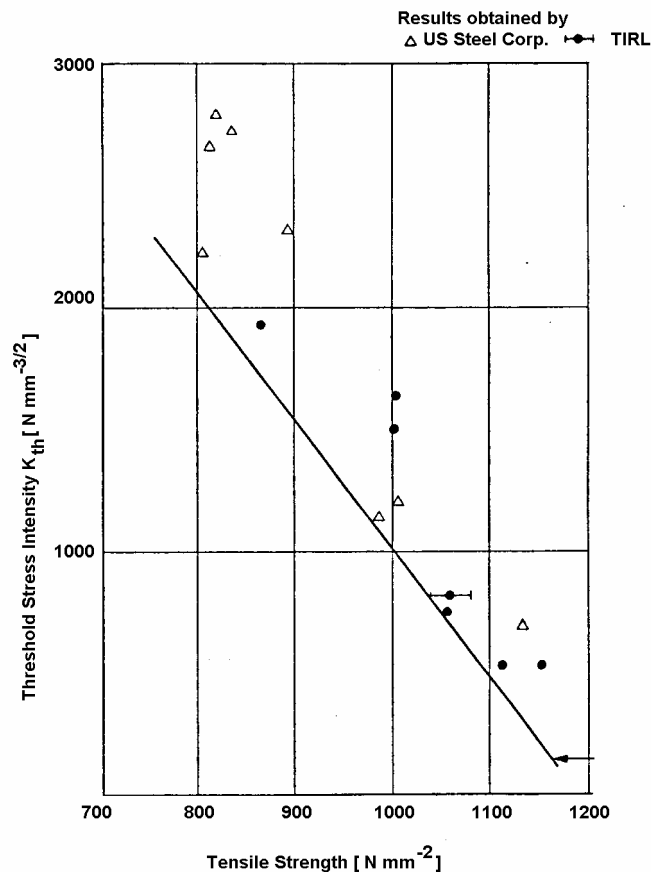


Figure 2: Threshold stress intensity for sustained load crack growth of CrMo steel in hydrogen of 200 bar

There have been a few failures where a hydrogen crack developed from an area with no pre-existing surface defect,. Here the cause of the failure was a steel with a very high tensile strength.

Another contributory factor to the failure mechanism was a poor base design. An example is shown in figure 3.

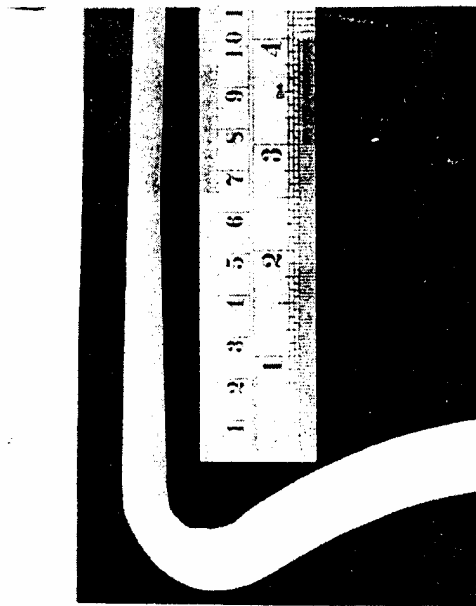


Figure 3: Base profile of a failed hydrogen cylinder

From this knowledge, the most obvious means of avoiding hydrogen accelerated failures, is to exclude critical surface defects and limit the ultimate tensile strength. In seamless steel cylinders and vessels/tubes, such defects either stem from the manufacturing process or they are produced during service, since many cylinders are not just exposed to hydrogen at various pressures but may experience unfavourable conditions which cause them to corrode. Incident data show that **internal surface defects have initiated at least 70% of all reported failures and that manufacturing defects alone account for approximately 50% of all reported failures i.e. 20 % due to service conditions.**

It is possible that localised corrosion in a hydrogen vessel is as important in initiating failure as manufacturing defects. Despite their relatively small size, corrosion pits appear to be efficient crack starters. At the present time, our knowledge is rather limited as to the possible reasons for this corrosion phenomenon. A major obstacle to identifying unfavourable conditions is the fact that most hydrogen cylinders have a rather exacting life with varying service conditions. Nevertheless, it seems that water vapour in hydrogen gas is not in itself sufficient to generate corrosion and other materials have to be present on a cylinder surface. Such materials might originate from the production process of the cylinder or might be introduced with the gas, possibly by back-flow at the customer site.

When considering the main factors which are responsible for the occurrence of failures, it should be borne in mind that the most important factor is the hydrogen susceptibility of the cylinder material itself. This material susceptibility is basically a function of tensile strength and microstructure [5] which is in turn, a function of the steel composition and the heat treatment.

1.3 Conclusion

Based upon the service experiences of EIGA companies and the results of their various research activities, the Industrial Gases Committee (IGC), started to issue recommendations for hydrogen cylinders and transport vessels/tubes in 1978. Long-term fatigue tests with actual hydrogen cylinders were carried out at experimental hydrogen test facilities. Low frequency fatigue tests with such cylinders have shown that the rejection criteria for internal surface defects in the IGC recommendations are sufficient to identify defects which might become critical [9].

The results of the various research activities and the fact that there have been no further incidents reported with hydrogen cylinders and transport vessels/tubes confirmed the IGC recommendations. These recommendations indicate that they are useful a means of minimising the risk involved in

transporting hydrogen in cylinders and pressure vessels/ tubes, provided both the gas industry and the manufacturers follow them.

1.4 Implementation

The requirements of this code have been widely implemented into national and international regulations and companies standards specifically for hydrogen cylinders and transport vessels/tubes have been promulgated.

During the implementation process the recommendations have required a significant commitment of human, manufacturing and financial resources. In addition, during the implementation-phase there was disruption of the supply of hydrogen to the consumer which significantly increased unit costs.

The recommendations of the working group are based on a combination of operating experience and on- going experimental work. Certain parameters that affect the life of the cylinder or transport vessel are fully understood, and that the following schedule for implementing the recommendations are safe and cost-effective.

Recommendation no. 1 (see clause 3) is applicable to all new quenched and tempered chromium-molybdenum seamless steel hydrogen cylinders and transport vessels/tubes.

There are other steels e.g. quenched and tempered manganese steels which are used at similar strength levels. These steels are treated in line with recommendation no. 1. (See ISO 11114-4 for guidance.).

Recommendation no. 2 (see clause 4) is applicable to all new seamless hydrogen cylinders and transport vessels/tubes.

Recommendation no. 3 (see clause 5) should be used immediately to review the design of mounting systems of seamless hydrogen cylinders and transport vessels/tubes and modifications to such systems should be carried out on a programmed basis. The duration of the programme should take into account the severity of any problems found during the review.

Recommendation no. 4 (see clause 6) should be used as a review document when implementing any of the other recommendations.

Recommendation no. 5 (see clause 7). The internal visual inspection should be carried out with great care for all seamless hydrogen cylinders and transport vessels/tubes at the next available periodic re-test. This is because of the role played by defects in the hydrogen embrittlement mechanism.

The tests to be carried out «once only» should be implemented at the next available periodic test for all seamless hydrogen cylinders and transport vessels mounted on trailers and for all frequently filled seamless hydrogen cylinder and transport vessel groups such as bundles.

In the case of single cylinders, which are filled less frequently, it is felt that the part of the recommendation «tests to be carried out once only» can be implemented more slowly over several national periodic tests since the risk, both in terms of filling frequency and releasable volume, is much smaller.

2 Scope

The Industrial Gases Committee of EIGA established the Working Group WG-O «Hydrogen Cylinders and Transport Vessels» * to formulate hydrogen-specific recommendations regarding design, material, manufacturing, testing, use and re-testing of seamless high pressure hydrogen cylinders and vessels, for use

- individually
- in bundles
- on trailers.

This document does not apply to static tanks, see document 15/96 Gaseous Hydrogen Stations. Subsequently the document has been revised by IGC/WG-2.

This work is based on the experience of EIGA members, particularly the evaluation of reports on failure of hydrogen cylinders. It is also guided by the literature and unpublished work on the effects of hydrogen on the mechanical properties of steels, by French metallurgical work [1], experience [2] and more recent results which has led to the publication of EN ISO 11114-4.

The recommendations have been discussed with European Cylinder Manufacturers.

The recommendations have been prepared with the understanding that they should be used together with sound engineering judgement based on training and experience.

The Working Group WG-2 has submitted recommendations to national and international organisations related to other problems as knowledge has been acquired.

* definition of cylinders: see [3] article 1.1 **

definition of transport vessel: seamless cylinders with outside
diameter ≥ 300 mm or
water capacity ≥ 150 litres

** for extract see appendix I

3 Recommendation no. 1

HEAT TREATMENT AND PROPERTIES OF 34 Cr Mo 4 Type Alloys

Summary: «Requirements of heat treatment conditions to achieve a microstructure resistant to hydrogen embrittlement and at the same time realise the optimum properties for this duty with 34 CrMo 4 type alloys».

3.1 Scope

This recommendation refers to all new seamless high-pressure hydrogen cylinders and transport vessels/tubes.

3.2 Composition, mechanical properties and heat treatment

34 CrMo 4 was commonly used in the past for the manufacture of high-pressure gas cylinders and transport vessels/tubes. The chemical composition and mechanical properties * most commonly used in the different countries were as follows:

		UK	Germany	France	Italy
Carbon	wt. %	< 0,37	0,30-0,37	0,30-0,38	0,27-0,34
Silicon	wt. %	010-0,35	0,15-0,35	0,20-0,40	0,15-0,35
Manganese	wt. %	0,40-0,90	0,50-0,80	0,70-1,00	0,5-0,70
Nickel	wt. %	< 0,50	-	-	-
Chromium	wt. %	0,80-1,20	0,90-1,20	0,90-1,20	0,8-1,2
Molybdenum	wt. %	0,15-0,25	0,15-0,25	0,25-0,40	0,15-0,25
Sulphur	wt. %	< 0,05	< 0,035	< 0,035	< 0,03
Phosphorus	wt. %	< 0,05	< 0,035	< 0,035	< 0,03
Rm (Nmm ⁻²)		890-1030	880-1030	880-1030	880-1030
Re (Nmm ⁻²)		> 755	> 755	> 735	> 700
Elong. (%)	(%)	> 14	> 16	> 16	> 14
(Lo=5.65√A)					

The ultimate tensile strengths (R_m) are sometimes in the upper range of the specified values. Experience in some European Countries of investigations of failed cylinders and present knowledge e.g. [ref. 5, 8, 10, 17] indicate the dependence of hydrogen induced failures on both the microstructure and tensile properties.

In any environment to utilise the potential tensile properties of high tensile steel without the danger of some mode of brittle failure, a number of well known precautions are necessary.

In recent years both at CEN and ISO standards have been developed with other tolerances and make reference to EN ISO 11114-1, dealing with hydrogen compatibility.

* 34 CrMo 4 is manufactured with comparable analysis and similar strength values in other countries.

Future chemical composition and heat treatment

For new hydrogen cylinders, the chemical composition of the steel shall satisfy the following requirements: $P \leq 0.025\%$ $S \leq 0.025\%$ $P + S \leq 0.04\%$.

For hydrogen cylinders there are additional requirements in the processes of heat treatment. These are:

- that the rate of quenching in the process of hardening (from the correct hardening temperature and austenitising time) is sufficient to give a fully quenched structure
- that the tempering temperature is sufficiently high to completely eliminate the original quenched martensitic structure

When these heat treatment requirements have been complied with, the following tensile properties of 34 CrMo 4, used for hydrogen cylinders and transport vessels/tubes, can be realised and are recommended:

Ultimate tensile strength (MPa)

$R_m \leq 950$ MPa (Work is in hand which with the use of further materials testing may result in tensile strengths greater than 950 MPa for 34CrMo4, (see ISO 11114-4). For other materials different R_m values may apply.)

Elongation ($L_0 = 5.65 \sqrt{A}$)

$E \geq 16\%$ (longitudinal) for machined cylindrical specimens**

Yield/Ultimate tensile strength*

Ratio: $\frac{R_{eA}}{R_m} \leq 0.90$

Impact toughness (J/cm^2)

No impact value shall be less than that indicated in the table below:

This table was taken from EN 1964-1.

Cylinder diameter D , mm		> 140			≤ 140
Direction of testing		Transverse			Longitudinal
Width of test piece, mm		>3 to ≤5	>5 to ≤ 7,5	>7,5 to ≤10	>3 to ≤5
Test temperature, °C		- 50			- 50
		Impact strength J/cm ²			
Quenched and tempered steels	Mean of 3 specimens	30	35	40	60
	Individual specimen	24	28	32	48
Normalized, or normalized and tempered steels with $R_m \leq 750$ MPa	Mean of 3 specimens	20			40
	Individual specimen	16			32

At the test temperature the crystallinity of the fracture surface shall not exceed 50%.

The tensile and impact properties have to be confirmed by tests, the results being certified by the manufacturer.

* for definition see appendix A

** some National regulations and industrial gas companies specify 14% elongation for longitudinal non-machined specimens and such experience indicates this does not present a problem

3.3 Confirmation of properties for cylinders

3.3.1 Tensile and impact tests

Tensile and impact tests shall be made on two separate cylinders according to European Directive 84/525/EEC, EN 1964-1 or ISO 9809-1.

If one of them fails either test, the batch shall be rejected and not used for hydrogen service unless they have passed the test after re-heat treatment.

3.3.2 Impact tests

The impact test shall be carried out in accordance with the relevant international standard e.g. EN 10045-1 or ISO 148.

The impact test pieces shall be taken longitudinally from the wall of the cylinder. The notch shall be perpendicular to the face of the cylinder wall. The test piece shall be machined all over (on six faces); if the wall thickness does not permit a final test piece width of 10 mm, the width shall be as near as practicable to the nominal thickness of the cylinder wall.

Note: These impact values only apply for new hydrogen cylinders. Existing hydrogen cylinders complying to the former Technical Note 26/81 need not necessarily meet the requirements of EN 1964-1.

3.3.3 Hardness test

At least two hardness tests, separated by about half the cylinder length, shall be performed on every cylinder on the cylindrical wall to check for the mechanical properties and homogeneity of the heat treatment.

The hardness value corresponding to 950 MPa shall be established by agreement between the manufacturer and user. If a Brinell test is used, it shall conform to ISO 6506-1, preferably with a ball diameter of 2,5 mm minimum.

3.4 Confirmation of properties for transport vessels/tubes

3.4.1 Tensile tests

Since the number of vessels per batch is small, one tensile test shall be made on a specimen taken from a control ring at least 200 mm long, cut from each tube before forging the ends and subjected to the same heat treatment as the finished vessel.

3.4.2 Hardness tests

At least six hardness tests shall be performed at various locations on the vessel. Bearing in mind the large surface area, the tests shall be located at points along different longitudinal axes in order to assess a reasonable area of the vessel. For large tubes, guidance is given in EN ISO 11120. The maximum hardness value corresponding to 950 MPa and the permitted difference between the maximum and minimum hardness values measured on the vessel shall be established by agreement between the manufacturer and the user.

3.4.3 Impact tests

If the wall thickness of the vessel does not exceed 10 mm the impact requirements shall be the same as mentioned above. If the wall thickness of the vessel is greater than 10 mm, the required impact values shall be according to EN ISO 11120.

At the test temperature of -20°C the crystallinity of the fracture surface shall not exceed 50%.

NOTE— The confirmation of the «crystallinity» shall be based on visual inspection of the fracture. The longitudinal test specimens shall be taken from the control ring.

If the wall thickness of the vessel exceeds 10 mm the test specimens should be taken as close to the inner surface of the control ring as possible.

4 Recommendation no. 2

NON-DESTRUCTIVE TESTING OF NEW CYLINDERS AND TRANSPORT VESSELS/Tubes

Summary: Requirements for non-destructive test methods (NDT) to identify defects on the internal and external surfaces of new hydrogen cylinders and transport vessels (particularly internal faces which cannot be inspected by a direct visual method), which would adversely affect the safe working of the cylinder or transport vessel/tube.

4.1 Scope

This recommendation refers to all new seamless high-pressure hydrogen cylinders and transport vessels/tubes.

4.2 Intermediate testing

The semi-finished cylinders (blanks or tubes) shall be examined prior to the final forming process to ensure that the material is free from harmful defects. This does not preclude earlier tests on the raw materials. If harmful surface defects are present they shall be removed.

4.3 Examination

Each cylinder shall have been examined in the course of its manufacture by the following tests for compliance with the following requirements:

4.3.1 Ultrasonic testing

Ultrasonic testing has proved to be a very reliable method to detect material defects that could lead to severe problems. Depending on the size of the vessel/tube see EN 1964-1 or EN ISO 11120.

Experience in some Countries has shown that existing cylinders and tubes manufactured from an ultrasonically tested tube did not require UT testing.

4.3.2 Wall thickness

4.3.2.1

Each cylinder must be inspected to ensure that it complies with the minimum wall thickness requirements.

4.3.2.2

The wall thickness shall be determined by a suitable and agreed method over the entire cylindrical body of the cylinder.

4.3.2.3

The base and neck areas of the cylinder shall be inspected to ensure that the geometry complies with the approved cylinder drawing.

Shape and wall thickness must be checked if the inner or outer surface has been machined, the method to be agreed between the manufacturer and the user.

4.3.3 Axial truth

Deviation from the axial truth shall not be greater than 2 mm per metre.

4.3.4 Roundness

The tolerance on the out of roundness shall be in accordance with the relevant regulations.

4.3.5 Internal surface

The internal surface shall be without sharp notched fissures, pits, cracks, folds, laps or other harmful defects. The surface shall be clean and free from loose rust or scale.

Because of the nature of hydrogen, it is essential that the internal surfaces of these cylinders and vessels/tubes shall be critically examined. Several methods may be used, for example (ultrasonic testing see 4.3.1) subject to agreement between the cylinder manufacturer and the user, provided these have been demonstrated to be suitable for the manufacturing method used. Regardless of the NDT method used at any stage of the manufacturing process, visual inspection shall be conducted on every cylinder.

If the surface is not originally free from such defects, it may be machined or otherwise treated to eliminate these defects and to ensure compliance with the specification. In regions of high stress concentrations i.e. the transition region between cylinder body and concave base, the internal surface should not be ground. Where grinding is carried out, great care shall be taken to avoid introducing further harmful defects.

After all repairs, the check of the wall thickness shall be repeated.

4.3.6 Internal surface of the neck

The internal surface of the neck shall be visually examined by an endoscope, mirror or other suitable means after the cylinder has been closed and the thread has been cut.

The inspection, machining of any defects and acceptance shall be as for example in EN 1964-1, Annex B.

4.3.7 External surface

The external surface shall be free from flats, bulges, hollows, laps, notches or other harmful defects and shall have been de-scaled.

4.3.8 Hydraulic test

The hydraulic test shall be performed in accordance with the relevant international standard.e.g. EN 1964-1

After this test the cylinder shall be cleaned and dried.

4.4 Inspection

Cylinders, which do not comply with this document or the previous versions, (TN 26/81) shall not be used in hydrogen service.

The test results shall be certified and cylinders which comply with the above specifications shall be marked with "H" in accordance with ISO 13769.

5 Recommendation no. 3

MOUNTING OF HYDROGEN CYLINDERS AND TRANSPORT VESSELS IN BUNDLES AND ON TRAILERS

Summary: When hydrogen cylinders are assembled in bundles or on trailers, certain mounting stresses in combination with other stresses (e.g. those due to cylinder surface defects) may affect the safe performance of the cylinders.

Some general notes of these effects are outlined.

5.1 Scope

This recommendation refers to seamless high-pressure hydrogen cylinders and transport vessels/tubes.

It is not possible to give quantitative information (e.g. permissible mounting stresses, impact loading, spacing distances) since the proven cylinder mounting practices vary from country to country and user to user. However, it is felt that in the interests of safety, these general recommendations should be followed with the aim of avoiding too high local stress concentrations due to mounting or corrosion.

It is recommended that the individual design should be checked against the following paragraphs.

5.2 Contact between cylinders

Cylinder mounting systems are a compromise between avoiding abrasion and impact stresses and avoiding local unfavourable stress concentration. The recommendations given below are good engineering practice for cylinders containing any gas, but are especially important for cylinders containing hydrogen.

5.2.1 Clamping devices

Clamping devices should minimise relative movement of the cylinders without causing unfavourable local stresses.

5.2.2 Spacers

Spacers should be suitably shaped to avoid unfavourable local stress concentration.

5.2.3 Metal to metal contact

Where the design allows metal to metal contact between cylinders, unfavourable local stress concentrations must be avoided.

5.2.4 Static loading

Attention should be paid to the static loading imposed on the lower rows of cylinders by the cylinders above them.

5.2.5 Expansion

Allowance should be made for changes in the dimensions of the cylinders due to filling pressure and temperature variations.

5.3 External corrosion

Particular attention should be paid to cylinder surface protection, e.g. painting. Adequate drainage should be provided to prevent the accumulation of water around the cylinders.

Bandages and supports should be of a non-water-absorbent material.

5.4 Valving and manifolding

Additional stresses in the cylinder neck during valving and manifolding should be minimised. The design of the manifold should minimise the possibility of vibrations of the manifold.

5.5 Vehicle design

The overall vehicle design should meet the dynamic force such as vibration encountered in service without transferring excessive stress to the cylinders.

5.6 Earthing

To reduce the risk of ignition from a leak, satisfactory earthing arrangements should be provided between the cylinders and the vehicle, as well as between the vehicle and the charging / discharging point.

5.7 Bundle design

Cylinders should be protected against being damaged during handling.

5.8 Example

An example of good engineering practice to avoid local unfavourable stress concentration and external corrosion is shown in appendix D.

6 Recommendation no. 4

DESIGN DETAILS, HYDROGEN QUALITY AND OPERATION OF EQUIPMENT

Summary: Based on present knowledge and experience of the influence of hydrogen on certain materials, attention should be paid to design details, hydrogen quality and operating procedures.

6.1 Scope

This recommendation refers to design and use of seamless high pressure hydrogen cylinders and transport vessels/tubes.

6.2 The design of cylinders and transport vessels, for example see appendix E

Attention should be paid to the neck and base regions of cylinders since the effect of local unfavourable stress concentrations is intensified by hydrogen, e.g. the inside surface of the neck of a cylinder or transport vessel/tube must be free from folds.

To ensure that the shape does not result in stress raisers and stress concentrations, it should be verified either by cycling tests, e.g. for cylinders in accordance with the international standard, or be calculated using an appropriate method, e.g. finite element analysis, that the design is acceptable.

Finally approved cylinder drawings must be fully dimensioned, especially in the region of the neck, the base and any machined areas, to enable verification of those dimensions during inspection.

Transport vessels/tubes shall be provided with end opening/s sufficient to enable complete visual internal inspection, the nominal diameter of the opening shall not be less than 60 mm.

The design of the openings and the plugs including the seal should allow for an increase of the vessel dimensions during filling. Seal welds shall not be used. The transition zone between the machined and un-machined parts of the internal surface, which come into contact with high pressure hydrogen, have to be smooth and free from notches or other injurious defects which could act as stress raisers.

The actual wall thickness must be adequate to allow the inside surface of cylinders and transport vessel/tubes to be shot-blasted during re-testing if necessary.

6.3 Hydrogen quality

The effect of water is not completely understood with respect to the hydrogen embrittlement mechanism. However free water in hydrogen cylinders should be avoided.

The hydrogen gas must not contain any harmful corrosive contaminants, see EN ISO 11114-1. This must be checked by appropriate analysis

As a general rule, the higher the hydrogen gas purity (especially with respect to oxygen content), the higher is the potential susceptibility of steels [11] to hydrogen embrittlement.

6.4 Gas mixtures with hydrogen

Experience shows that gas mixtures up to 5% hydrogen can be successfully used without applying recommendation no. 1.

Earlier research work [17] has indicated that hydrogen mixtures up to 50 bar partial pressure (25% hydrogen for 200 bar filling pressure) may not present a problem for quenched and tempered CrMo cylinders ($R_m \leq 1100$ MPa). Based on this research work, ISO 1114-1 has been modified to adopt the limit of 50 bar partial pressure which corresponds to a limit of 25% at 200bar.

6.5 Operation

Only cylinders that have been appropriately specified or selected for hydrogen service shall be filled.

Before filling all cylinders and transport vessels/tubes shall be checked that they are free from obvious damage and harmful contaminants.

Operating instructions must be available at each filling station.

Assemblies must be bonded and earthed during filling.

Backflow at the customer's site shall be prevented by suitable means, e. g. non-return valve.

7 Recommendation no. 5

PERIODIC RE-TESTING

Summary: Non-destructive tests that are to be carried out in addition to national periodic test requirements, either once only, or regularly at the time as defined by these requirements.

7.1 Scope

Many hydrogen cylinders and transport vessels/tubes in use, in Europe, were manufactured before this publication of the relevant IGC recommendations. Whilst it is felt that most of these cylinders and transport vessels/tubes are suitable for hydrogen service, the following recommendation has been written to eliminate those which are at risk. As well as this screening requirement, the recommendation includes certain additional non-destructive tests, which are to be carried out at each national periodic test of all seamless high-pressure hydrogen cylinders.

Transport vessels/tubes shall be similarly tested.

NOTE— Examples of severe defects are given in this recommendation (see Appendix F), but it should not be inferred from this that such defects are common in hydrogen cylinders or transport vessels.

7.2 Tests to be carried out at each inspection

Because of the nature of hydrogen, it is essential that the internal surfaces of hydrogen cylinders shall be most critically examined.

These tests are to be made in addition to periodic test requirements and at the same time.

7.2.1 Visual inspection of internal surface

Before performing any internal inspection or cleaning operation, it shall be ensured that the cylinder is fully evacuated from flammable gas in a safe manner.

Corrosion pits and manufacturing defects of the inner surface can act as crack starters, as evidenced by investigations of failed cylinders and transport vessels/tubes; the object of internal inspection is to eliminate cylinders containing these defects.

It is essential that the inner surface be free from loose corrosion products and scale, since these could obscure other more serious defects of the inner wall. Before further inspection and where necessary the inner surface shall be mechanically cleaned, e.g. by

- wire-brushing
- shot-blasting
- abrasive cleaning, water-jet

The internal surface shall be inspected, using visual aids, to ensure that it is free from the following defects:

- sharp-notched fissures
- pickling or corrosion pits
- laps or folds
- cracks
- scaling
- other injurious defects

For description and definition of rejection criteria see appendix F.

Typical visual aids increase illumination and/or magnification of the inner surface of the cylinder, e.g. fibre optics, television cameras or sources of illumination of sufficient intensity.

If doubt remains, the cylinder shall be further examined by appropriate non-destructive testing methods (see 7.3.2).

7.2.2 Visual inspection of external surface

Defects of the external surface might act as stress raisers.

It is important that the external surface be free from loose corrosion products and loose paint. Before further inspection and if necessary, the external surface shall be mechanically cleaned, e.g. by

- wire-brushing
- shot-blasting
- abrasive cleaning, water-jet

The object of external inspection is to eliminate cylinders containing the following defects:

- notches
- bulges, dents and flats
- laps
- corrosion pits
- hollows
- other injurious defects

For the description and definition of rejection criteria see appendix F.

7.2.3 Hydraulic test

To avoid corrosion, the cylinder shall be cleaned, dried and sealed immediately after any hydraulic test.

7.3 Tests to be carried out only once

Because of the nature of hydrogen it is essential that the ultimate tensile strength (R_m) may not exceed a recommended value.*

These tests shall be carried out when cylinders or transport vessels/tubes have not been manufactured in accordance with recommendations 1 and 2.

* for 34 CrMo 4 see recommendation no. 1

7.3.1 Hardness testing

Experience has shown that the ultimate tensile strength (R_m) is sometimes in the upper range of the specified values. Present knowledge indicates the dependence of hydrogen induced failures on the tensile properties. The hardness test is a convenient, practical non-destructive method to obtain approximate information of the ultimate tensile strength of an existing hydrogen cylinder. Where a cylinder or a transport vessel/tube has not been previously examined in accordance with recommendations no. 1 and 2, at the first available periodic test it shall be hardness tested in accordance with recommendation no. 1 and appendix G.

7.3.2 Other tests

Other tests are required in the following two cases:

Where a cylinder has not been previously examined in accordance with recommendations no. 1 and 2 or this Recommendation no. 5.

or where doubt remains following visual inspection or hardness testing.

In either of these cases the cylinder shall be inspected by ultrasonic examination using the appropriate parts of the procedures described in appendix B or by an equivalent inspection method proven by experience.

NOTE— To optimise the use of limited inspection resources, this further inspection may be confined to the regions of the cylinder that are at greatest risk (to be established by discussion between the user and the manufacturer for each cylinder type and manufacturing method).

Such tests shall preferably be carried out after any hydraulic test and not before as defects, e.g. cracks may grow during the hydraulic test.

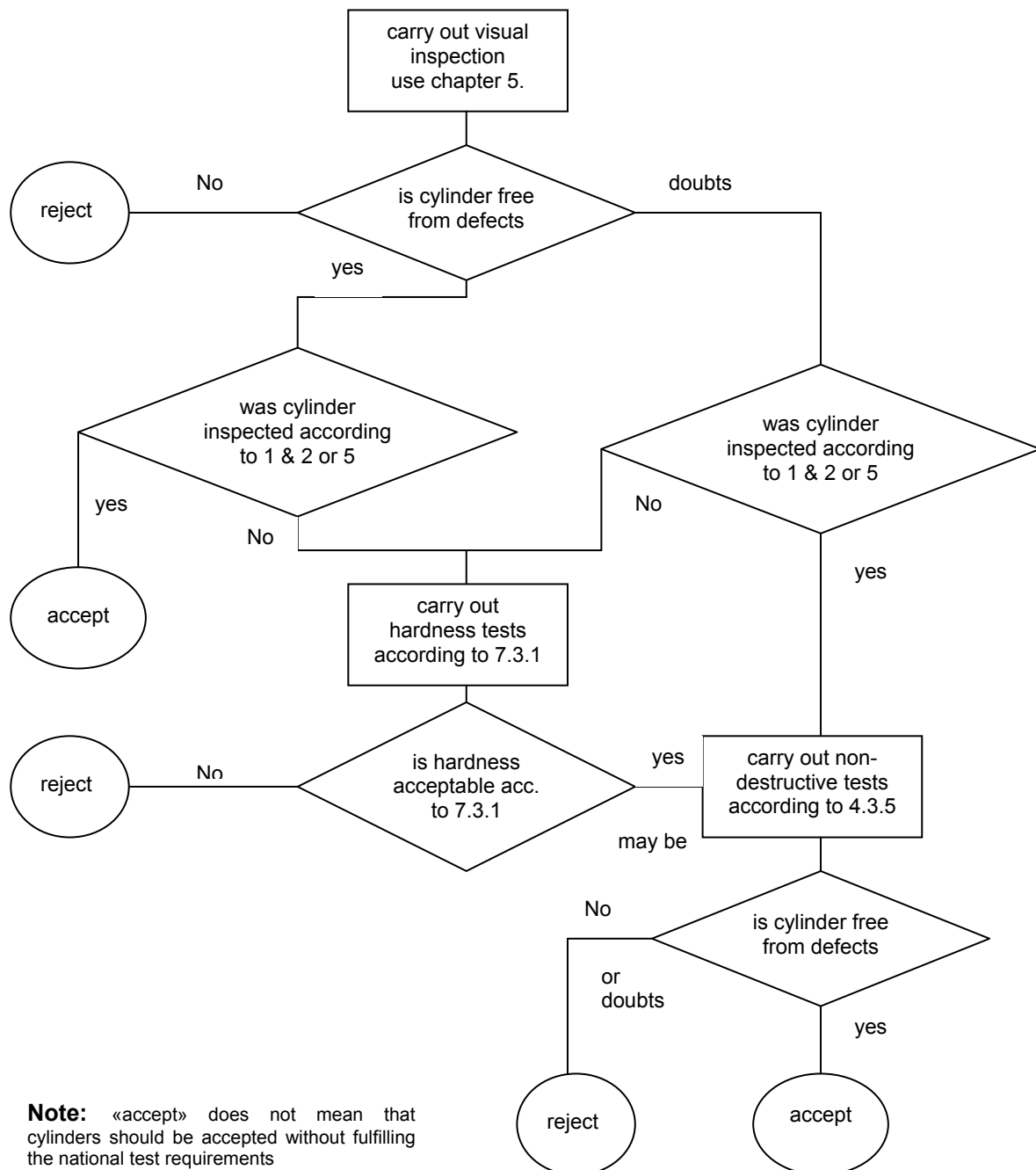
7.4 Inspection and certification

All tests shall be carried out by experienced and competent personnel.

The test results for all cylinders shall be documented and certified by the user's inspector.

7.5 Flow sheet

The recommended procedure is explained in a flow chart, figure 4.



Note: «accept» does not mean that cylinders should be accepted without fulfilling the national test requirements

«reject» means reject from hydrogen service

Figure 4: Requirements in addition to national periodic test

Appendix A: Definition of Yield Point

For definition of the yield stress/ultimate tensile strength ratio the yield value shall be as follows:

R_e = specified yield
 R_{ea} = actual yield
 R_m = actual tensile

For the definition of the yield stress/ultimate tensile strength the yield shall be as specified in the relevant international standard e.g. EN 1964-1.

In the present standard the yield value used for the calculation of the parts under pressure are the following :

- When a steel does not display a lower and an upper limit of flow, the value of the conventional yield point R_e shall be taken.
- When a steel displays a lower and an upper limit of flow the value to be taken is either:

- R_{eL}
or - $R_{eH} \times 0.92$
or - $R_{p0.2}$

R_{eL} = is the minimum value of the lower limit of yield guaranteed by the manufacturer in MPa.

R_{eH} = is the upper value of the limit of yield guaranteed by the manufacturer in MPa.

$R_{p0.2}$ = is the Conventional 0.2% yield limit guaranteed by the manufacturer in MPa

Appendix B: Ultrasonic Testing

Summary: Requirements for the efficient and accurate operation of ultrasonic testing equipment when used for the inspection of new and existing hydrogen cylinders, transport vessels/tubes.

For details of this test refer to the following standards as appropriate:

For new cylinders see EN1964-1

For transport vessels/tubes see EN ISO 11120

For retesting see EN 1968.

.

Appendix C: Examination of cylinder neck folds

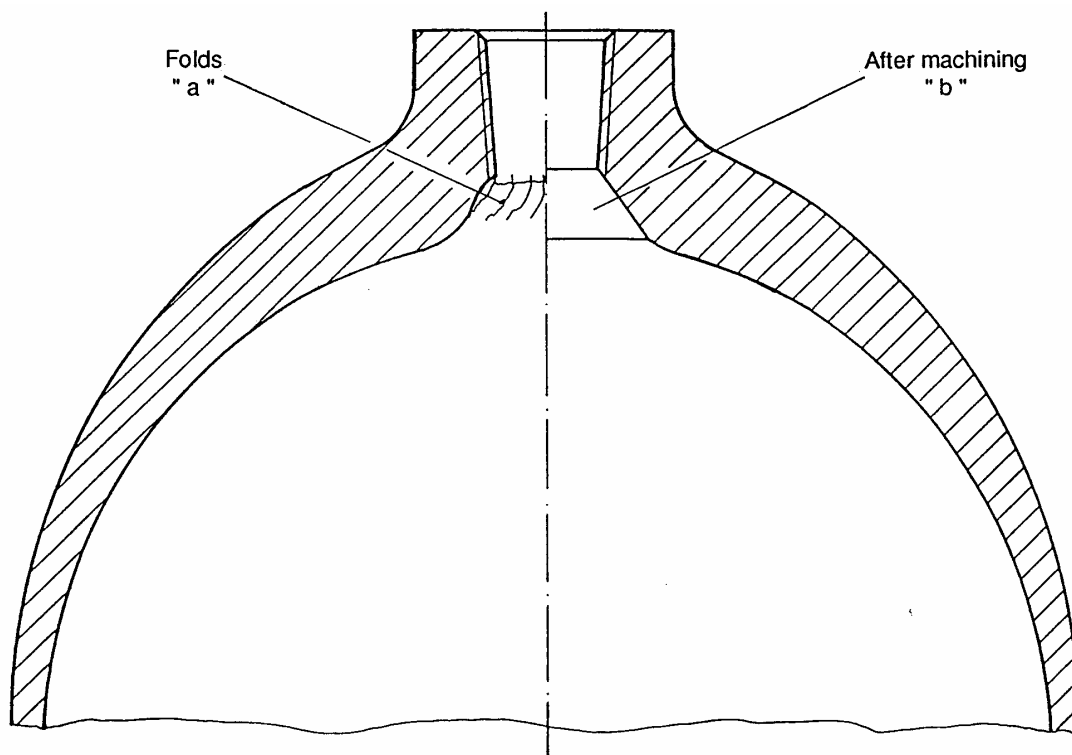
8 Examination for neck folds

Each cylinder shall be examined for neck folds and, in the case of cylinders made from seamless tube, for base folds by means of an endoscope or other suitable appliance.

Folds that are clearly visible as depressions having rounded peaks and roots shall not be deemed to constitute defects, but those which have sharp profiles or whose shape cannot definitely be identified, particularly those that are only discernible as a crack or a line of oxide on the cylinder surface or in the threaded portion, as shown in figure C/1 ("a"), shall be removed by a machining operation which produces a contour similar to that in figure C/1 ("b").

After machining, the whole area shall be re-inspected and measured for thickness. If the defects have not been completely removed, or, if the thickness at any part of the machined area is less than twice the minimum wall thickness shown on the drawing, the cylinder shall be deemed not to comply with the requirements of this document.

Figure C/1: Cylinder neck folds before and after machining



Appendix D: Bundle design example

One example of hydrogen cylinders mounted in a bundle (see EN 13769) to avoid local unfavourable stress concentration and external corrosion, is shown in figure D.1 and D.2

The cylinders are mounted using a steel frame, distance spacers and rubber spacers (approximately 70 Shore) of 40 mm width and 16 mm thickness.

- to avoid unfavourable metal to metal contact between the cylinders and between the cylinders and the steel frame
- to avoid abrasion of metal at the cylinder surface
- to avoid local stress concentration and impact stress in the cylinder wall. To this end the width of the rubber spacers has been optimised
- to provide an adequate drainage thus avoiding dirt accumulation and contact corrosion
- to allow for an increase in the cylinder dimensions during filling

Lifting points are provided near the top of the bundle. The structure is of sufficient stiffness so that excessive stresses are not induced in the cylinder wall by deflection during lifting or transportation.

The outer cylinder surface has to be adequately protected against corrosion.



Figure D.1 : Modern hydrogen bundle design, according to EN 13769, containing 12 cylinders with 50 ltr. watercapacity/ 200 bar filling pressure each

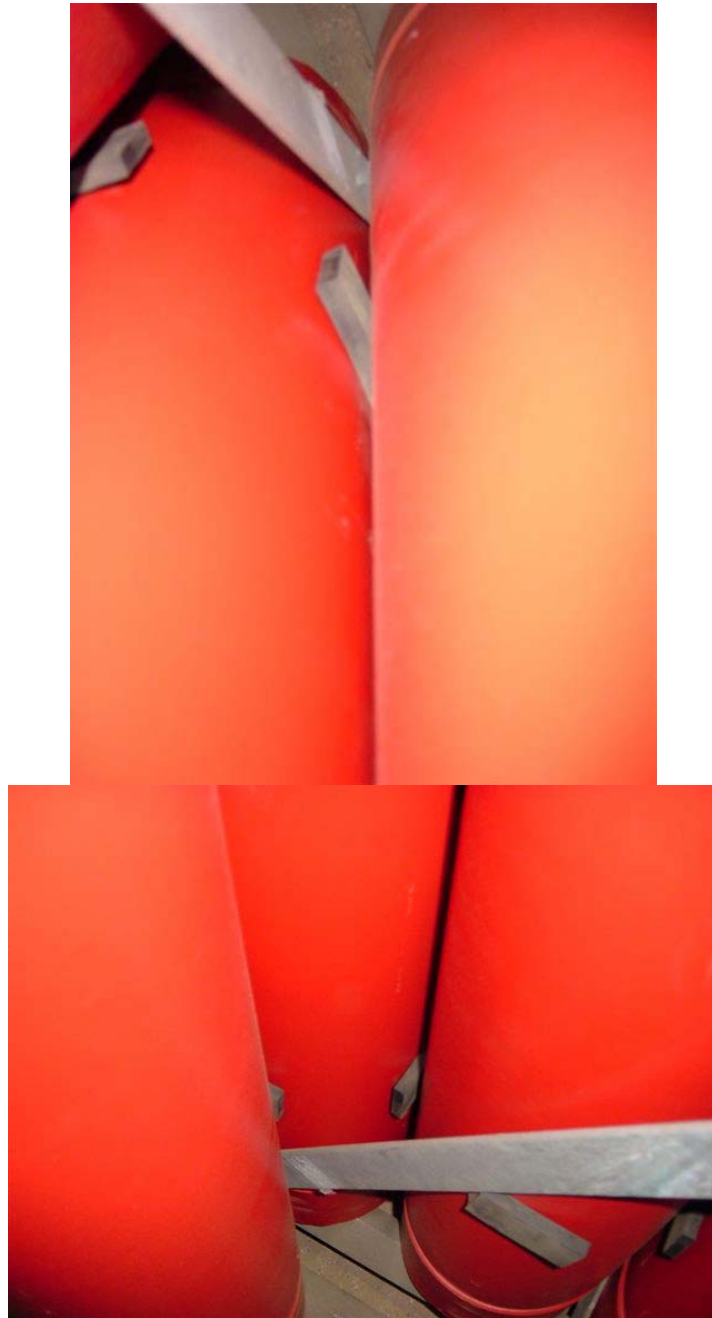
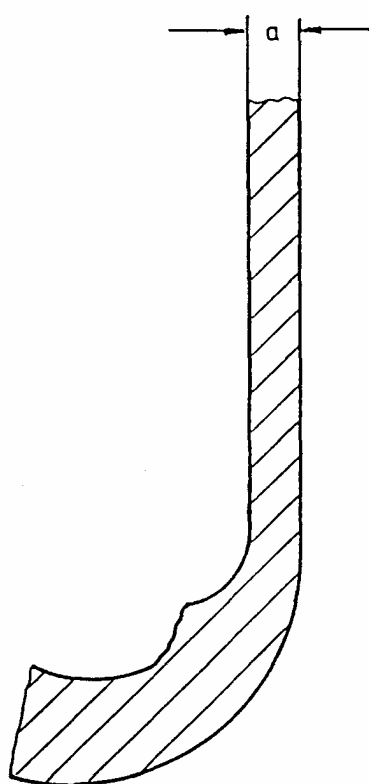
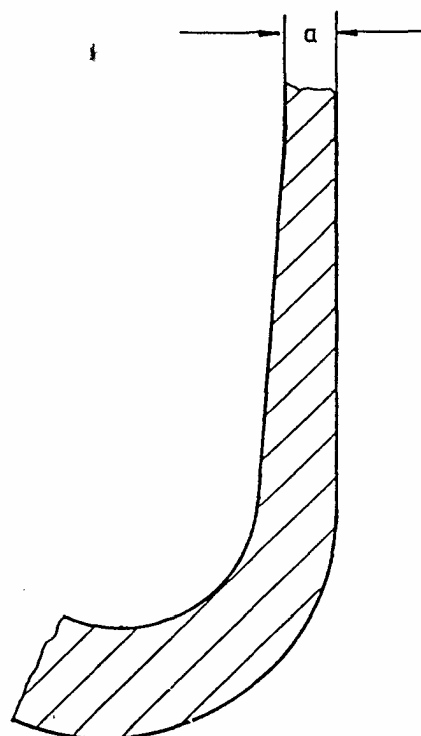


Figure D.2

Bundle design example

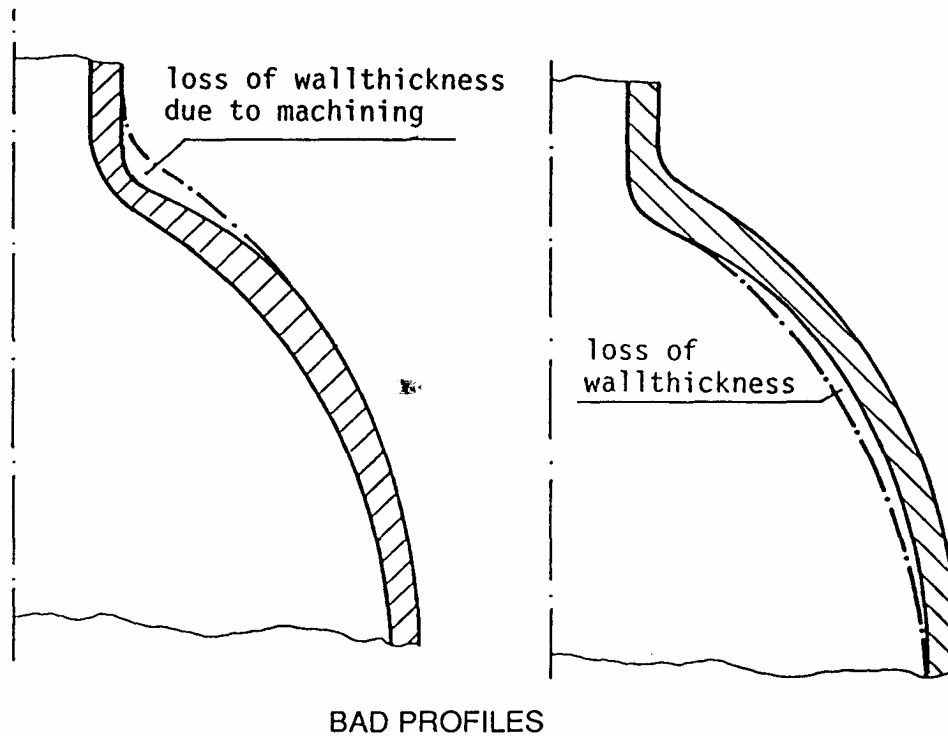
Appendix E: Design of cylinders and transport vessels - examples**9 Knuckle region of a cylinder with a concave base**

The thickness of a cylinder wall shall increase progressively in the transition region, from the cylindrical part to the base, without abrupt changes of profile.

**BAD PROFILE****GOOD PROFILE**

10 Neck region of cylinders and transport vessels

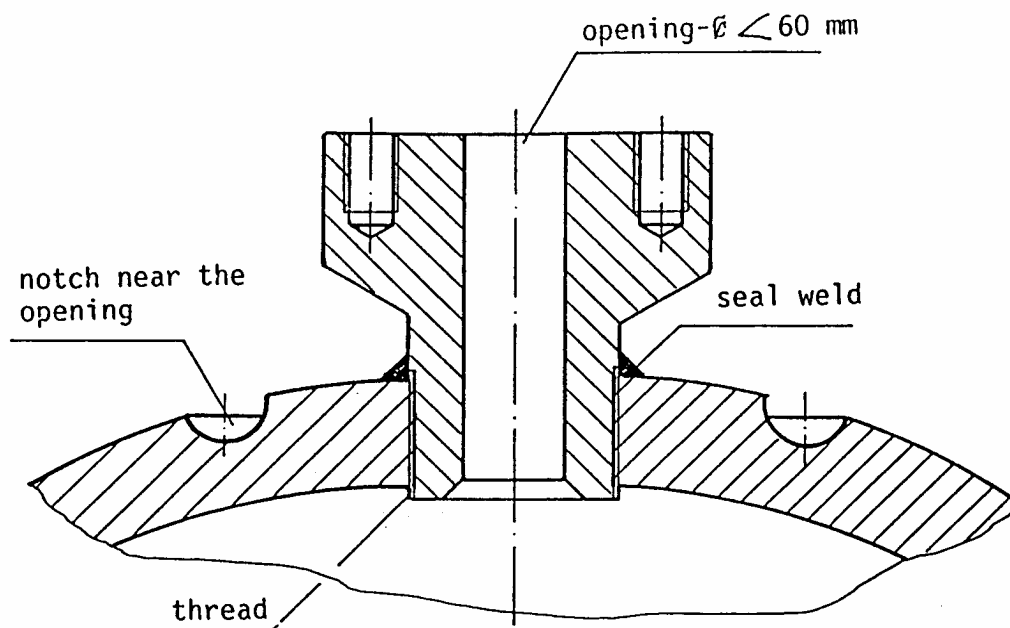
The wall thickness of the neck shall increase progressively from the cylindrical part to the opening. Care should be taken if parts of the neck have been machined.



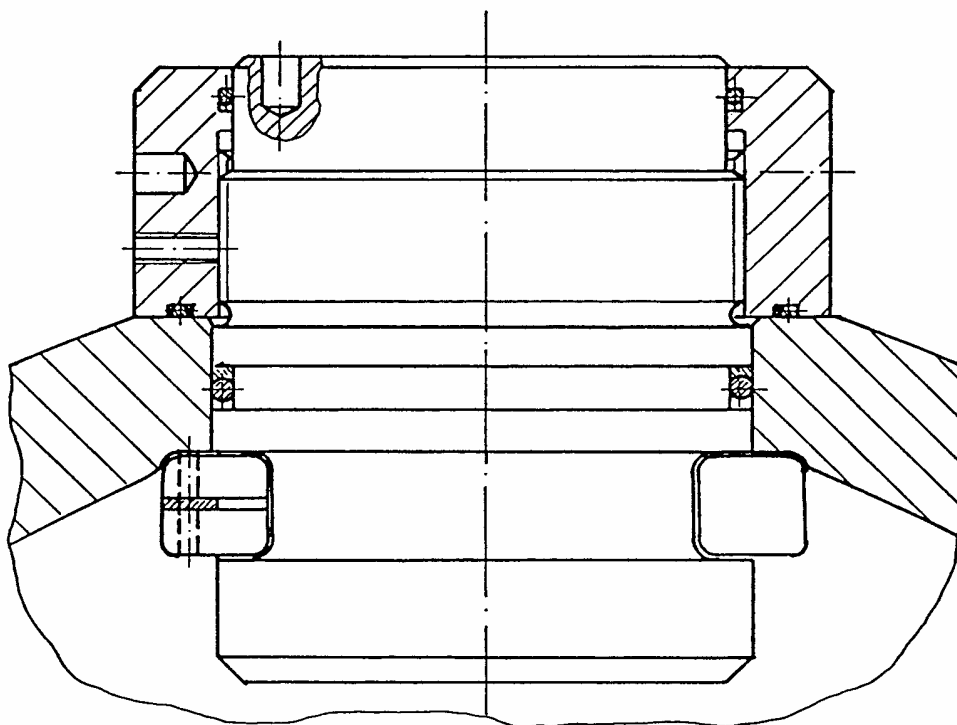
11 Openings and plugs of transport vessels

For the design of the openings and plugs, attention should be paid to the following items:

- small diameters of the opening do not allow a sufficient visual inspection of the inner surface
- seal welds on a high strength steel may crack and should not be used
- notches on the outer surface near the opening can act as stress raisers
- threads which are in contact with hydrogen can act as stress raisers and should be avoided
- material used for plugs or end closures shall be selected from high quality steel and shall be free from significant internal defects
- gasket material should be hydrogen compatible



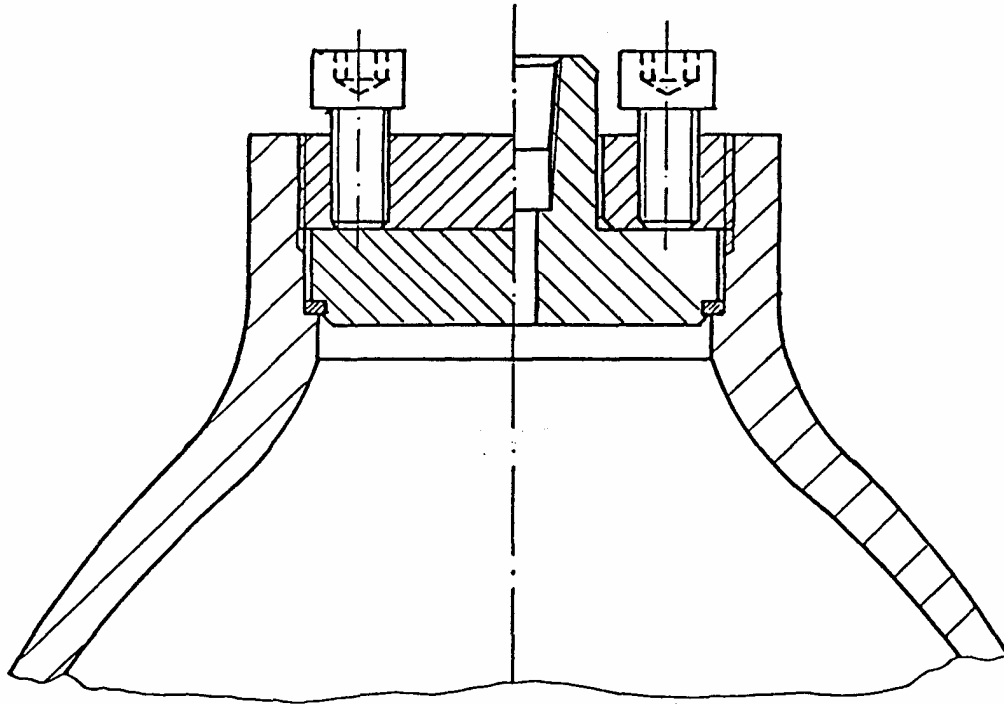
Example with unsatisfactory experience



Example with satisfactory experience

Example 1 *

* Rapid-Closing connection for seamless pressure vessels of Mannesmann Stahlflaschen AG, Düsseldorf



Example with satisfactory experience

Example 2 **

** Vallourec System

Appendix F: Description and evaluation of defects and criteria for rejection of seamless high pressure hydrogen cylinders and transport vessels/tubes at the time of visual inspection

12 General

Hydrogen cylinder defects may be physical or material or due to corrosion, as a result of environmental or service conditions to which the cylinder has been subjected.

The object of this appendix is to give general guidelines to the hydrogen cylinder users for the application of rejection criteria.

The figures should not be considered as strict limits, they are based on experience and have not been proved by appropriate analytical methods.

13 Physical or material defects

Evaluation of physical and material defects shall be in accordance with table 1.

14 Corrosion

14.1 General

Internal corrosion of the metal can also occur owing to service conditions.

There is difficulty in presenting definite rejection limits in tabular form for all sizes and types of cylinders and their service conditions. The limits of rejection have been established following considerable field experience.

Extensive experience and judgement are required in evaluating whether cylinders with internal corrosion are safe and suitable for return to service.

It is important that the surface of the metal is completely clean of corrosion products prior to the inspection of the cylinder.

14.2 Evaluation of corrosion

If the bottom of the defect cannot be seen or when its extent cannot be evaluated using special equipment, the cylinder shall be rejected.

The corrosion of the cylinder wall shall be evaluated in accordance with table 2.

15 Removal of external defects

Any defect on the external surface presenting a sharp notch may be removed by grinding, machining or other approved methods. After such a repair, checking of the wall thickness shall be repeated.

This table is taken from EN 1968 and applies to retested cylinders. However, for new cylinders, refer to the appropriate rejection limits for new manufactured cylinders/vessels, (e.g. EN 1964-1)

Table 1A — Rejection limits relating to physical and material defects in the cylinder shell

Type of defect	Definition	Rejection limits ^a	Repair or render unserviceable
Bulge	Visible swelling of the cylinder	All cylinders with such a defect	Render unserviceable
Dent	A depression* in the cylinder that has neither penetrated nor removed metal and is greater in depth than 1 % of the outside diameter	When the depth of the dent exceeds 3 % of the external diameter of the cylinder; or When the diameter of the dent is less than 15 times its depth	Render unserviceable Render unserviceable
Cut or gouge (see Figure C.1)	A sharp impression where metal has been removed or redistributed and whose depth exceeds 5 % of the cylinder wall thickness	When the depth of the cut or gouge exceeds 10 % of the wall thickness; or When the length exceeds 25 % of the outside diameter of the cylinder; or When the wall thickness is less than the minimum design thickness	Repair if possible ^b Repair if possible ^b Render unserviceable
Crack (see Figure C.2)	A rift or split in the metal	All cylinders with such defects	Render unserviceable
Fire damage (see Figure C.3)	Excessive general or localized heating of a cylinder usually indicated by: a) partial melting of the cylinder; b) distortion of cylinder; c) charring or burning of paint; d) fire damage to valve, melting of plastic guard or date ring.	All cylinders in categories a) and b) All cylinders in categories c) and d) may be acceptable after inspection and/or testing	Render unserviceable Repair if possible
Plug or neck inserts	Additional inserts fitted in the cylinder neck, base or wall	All cylinders unless it can be clearly established that addition is part of approved design	Repair if possible
Stampmarking	Marking by means of a metal punch	All cylinders with illegible, modified or incorrect or incorrectly modified markings	Render unserviceable ^c
Arc or torch burns (see Figure C.4)	Partial melting of the cylinder, the addition of weld metal or the removal of metal by scarfing or cratering	All cylinders with such defects	Render unserviceable
Suspicious marks	Introduced other than by the cylinder manufacturing process or approved repair	All cylinders with such defects	Continued use possible after additional inspection
Grinding or machining	Local reduction of wall thickness or hardened heat affected zone	When the wall thickness is reduced to below the minimum wall thickness. When the hardness locally exceeds the maximum specified value.	Render unserviceable

* see also excessive grinding or machining

Table 1 — Rejection limits relating to physical and material defects in the cylinder shell

Type of defect	Definition	Rejection limits ^a	Repair or render unserviceable
Vertical stability		Deviation from verticality which can present a risk during service (especially if fitted with foot-ring)	Repair if possible or render unserviceable

^a When applying the rejection criteria given in this table, the conditions of use of the cylinders, the severity of the defect and safety factors in the design shall be taken into consideration.

^b Provided that after repair by a suitable metal removal technique, the remaining wall thickness is at least equal to the design minimum wall thickness.

^c If it can be clearly established that the cylinder fully complies with the appropriate specifications, altered operational and modified marking may be acceptable and inadequate markings may be corrected, provided there is no possibility of confusion.

Table 2 — Rejection criteria for corrosion of the cylinder wall

Type of corrosion	Definition	Rejection limits ^a	Repair or render unserviceable
General corrosion (Figure C.5)	Loss of wall thickness over an area of more than 20 %* of the total surface area of the cylinder	If the original surface of the metal is no longer recognizable; or If the depth of penetration exceeds 10 %* of original thickness of wall ^b ; or If the wall thickness is less than design minimum wall thickness.	Repair if possible Repair if possible (repeat requirements of clauses 6, 7 and 8) Render unserviceable
Local corrosion	Loss of wall thickness over an area of less than 20 %* of the total surface area of the cylinder except for the other types of local corrosion described below	If the depth of penetration exceeds 20 %* of the original thickness of the cylinder wall ^b ; or if the wall thickness is less than design thickness	Repair if possible (repeat requirements of clauses 6, 7 and 8)
Chain pitting or line corrosion (Figure C.6)	Corrosion forming a narrow longitudinal or circumferential line or strip, or isolated craters or pits (Figure C.7) which are almost connected	If the total length of corrosion in any direction exceeds the diameter of the cylinder and the depth exceeds 10 %* of the original wall thickness ^b	Repair if possible ^c

^a If the bottom of the defect cannot be seen and if its extent cannot be determined using appropriate equipment, the cylinder shall be rendered unserviceable.

^b If corrosion has reached limits of depth or extent, the remaining wall thickness should be checked with an ultrasonic device. The wall thickness may be less than the minimum, with the acceptance of the inspection body, e.g. small (depth and extent) isolated pits. When applying the rejection criteria given in this table, the conditions of use of the cylinders, the severity of the defect and safety factors in the design shall be taken into consideration.

^c Provided that after repair by a suitable metal removal technique, the remaining wall thickness is at least equal to the design minimum wall thickness.

* If the manufactured wall thickness, i.e. the actual wall thickness on an area where there is no corrosion nor defect, approximates closely to the minimum design wall thickness, greater care shall be exercised in applying the above rejection limits.

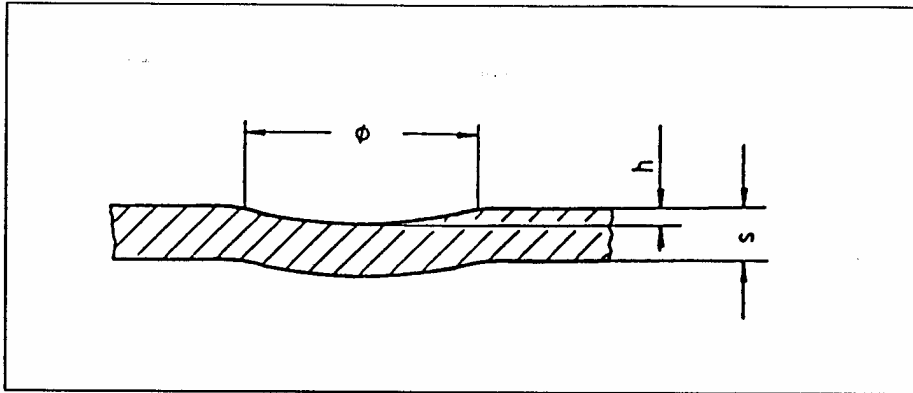


Figure F/1: Dent

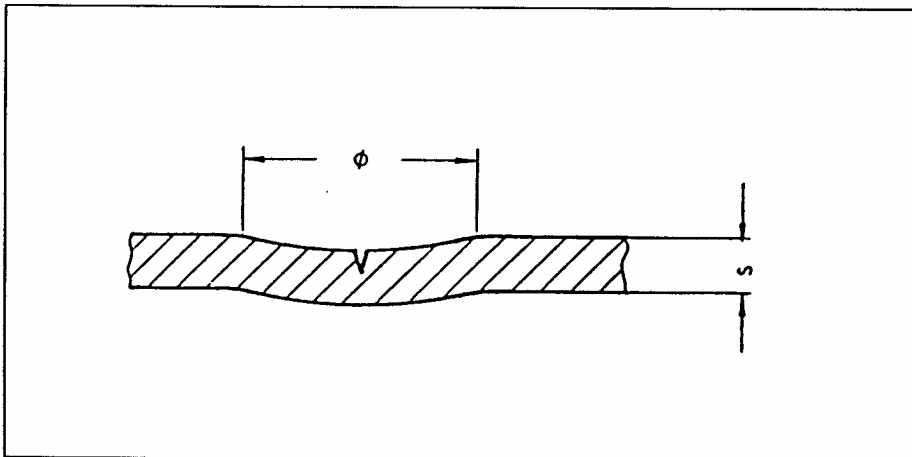


Figure F/2: Dent containing cut or gouge

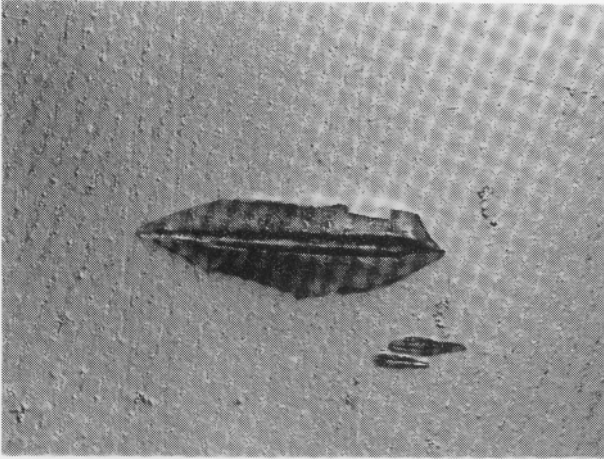


Figure F/3

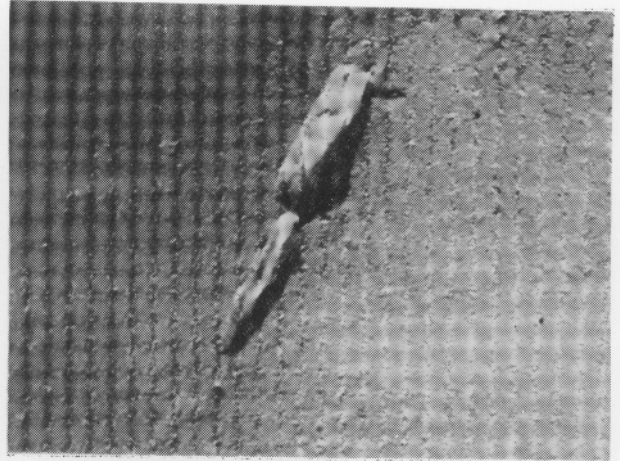


Figure F/4

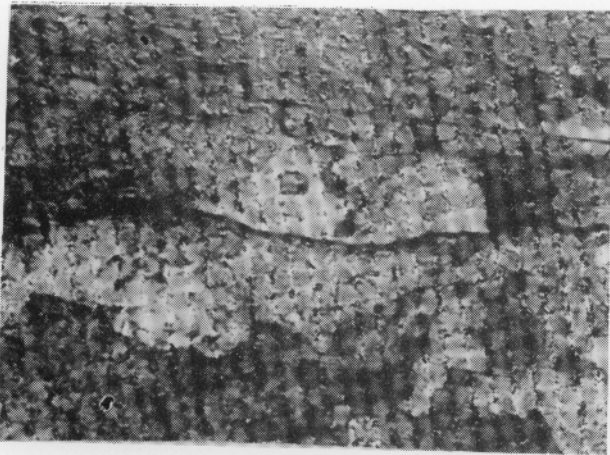


Figure F/5

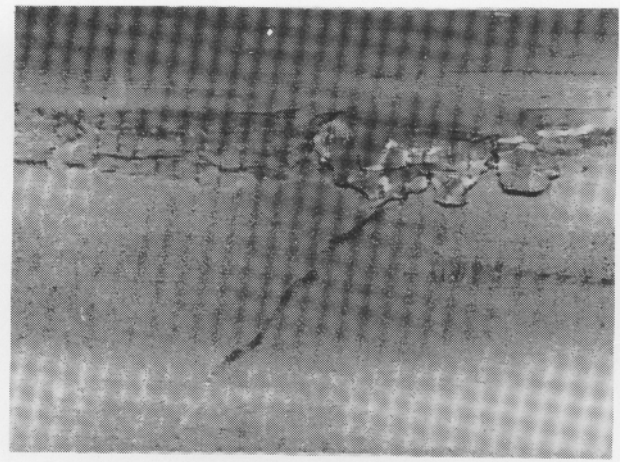


Figure F/6

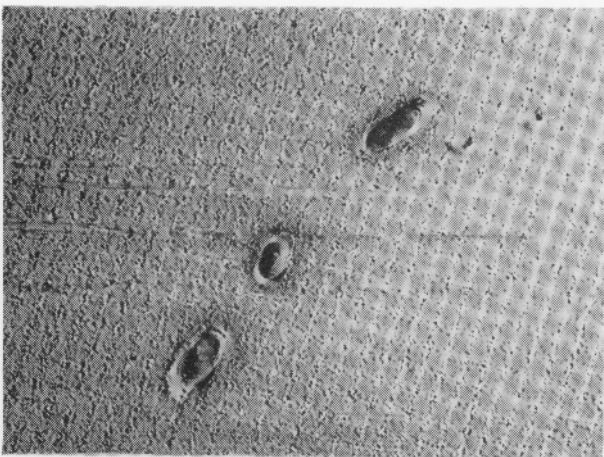


Figure F/7

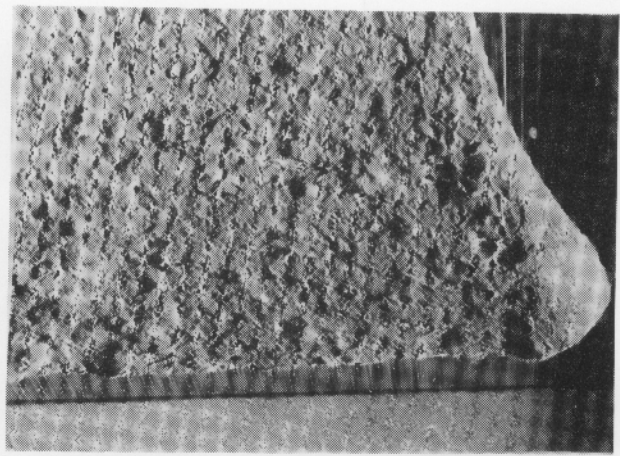


Figure F/8

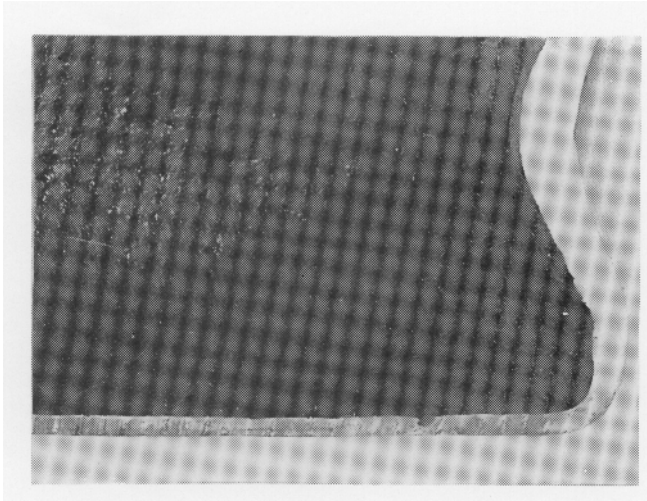


Figure F/9

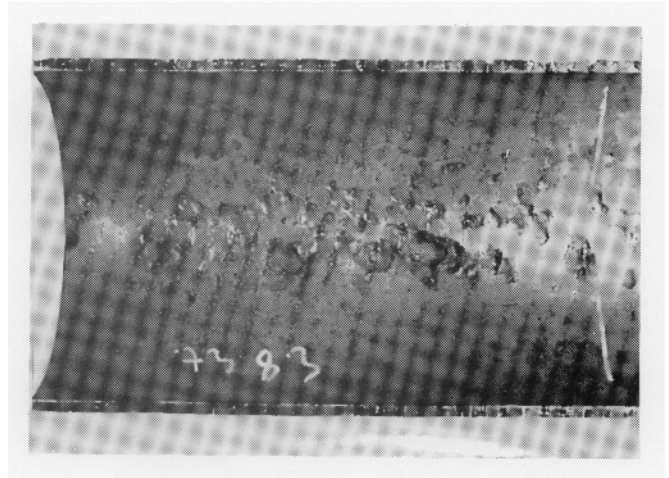


Figure F/10

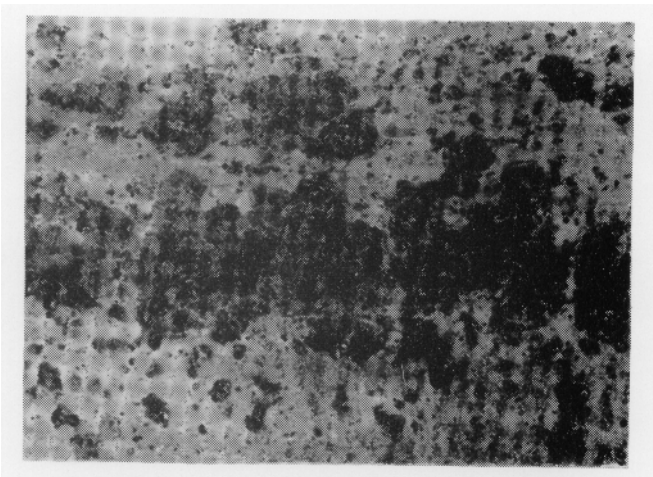


Figure F/11

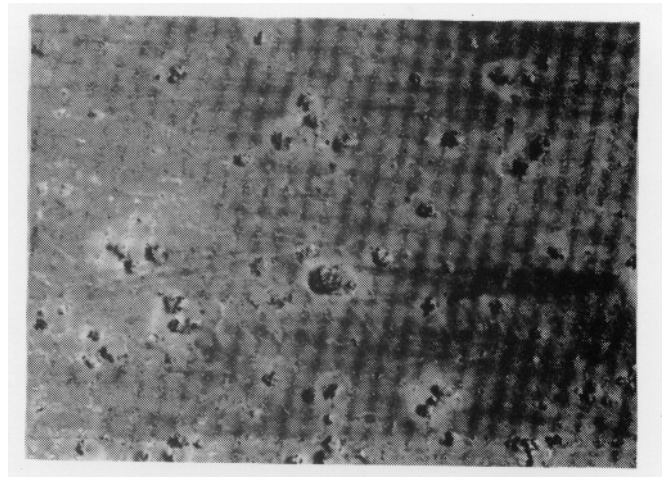


Figure F/12

Appendix G: Hardness testing and evaluation of test results for rejection of seamless high pressure hydrogen cylinders and transport vessels/tubes

16 Relationship between hardness test results and tensile strength values

The conversion of hardness test results into tensile strength values is subject to an unavoidable statistical error.

After having established the scatter band, the most representative correlation between hardness and tensile figures for a certain cylinder design, material and manufacturer shall be used for conversion, for example see figure G/1 [13] where \bar{H}_B is the average value of the hardness measured.

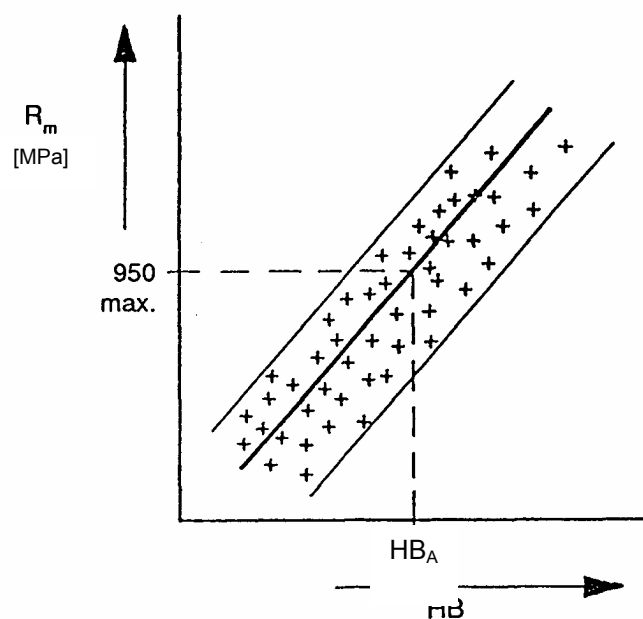


Figure G/1: Diagram for HB/Rm-Conversion

This correlation has to be determined, preferably by the cylinder manufacturer, using test pieces taken from manufactured cylinders.

NOTE- Identical Brinell- and Vickers-hardness numbers are equivalent to different R_m -values [13].

17 Technique of hardness testing

In order to obtain hardness test results during re-testing cylinders with an accuracy of about ± 50 MPa for a single indentation HB-value [14] the following should be considered:

- The largest possible ball diameter should be used to ensure accurate measurement and to minimise the influence of inhomogeneities in the microstructure of the material and in surface preparation.
Since the load increases with increasing ball diameter, consideration must be given to the mechanical integrity of the cylinder being tested, e.g. if a ball diameter of 10 mm is used, the hardness test in the middle of the cylindrical part of a 50 l cylinder is not practicable.
- The surface must be carefully prepared to remove the decarburised layer. The final surface finish should be in accordance with the recommendation of the manufacturer of the hardness testing equipment, in the absence of this the following table [15] figure G/2 may be used as a guide:

diameter of indentations mm	allowable roughness of the surface μm
5,0 - 3,0	15
3,0 - 2,0	10
2,0 - 1,0	5
1,0 - 0,6	3
0,6 - 0,4	2

Figure G/2: Allowable roughness of the surface

At least three indentations shall be made for one hardness value, so that the accuracy of that value is about ± 30 MPa.

18 Rejection Criteria

Where rejection criteria have not been agreed between a cylinder user and the appropriate national authority then the following criteria shall be used.

When testing the hardness of hydrogen cylinders distinguish between three groups of cylinders:

- Any cylinder exhibiting hardness values which clearly indicate that its tensile strength at all points is below the maximum agreed limit, can be safely returned into hydrogen service, e.g.
Example: measured hardness $< \text{HB}_A - 10$
- Any cylinder exhibiting hardness values which clearly indicate that its tensile strength at any point is above the maximum agreed limit shall be withdrawn from further hydrogen service, e.g.
Example: measured hardness $> \text{HB}_A$. Such cylinders may either be rendered unserviceable or reheat treated to meet the correct HB_A value or, following a successful ultrasonic test be converted into a non hydrogen related gas service.
- Any cylinder exhibiting hardness values which indicate that its tensile strength at any point is near the agreed limit can be returned into hydrogen service only after having passed a further examination for internal defects by non-destructive inspection method, following recommendation no. 2, e.g.
Example: $\text{HB}_A - 10 < \text{measured hardness} < \text{HB}_A$

The maximum acceptable hardness value, HB_A has to be determined according to chapter 23 of this appendix.

NOTE- In absence of the manufacturer's HB_A value for the type of cylinder to be tested, the maximum value given in the relevant IGC recommendation* shall be used as the maximum acceptable hardness value.

* for 34 CrMo 4 see chapter 23

The above classification is an empirical one and is based on sufficient experimental evidence . . . Some explanations are given in appendix H.

19

It is in the interest of the user to maximise the accuracy of the relationship between HB and R_m . To this end the user has at least two possibilities:

- Reference can be made to the original batch sample information that was supplied by the manufacturer.
- A random sample cylinder can be subjected to destructive tests.

Appendix H: Remarks on the relation between the hardness of the cylinder material and its susceptibility to hydrogen embrittlement

The susceptibility of a steel to embrittlement by gaseous hydrogen increases with increasing mechanical strength. This is a general tendency which has been proved experimentally [8, 16] and holds as long as the respective microstructure of the material remains basically the same.

For quenched and tempered cylinder steels, therefore, it is essential not to exceed a certain strength level.

For hydrogen cylinders, the ultimate tensile strength can be estimated only from subsequent hardness tests, making use of the above mentioned empirical relationships which have been established between hardness and ultimate tensile strength for a particular steel. Following this procedure, however, the reliability of the estimated results is limited, due, to the experimental scatter. In addition, general experience shows that even in one cylinder, the tensile properties may vary, to a certain extent, because of unavoidable changes in the course of the production process.

It should also be noted that apart from a certain susceptibility of the material towards hydrogen embrittlement, a critical surface defect is needed at the internal surface of a hydrogen cylinder before failure becomes probable.

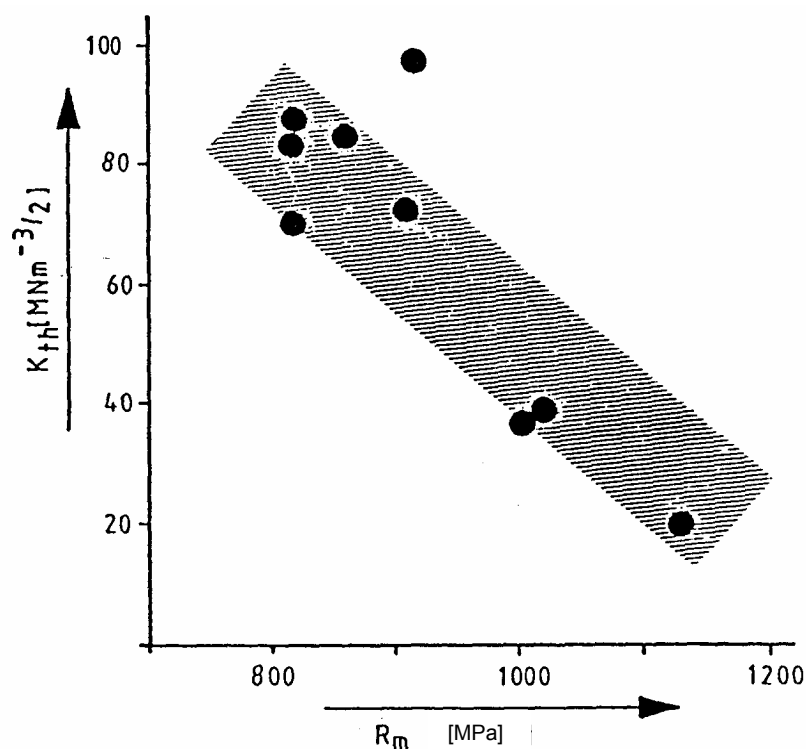


Figure H/1: Comparison of CrMo threshold stress intensity data

As an example figure H/1 shows the results of fracture mechanics experiments [8] which demonstrate how the threshold stress intensity for sustained crack growth in hydrogen decreases with increasing tensile strength of the steel.

From such findings it is evident that a hydrogen cylinder being made from steel with high tensile strength, necessarily requires more stringent criteria when examined for internal surface defects.

Appendix I: Literature

- [1] Fragilisation par l'Hydrogène des Aciers 30 CD4 et CDV 10. Mémoire présentée en vue d'obtenir le Diplôme d'Ingénieur C.N.A. Conservatoire National des Arts et Métiers Paris.
Par Pierre ANTONISSAMY.
- [2] Chambre Syndicale Nationale des Fabricants de Gaz Comprimés Liquéfiés ou Dissous.
Circulaire générale No 482, 31 mai 1972,
Bouteilles en Acier affectées à l'Hydrogène.
- [3] Gas and Cylinder Manufacturers Proposal for a European Directive on seamless steel gas cylinders regulations.
Available at: PUBLICATION DE LA SOUDURE AUTOGENE
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