REGULATIONS AND RESEARCH ON RC&S FOR HYDROGEN STORAGE RELEVANT TO TRANSPORT AND VEHICLE ISSUES WITH SPECIAL FOCUS ON COMPOSITE CONTAINMENTS

Mair, G. W. and Pöschko, P.
Department 3.2, Federal Institute of Materials Research and Testing BAM, 12000 Berlin, Germany, georg.mair@BAM.de

ABSTRACT

Developers interested in high pressure use and especially in mobile use of hydrogen rely on composite vessels for dangerous goods transport and onboard storage. Thus, composite materials and systems deserve special consideration. History gives interesting background information important to the understanding of the current situation as to regulations, codes, and standards.

Based on this review, origins of different regulations for the storage of Hydrogen as dangerous good and as propellant for vehicles will be examined. Both categories started out using steel and sometimes aluminium as cylinder material. With composite materials becoming more common, a new problem emerged: vital input for regulations on composite pressure systems was initially derived from decades of experience with steel vessels. As a result, both regulatory fields suffer somewhat from this common basis. Only later developments regarding requirements for composite vessels have begun to go more and more separate ways. Thus these differences lead to some lacks in regulation with respect to composite storage systems.

In principle, in spite of separate development, these lacks are in both cases very much the same: there are uncertainties in the prediction of safe service life, in retesting procedures of composite vessels and in their intervals. Hence, different aspects of uncertainties and relevant approaches to solutions will be explained.

1.0 INTRODUCTION

For the most part, pressure vessels used for the transport of dangerous goods (pressure receptacles) and for onboard storage of gaseous fuels (fuel gas storage containers, often spuriously called tanks) are currently made of steel. Depending on the region of the world, aluminium is also used to a lesser or greater degree. Composite materials for mobile pressure receptacles used with reference to the first approvals in the USA and Canada and since the beginning of the 1980's also in Europe are not a complete novelty today. There was, however, some initial insecurity in handling these materials.

Good reasons both for and against the use of composite vessels exist. The main argument against their widespread use is cost-related: the material, manufacturing and testing costs are higher than those of steel cylinders. The reason why this expensive type of cylinder construction is still being increasingly used is due to their low weight.

With the advent of composite cylinders, new design and test requirements were established by CEN and ISO. They partially accounted for the particularities of the material, yet these requirements did not deviate much from preconceptions and experiences based on manufacturing of metal pressure vessels. This also holds true for the standards concerning periodic inspections for pressure receptacles and fuel gas storage cylinders, as will be explained below. On the basis of insights gained from literature, experience, and plausibility considerations, fundamental differences can be found with regard to this particular aspect. Comparative examinations of exemplary cylinders yield particularly instructive results.

In 2007 a conference of the European Commission took place called “Hydrogen review days”. One result of the session on RC&S was this: current regulations mean big hurdles for or even prevent an optimised design of storage systems (see [1]). Another result was that storage systems for propellant gases were still too expensive and too heavy.

Therefore there are some additional attempts in the ongoing process on all fields of mobile hydrogen storage to improve performance based test procedures. Thus the confidence in simulating real operational loads during approval tests and in life time prediction based on brand new specimens should be increased.

A further point in the focus of today’s consideration is the quantification of residual strength for safety surveillance. While there are few doubts about the safety of brand new pressure vessels of an approved design, it still remains difficult to assess the safety of operated ones or their residual time of safe operation. Even though for metal pressure receptacles (transport of dangerous goods: TDG) and storage containers (onboard storage for propellant vehicles OSV) ultrasonic testing in combination with or instead of hydraulic testing has shown good results for more than one decade, no non-destructive technique is available and sufficiently mature for the assessment of composite pressure receptacles. On the contrary, most experts are convinced that hydraulic retesting – that is currently mandatory for the periodic inspection of pressure receptacles (related to TDG) – has negative impact on the strength while it does not yield any reliable result with the only exception of tightness for liquids. Therefore significant changes of RC&S are necessary to achieve reliable safety surveillance.

One proposal is to introduce a probabilistic approval approach (see [2]). That would give more freedom in adapting and optimising the design to users’ requirements and thus to save production costs while the safety according to a certain reliability level has to be proven by statistical methods. This means quantification of average and scatter values on an enlarged sample of prototypes during the approval testing stage. Disadvantages of this approach are a) more effort per test because some tests must not stop before a failure occurs and b) the increased number of specimens and tests.

Another proposal is to concentrate more on the safety degradation than on the prediction of life time as is currently done by using performance based approval tests on a large scale. For this approach it is essential to discuss relevant aspects of performance based on pre-conditioning and sufficient methods for quantification of residual strength. As long as no NDT method is available, destructive tests have to be used. Thus surveillance of degradation requires some kind of destructive tests parallel to operation.
The major advantage of this proposal is the high confidence in the assessment of degradation effects that allows a reduction of the high requirements on life time prediction at the point of time of design approval. The disadvantages of this approach are its logistic effort and the additional effort for destructive tests on additional samples of used pressure vessels.

Since none of these proposed approaches shows a significantly better ratio of advantages to disadvantages it is expected that a combination of all of these three aspects may become part of future RC&S.

Deficits appearing at all approaches are the lack of established test procedures appropriate for quantifying the residual strength of either operated pressure vessels or new ones.

2.0 SOME HISTORY FROM A GERMAN PERSPECTIVE

Since the first applications of steam as actuator for stationary engines (end of the 17th century), and later for mobile applications such as locomotives and steam boats, fatal accidents have been caused by insufficient strength against pressure loads. These accidents led to increasing but at first regional efforts to avoid accidents. Therefore regulations and inspection bodies were created and implemented.
In Germany, technical associations of steam engine operators were founded, with the goal to organise a high safety level at members’ facilities by abiding by their very own standards. These technical associations of steam engine operators have been so successful in increasing the safety level that they were rid of governmental inspections in 1871. Later on the Technical Supervisory Association (TÜV) developed from theses early societies.
In other countries like England owners were held more liable for consequences of accidents. This resulted in the necessity to found insurance companies and (consequence) classification societies. For this reason e.g. the UK developed a system of causers’ liability, while e. g. in Germany the focus was set on detailed approval procedures carried out by competent experts on behalf of the authorities, with the side effect of cost allocation between health insurances and pension funds.
Driven by different cultural backgrounds and different concepts, deviating rules and regulations concerning safe use of steam vessels and later, of pressure equipment, grew all over Europe and across

the world. In Germany, the first regulation for steam engines was enacted in 1908. This regulation had been developed from the rules of the Technical Supervisory Associations.

In most European countries, the first regulations were created to achieve control over the use of pressure equipment with respect to labour aspects. When the first regulation for the approval of pressure cylinders was enacted in Germany in 1968 ("Druckgasverordnung"; see [3]), a global system of approval and retesting by authorised experts was installed. Until most member states of the European Union decided to join the international agreements for the transport of dangerous goods on road (ADR 1957; current version see [4] nationally implemented by [5]) and on railways (RID 1980 based on the "First Berner Agreement" on the international goods transport by railways (CIM) from 1893), the transport of gases from one country to another had been more or less impossible.
Then, in the mid-1990s, the frame work directives of the European Union for transport of dangerous goods on roads and on railways initiated some changes by implementing international agreements for the TDG within the EEC (European Economic Community): the international agreements began to be used for national transport and thus a big step of harmonisation was achieved. Under these agreements, it became possible to transport gas cylinders from European gas suppliers to consumers all over the ECC without multiple approvals within the EEC. Then transport became even more flexible by the introduction of the so called "epsilon" cylinders, gas cylinders built in accordance with one of three directives (84/525…527/EEC) that give requirements on design and testing of seamless steel, welded steal, and aluminium gas cylinders as we know today from referenced standards. Cylinders tested according to these directives are very heavy but have in most cases excellent safety properties. From this starting point, an important impetus as to the standardisation work at CEN was set.

By applying RID/ADR as first harmonised regulations on TDG, the differences between national regulations all over Europe became obvious. But because of the focus on labour safety in most European countries, operating (especially filling) of pressure receptacles not approved or retested in line with national regulations remained prohibited until the directive on transportable pressure equipment (TPED; see [6]) was introduced into national laws of the EU member states. TPED was to be implemented for transportable gas cylinders not later than July 1st of 2001. The TPED uses the Greek "Pi" as conformity symbol and is not a directive based on European agreements on the common market (compare: Art. 100a EGV (95 EUV) symbolised by CE-marking). The TPED is a directive based on the agreement on harmonisation of regulations for (safe) international transport (compare: Art. 75 EGV (71 EUV)). Simultaneous to this development, the basics of approval regulation for pressure receptacles in Germany were removed from the law on labour safety and work protection and introduced into the law on transport of dangerous goods. Before that, gas cylinders approved according to workers' safety regulations ("Druckbehälterverordnung") had been permitted to be used for automotive applications. By implementing the TPED it became necessary to harmonise regulations for onboard storage containers like ECE R 110 separately.

This is the situation as we experience it today and as it is being discussed in meetings concerning regulations for TDG and for vehicle approval regulations. Regulations on hydrogen storage systems for stationary application are based on a European directive (97/23/EC: PED). Nevertheless refuelling stations and other facilities with high hazard potential have to be approved and supervised by local authorities. Thus, it is evident that the use of pressure equipment for stationary application is neither structured nor harmonised on a level equivalent to that of either of the other fields of regulations (for TDG or vehicles). The field of TDG is dominated by international harmonised regulations for the land mode (RID/ADR), the sea mode (IMDG-Code; see [7], nationally implemented by [8]) and the air mode (ICAO-IT or IATA Dangerous Goods Regulations; see [9]). But all of these are based on the UN Model Regulations, called "Orange Book" (see [10]). After having integrated technical requirements of the TPED into the RID/ADR, the European Commission revised the TPED and published the Directive 2010/35/EC (see [11]). In the field of hydrogen vehicle approval in Europe there is only the EC-Regulation 79/2009/EC in combination with Regulation (EU) No. 406/2010. In addition, there is a task group associated with Working Party 29 of the UN ECE, called Subgroup on Safety (SGS) of Hydrogen-/Hydrogen Fuel Cell Vehicles which has been entrusted to create a world wide Global Technical Regulation (GTR) for the approval of hydrogen vehicles. But it is not clear whether this group will be successful in harmonising all the different regulatory approaches and interests with respect to economical and safety issues within the limited time frame ending in 2011 or even within a possible extension. Europe has expressed that the European vehicle regulation will be substituted partly or totally by this GTR as far as relevant items are covered and the achieved safety level will be at least equal.

Since all the regulations attempt to achieve or to keep up a high level of safety, it is necessary to discuss the meaning of safety.

3.0 WHAT IS SAFETY?

The use of the term "safety" is diverse. Everybody uses this word in everyday life to express that, based on their experience, they do not expect an incident. But very often it is used to express the expectation that nothing can happen. As we know such an expectation is not realistic at all. Every technology and their uses come with the potential of a minor or even a major accident – possibly involving casualties. That is only a question of frequency or probability, which is to say, a question of time.

Therefore it is necessary to define the term "safety". This term describes a situation that exists in the absence of "danger". The situation of "danger" exists in the case of risk levels being higher than the respective "risk limit" as is accepted by law and/or the public. This risk level has not necessarily been put down in writing. It may be solely based on a common understanding among citizens.

Also the term "risk" in everyday life is often used in different meanings, e. g. "the consequence is zero". Scientifically it is the complement to "chance". That can be expressed as follows: If you take a chance you always should be aware that something may go wrong; or if you try to take advantage of a tool you accept the risk of getting injured by this tool. "Risk" is ultimately described by the product of frequency of an incident and consequence of this incident. Thus the frequency of an incident with small consequences (cutting a finger with a knife) is usually much higher than the accepted frequency of an accident with high consequences (e.g. worst case burst of a hydrogen onboard storage container). By the way, independent of frequencies "almost zero", which are often irrationally treated as "will not happen" there are accidents of such high potential consequence that the technique is not accepted – regardless of predicted frequency/risk values – or tolerated by a major part of inhabitants at all (e. g. nuclear power). In their view no one can predict when a very improbable accident may occur. Thus use of technologies with extreme consequence potential is de facto not accepted or is looked upon very suspiciously by the public. The accident of Fukushima shows in addition that, caused by unpredictable circumstances, frequency of such failures may be much higher than originally expected and "almost zero" is not zero.

The potential consequence of hydrogen storage systems is high but not extreme and can be controlled via design properties concerning probable failure modes. The second part of risk control of those systems can be managed via the frequency of system failures. This means with respect to e. g. fatigue failure that a crack growing that leads to a rapid rupture shall be managed with a different reliability or survival rate (safe life design) than a fatigue crack that does reliably end in a leak and not in a rupture (fail-safe/damage tolerance design) to meet the same risk level. Therefore each type of composite cylinder designs, whether it is hoop wrapped or fully wrapped, whether it has a load sharing liner or a non load sharing liner, must be able to meet different requirements concerning minimal strength. Since such interaction between type of design and failure modes is not compulsory, in the end the properties of all designs should be discussed individually.

As soon as we try to quantify the frequency of failures for the purpose of risk assessment (or a step further, risk control) we have to use statistical tools. In cases of insufficient data on failure frequency over the course of the entire period of operation, as is currently the case for hydrogen onboard storage systems, it will be necessary to predict the failure frequency by probabilistic tools – preferably for each design.

4.0 PROBABILISTIC APPROVAL APPROACH (PAA)

4.1 Introduction

Demonstration of safety properties by mainly destructive tests within a probabilistic approval approach as proposed here is intended to guarantee a safety level at least equal to the requirement of the rules applying to the approval of hydrogen vehicle components and systems. Finally it should guarantee the adherence to a certain risk level as it may be required in future regulations. A global probabilistic approach requires covering all aspects by statistical means, even the accidental ones. With respect to preceding statements on this (see [2], [12], [13]) for making first steps into regulations, in the following the focus is set on those tests that reflect operational loads under normal conditions.

The general concept of the probabilistic approval approach in line with [12] - [16] is to perform the essential tests in such a way that the load that addresses one or even more detailed failure procedures has to be sustained continuously until final failure. The first or, if necessary, the sequence of failures has to be monitored and evaluated in a statistical manner so that the probability distribution can be described by parameters in conjunction with the exact loads and other circumstances of testing. The significance of a certain test depends on the number of tests. In this way, a higher number of demonstration tests (samples) yields a lower uncertainty. It might be left to the manufacturer to decide upon the size of the samples and to benefit from the given safety level for systems produced in large-scale production. Thus the probabilistic approach might be of more interest for the automotive industry than for the transport of dangerous goods.

At this point it is necessary to mention that statistical tools are available for determining the minimum or optimal sample size. But on the one hand a procedure for the determination of samples sizes implemented into regulations has to be built on statistical experience with relevant tests and specimen design type properties. On the other hand, theory without sufficient experience on real scatter would detract from first steps like the search for appropriate test procedures.

The testing procedures may be described in a separate mandatory annex of a regulation itself or as a mandated standard, which has to be approved by the regulators and referenced in regulations in a dated version. In this way, the limits of safe use can be determined for each design type of storage containers. The central questions of safe use, pressure level, maximum filling temperature, life time, and accidental loads can be answered in a satisfactory manner.

Various standardisation and regulation bodies all over the world are reacting to the increasing need for improvement of approval requirements: On the one hand, some minimum requirements might be too stringent. Thus they might cause unnecessarily heavy weight and expensive storage systems. Therefore, mandatory test procedures have to be defined in such a manner that the potential of material reduction can be utilised by intelligent design improvement. On the other hand, there are justified doubts whether current test procedures and safety factors are able to guarantee the necessary safety level under all normal conditions. This means the safety level of the regulations in question must be sufficient for the expected percentage of composite vessels (including hydrogen vehicles within the next 10 years at least) such that the perception of inevitable incidences does not endanger the acceptance of composite vessels or hydrogen vehicles.

Regarding loads which may lead to a sudden rupture, the focus on fire, crash, and pressure loads – more or less static loads during emptying and cyclic loads due to refilling – should cover the most relevant safety aspects as a first step. Crash and fire engulfment aspects are mainly determined by the properties of the whole system, including e. g. the vehicle chassis or thermally activated pressure relieve devices (TPRDs). These have to be tuned to the properties of the storage containment. With regard to these aspects it is necessary to know how to balance these systems and how to prove their functionality on the necessary level of reliability in conjunction with relevant accident statistics.
The operational loads of the storage systems mainly depend on filling stations (maximum filling pressure; developed gas temperature), and users' operating profiles (mileage, pressure before filling). In the following it is attempted to demonstrate the relevance of statistical aspects of safety with respect to cycle fatigue and required filling cycles.

For the purpose of a basic discussion of principles it is exemplarily assumed that a number of 1,000 filling cycles is sufficient for nearly all non commercially driven vehicles. For safe and cost optimised designs, the number of fillings has to be limited to a maximum number of filling cycles by technical measures. Also, the maximum pressure and temperature have to be strictly limited. This combination of load aspects describes the main point of operation. Based on these measures, the safety level can be described by reliability against leakage or burst at this point of operation only.

4.2 Aspects of survival rate and fatigue life of materials

Figure 1 shows the interaction of load cycles, survival rate, and statistical variations in terms of the spread of distribution which is called "Streuspanne" for material specimens (referred to as T_N). T_N is defined as the number of cycles at 90% survival rate divided by the number of cycles at 10% survival rate; this is a common parameter for the discussion of the distribution of load cycle values in a logarithmic fatigue curve known as the S-N curve (WÖHLER-diagram):

Spread of distribution "Streuspanne": $T_N = N_{90\%} : N_{10\%}$ (1)

This can be converted to log-standard deviation by: $\sigma_{\log N} = \left| \frac{1}{2{,}56} \cdot \log_{10}\left(\frac{1}{T_N}\right) \right|$ (2)

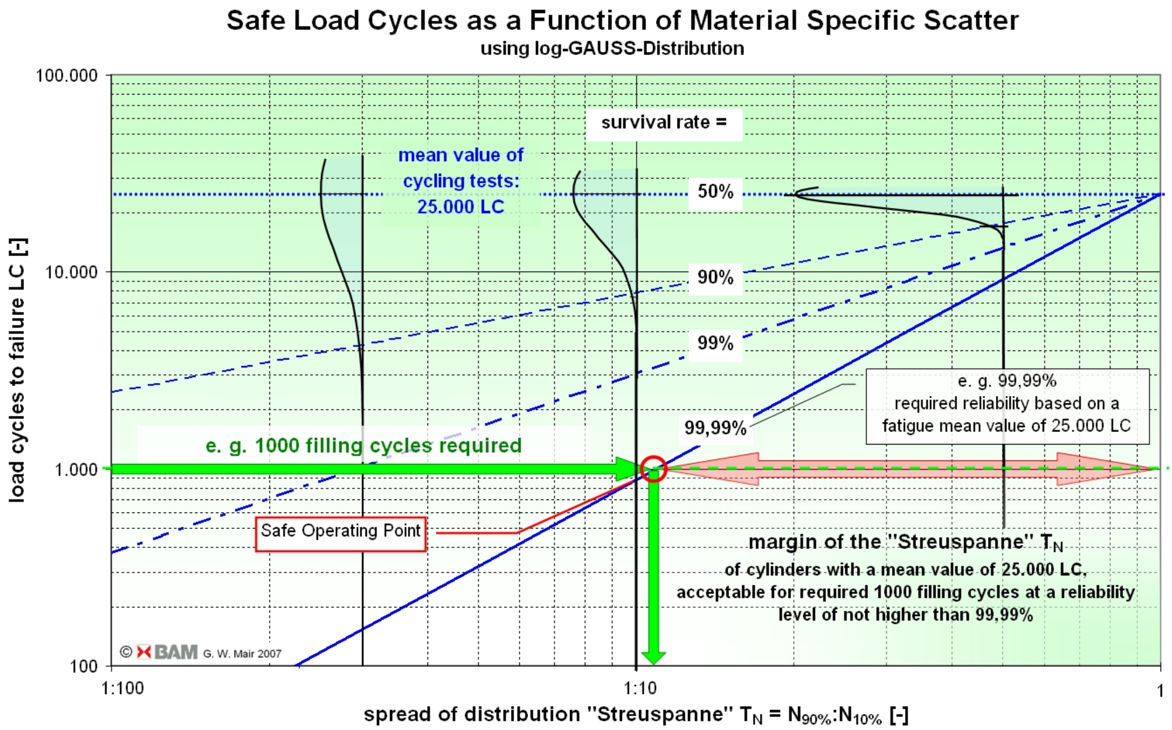

Figure 1: Mathematical analysis of GAUSS standard deviation based on 25,000 LC mean value

Fig. 1 is exemplarily based on a mean value of test results concerning fatigue life of 25,000 cycles. The interaction with static load aspects and other degradation processes is not taken into account. The diagram shows the interaction between load cycles *LC* (or sometimes *N*) and survival rate *SR*. The smaller the T_N-value, the lower the guaranteed cycle number N_S at the relevant survival rate. The margin acceptable for T_N as a material property (red arrow) is shown as an example for the requirement of 1,000 load cycles and addressing a reliability level of 99,99% (green arrows).

For an easier interpretation of the following scenarios reflecting cylinders safety based on material properties, three simplifying assumptions have to be made:
It is assumed that the manufacturing process of the cylinders is highly accurate and generates only a negligible scatter adding to the material properties distribution. Thus the **material specific scatter is used for the cylinder** as a first approach. Experience shows that vessels often exhibit a lower scatter than relevant material probes.
In line with this first assumption, the survival rate of material can be discussed as reliability level of a cylinder at the above mentioned operation point. It is assumed that fatigue failure of the containments

means leakage and does not lead to burst. The reliability against sudden burst has to be much higher than against leakage anyway; however, this will not be part of the following considerations.

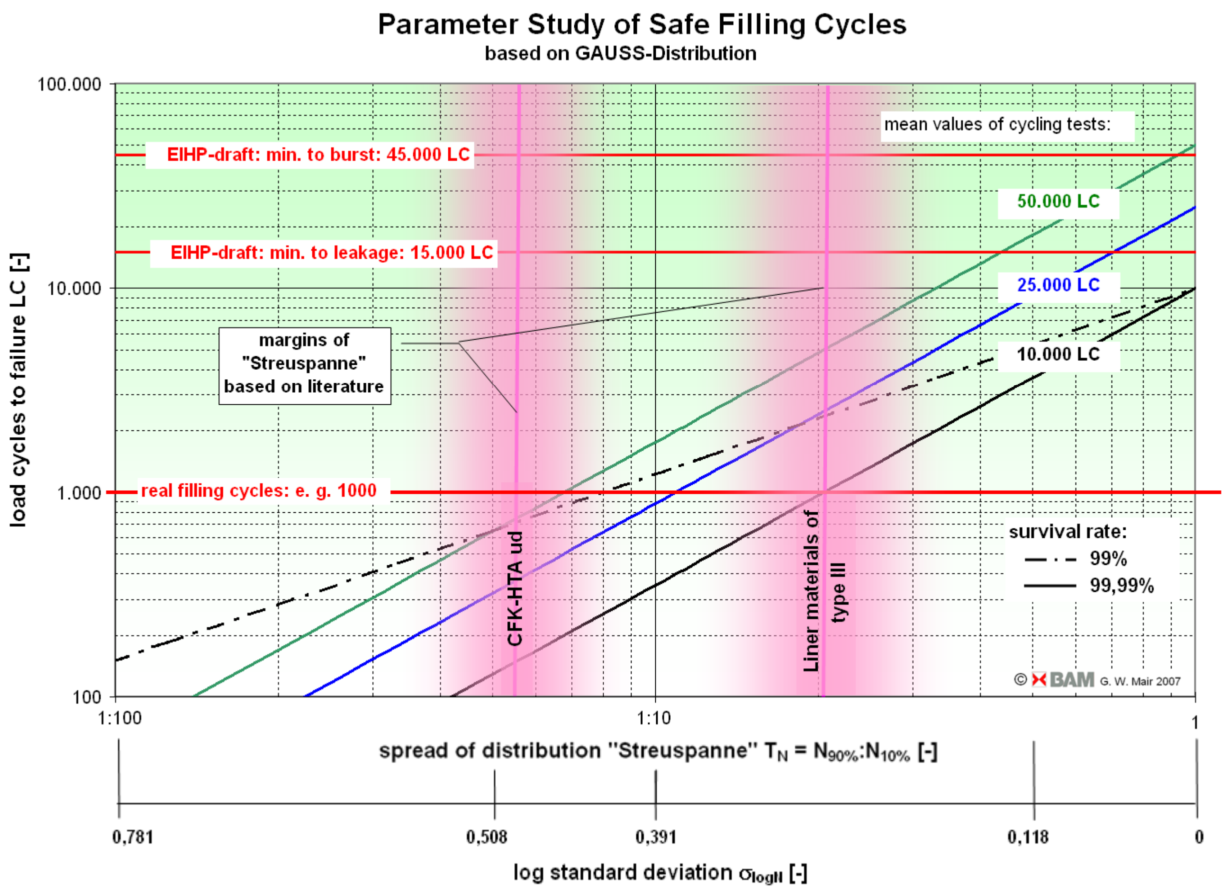

Figure 2: Safe number of fillings depending on mean values of fatigue figures

Figure 2 shows the principle of maximum allowable load cycles as function of T_N for a set of three selected levels of mean values. Additionally, average values of T_N of metal liners (as currently the weakest part of type III cylinders) and of carbon fibres (as the only load sharing material of type IV cylinders) are illustrated for comparison.
Due to the low strength degradation of CF by cyclic loads, T_N of these fibres is relatively high (see [17] and [18]). In cases of matrix or plastic liner failure with the consequence of leakage, the relevant T_N-value of type IV cylinders might be comparable with the one gained from type III cylinders. For the aspect of sudden bursts, a safety goal of 99.99% reliability at 1,000 filling cycles will not be acceptable anyway.

If it is not possible to increase T_N-value “Streuspanne” by decreasing the material scatter of a cylinder or to reduce the requirement of minimum guaranteed filling cycles, the design must be improved in such a way that the mean value of fatigue life of the cylinder increases to the necessary level.

The leakage scenarios addressed in fig. 2 points up:

Type III cylinders with metal liners such as AA6061 and with CF-wrapping require an average of cycles to leakage of least 10,000 (set of black lines) at a spread of LC-distribution of the liner material of about T_N = 1:9, as shown in fig. 2 or higher (means e. g. T_N = 1:5). A number of filling cycles of e. g. 1,000 does not in this case result in leakage with a probability of about 99.99%.

Type IV cylinders with CFRP show a low strength degradation under cyclic loads. Because of this the spread of material LC-distribution is much higher than with metal liners. Fig 2 shows a spread of about

T_N = 1:50. Thus type IV cylinders have to exhibit a much higher average of pressure cycles to leakage for demonstrating a probabilistically comparable safety level of e. g. 99.99% at 1,000 filling cycles.
Current regulations and design standards are based on fixed cycling numbers to leakage without taking into account the different cylinder types and material properties. Hence, these requirements can not guarantee the same safety level against leakage for type IV as demonstrated for type III by exclusively taking into account the behaviour of fatigue to leakage.

In fig 2 the blue line represents a design with 25,000 LC fatigue mean value. For type III cylinders with metal liner and a spread of LC-distribution of not more than about T_N =1:9 a number of filling cycles of 1,000 does not result in leakage with a probability of much higher than 99.99%. If the filling cycles were be limited by technical measures this value would open up the possibility of reducing the strength of the composite layers such that 1,000 cycles at 99.99% will be met without additional margins of load cycles. Reduction of strength often means a reduction of wall thickness.

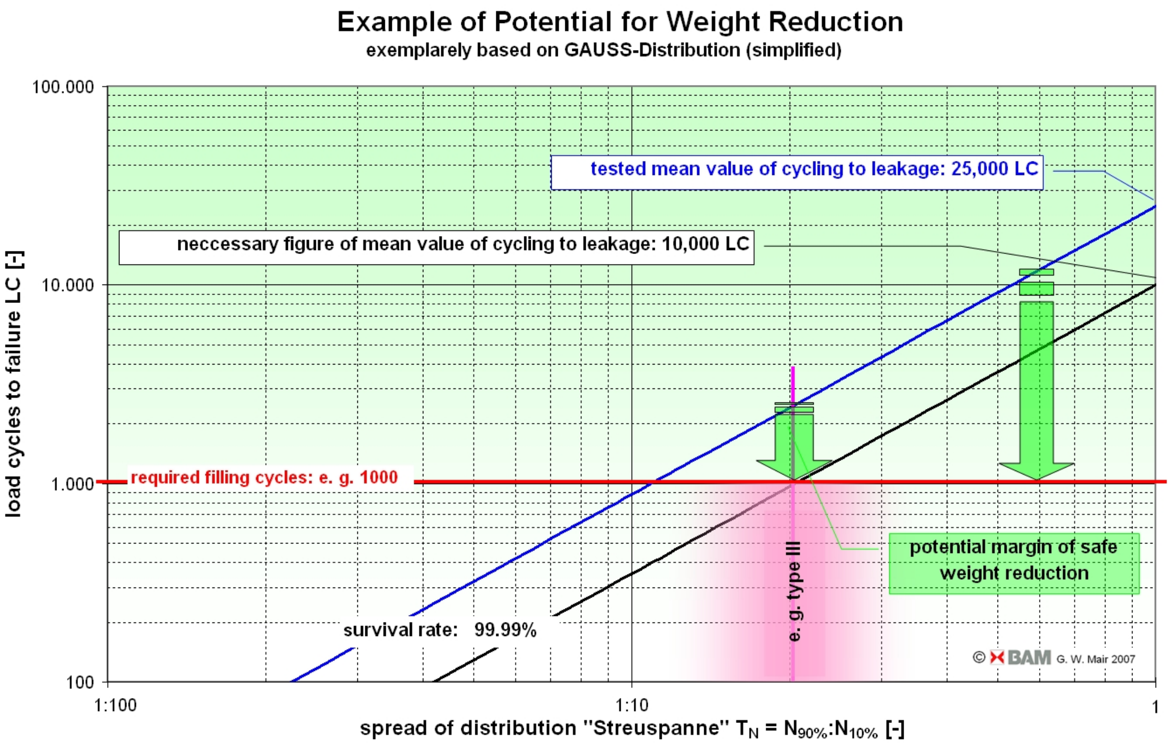

Figure 3: Potential for weight reduction based on cyclic aspects

Figure 3 shows two lines of 99.99% survival ratio: one is based on a mean value of fatigue life of 25,000 LCs (blue line), the other one is based on 10,000 LCs (black line). Currently some type III cylinders demonstrate 45,000 LC at ambient temperature and also accomplish 25,000 LC at -40°C. Thus at a T_N-value of about 1:9 or higher (e. g. 1:5) the surplus of fatigue strength could be reduced to the intersection with the limited number of filling cycles of 1,000 (red line). This potential is indicated by the shorter left green arrow; it might allow a reduction of fibre material. In cases of lower scatter the potential (distance from blue to red line; the right green arrow applies for T_N = 1:5) increases because the minimum required mean value can be reduced. The related total amount of fibre reduction can not be quantified blanket. It depends on design and manufacturing aspects and on approval requirements.

As has already been mentioned, it is not generally acceptable to use solely material data for the assessment of fatigue behaviour of cylinders. So ultimately it will be necessary to change the testing procedures and to evaluate relevant properties of the manufactured hydrogen storage containments in comparison with material properties. But nevertheless the principle of saving material can be explained by using material properties and will work on cylinders in principle.

4.3 Aspect of Cost Reduction

There is great potential for weight and material reduction when evaluating a good design and a smaller or perhaps “negative” potential by assessment of a poor design.
The necessary supplement of tests will cost additional money, as will the effort in R&D for cylinder improvement. So it is worth investigating the interaction of cost reduction and of additional investments like R&D and approval testing. This is shown in principle in f**igure 4**. Here the investment costs and the cost reduction per vehicle are diversified. The resultant costs are calculated as a function of vehicles manufactured with one type of containment. The break-even point at different production volumes varies between 150 vehicles at an additional investment of 100,000 € and a saving of 15% of CFRP-wrapping and about 10,000 vehicles at an additional investment of 500,000€ and a saving of 1% of CFRP. An expected reduction by 5% with a total production volume of 2,000 vehicles diminishes the resultant costs by some 100 € per piece in spite of the additional investment costs of 250,000 €.

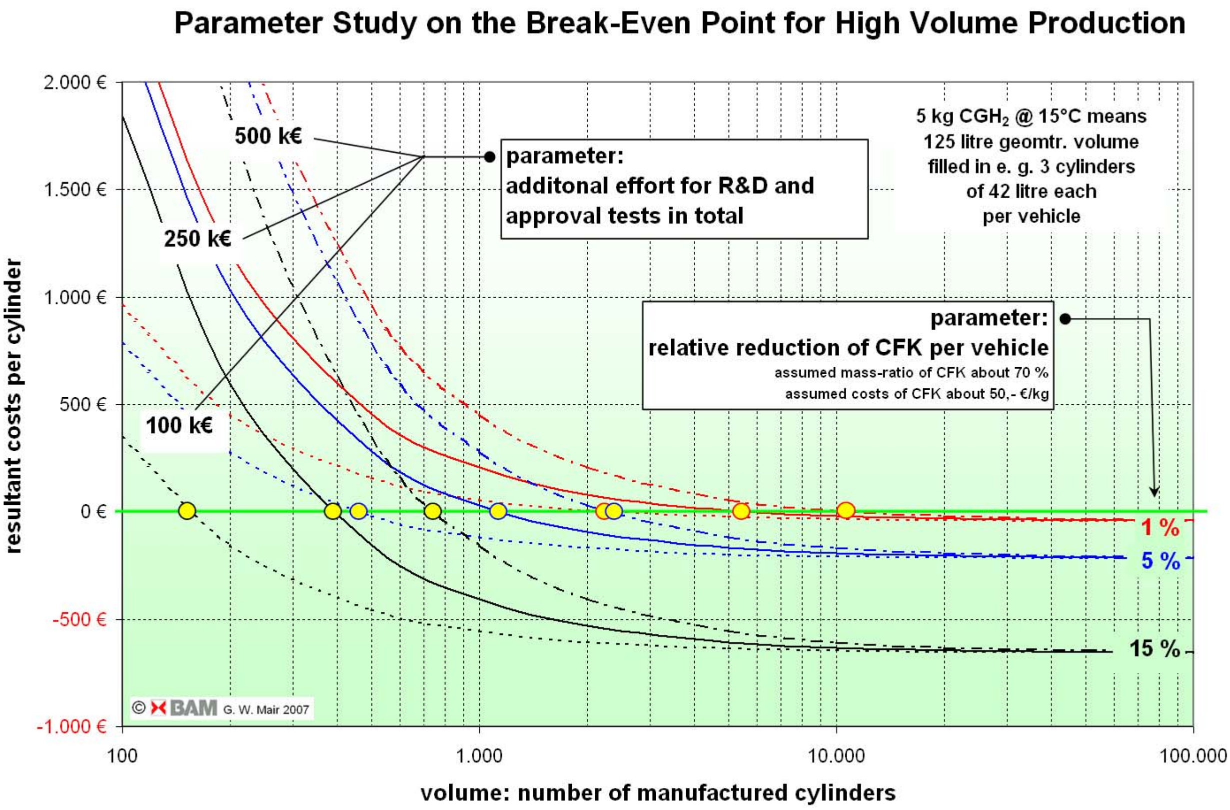

Figure 4: Break-even point of higher design and approval effort

4.4 Conclusions on the Probabilistic Approach and Outlook

A concept of probabilistic design and test procedures was developed for aeronautics first. Over the course of the last decades it has become more and more common for design optimisation. Experience shows that quantitative risk assessment based on probabilistic procedures is prospectively the most promising procedure for a reasonable safety assessment of designs. It also hints to an insufficient degree of safety in some cases but in most cases, material could be reduced by probabilistic optimisation and approval requirements.

For this purpose, statistical validation of cyclic and static fatigue properties of cylinders is necessary – the choice of the statistical deviation employed has to be based on the distributions of test results (e. g. WEIBULL). Statistical evaluation shows the survival ratios of virgin cylinders at the determined limit of

filling cycles (operation point). This is an important issue when it comes to risk management aspects.
The probabilistic approach takes into account the material, design and manufacturing specific scattering of strength. Thus the currently necessary and existing but not sufficient differentiation in the regulations concerning material (stress ratios depending on the fibre material) or design based safety margins (number of required hydraulic load cycls) would become obsolete (see fig. 2).
The probabilistic approach allows reducing unnecessary material consumption towards the required safety level (see fig. 3). Thus gas driven vehicles would finally become cheaper while maintaining or increasing the safety level (see fig. 4).

Other aspects of degradation of strength and stiffness have to be dealt with in parallel or as a kind of pre-damaging related to destructive static or cyclic fatigue life tests.

A probabilistic approval approach allows a task sharing which is different and seems to be helpful for all parties involved:

- ✓ The vehicles manufacturer, who is responsible for the determination of the actual number of refuelling cycles;
- ✓ The cylinders manufacturer, who is responsible for the demonstration of sufficient safety properties of the cylinders, which requires a standard-based third-party-testing;
- ✓ The regulatory authority, who is responsible for the determination of required reliability levels including requirements for the technical measures which have to be taken in order to limit the number of refuelling cycles.

The aspects of burst caused by fibre failure due to static and cyclic loads should be treated similarly. But the strength degradation by static loads and the degradation of composite stiffness in operation need basic research beforehand.

In the end, safety depends on scattering of strength properties and loads at least as much as on the properties that need to be proven in line with current regulations.

5.0 PERFORMANCE BASED TESTS – SIMULATION OF OPERATIONAL LOADS

Containments for mobile storage of hydrogen are exclusively tested in line with so called performance based tests. These test procedures intend to simulate operational loads as to intensity and frequency as they will occur during operation.
Thus it becomes obvious that there are three aspects very important to the adequacy of relevant requirements in RC&S:

Which kind of load should be simulated during approval tests and how to best simulate these loads?

What is an adequate frequency of periodically charged loads to represent expected maximum life time?

To what level should the relevant load amount in order to guarantee intended safety levels?

Since it is always ambitious to cover a wide range of different forms of operation by general figures, the answers to these questions can not be given easily. Composite paint ball cylinders constitute an excellent example. These cylinders are mainly built in line with standards as mandated or referenced for the TDG. TDG-cylinders are quite possibly not being filled more often than 10 - 20 times per year. Thus within an approximate 50 years’ life span, the total number of fillings is not expected to be higher than 500 - 1000 before rejection by periodic inspection (metal: fatigue cracks, corrosion, or scratches). The number of hydraulic load cycles shall be 12,000 for cylinders with a non limited life (see EN 12245: 2009; see [19]). This implies a factor of 12 from hydraulic load cycles to filling cycles. Bearing in mind the above explained interaction of mean value of fatigue strength and reliability, it becomes obvious that this factor of 12 states a safe level if the assumption can be confirmed that, if the small number of specimen shows no failure until 12,000 LC, the mean value of the population is higher. (Remark: This is an assumption not confirmable in general that has been one of the very reasons for the proposal of a probabilistic approach.) But the same paint ball cylinders may be refilled at a commercial paint ball site a few times a day. This may add up to 500 refuelling cycles per year easily. Consequently, the number of cycles that results from experience in the transport of industrial gases might be achieved within one or two years – but it is still a design with non-limited life. Formally those paint ball cylinders (in Germany) are subject to additional requirements as work equipment. This is especially relevant for the use and refilling at commercial sites without ever going anywhere else. But since there is no NDT method like e. g. acoustic emission (see [20]) which is sufficiently sophisticated to monitor the safety level during operation of composite cylinders, these additional requirements do not help as long as the major part of safety and the end of use is in most cases provided by design type testing.

Thus it became logical to ask for destructive tests parallel to operation in order to truly assess degradation as long as NDT methods do not yield sufficiently reliable results.

6.0 TESTING PARALLEL TO OPERATION (TPO)

Bearing in mind the deficits and uncertainties of design type tests and the associated life time prediction, it is logical to introduce two or at least one spot sample for intermediate checks of the safety level. Especially for composite cylinders, which undergo degradation under cyclic as well as under static loads, for prediction issues residual strength and safety have to be checked in comparison with properties of new samples. These more or less most important tests of the design type procedure have been called "key tests". By repetition of these key tests after e. g. 1/3 and 2/3 of the originally predicted life time, cylinders can be reassessed in a safe manner and degradation under operational loads can be monitored. In principle, degradation during operation should be comparable with the artificial degradation effects during design type testing. Thus some designs may face a stringent reduction of approved life span; others' life span might be extended. But ultimately for composite containments, "non-limited life" is physically not possible and therefore those approvals – as they are routine for pressure receptacles (TDG) – should not be possible any longer. Life time may be figured to be as long as 30 or 50 years, but it should consequently not be called a non-limited life any longer – as is already praxis with onboard storage systems.

An important issue of this concept is the performance of the appropriate key test. In this field BAM (Federal Institute of Materials Research and Testing; Germany) conducts ongoing studies with respect to cycle and burst tests. Final test results can not be shown yet. But one important point has been confirmed: While cycle tests on containments made from metal or on composite cylinders with metal liners in all cases (currently more than 100 at BAM) led to fatigue cracks, composite cylinders without metal load sharing liners must be treated in a different manner. For demonstrating statistically sufficient cycle numbers, the effort is (at least with respect to the high scatter values of composite fatigue strength) unacceptably high. The effort for sustained load or creep tests is also unacceptable due to the time to rupture. Thus BAM propose to use very slow burst tests with a constant pressure increase over the course of 8 to 100 hours. The pressure increase rate (pressure rate) has to be kept low enough that creep and static fatigue effects of fibres can continue to grow from the current state of degradation.

An additional step that increases the efficiency of those tests is an early classification of design types as "cycle fatigue sensitive" or "non cycle fatigue sensitive" as mentioned in [18] and described in detail in [21]. It is intended to assess cycle fatigue sensitive designs by cycle tests to leakage and non cycle fatigue sensitive designs by slow burst tests as described above. This classification allows testing all design types with respect to identical criteria without reflecting material or load sharing aspects.

This leads directly to detailed explanations concerning the analyses of residual strength.

7.0 TEST PROCEDURES FOR QUANTIFICATION OF RESIDUAL SAFETY

For the well investigated pressure receptacles and storage containers made of steel or aluminium or for composite pressure cylinders or storage systems with load sharing metal liners, the hydraulic cycle test allows testing specimens until the point of failure and to quantify the original strength of new specimens and the residual strength of operated ones such that it can be assessed statistically.

For the other types of composite containments, due to effort and time limitation the cycle test in most cases can not be continued until relevant failures, neither until leakage nor rupture. This means that collection of relevant data and statistical analysis are not possible. In this case the burst test often has been and is still being used for the assessment of strength and residual strength. But this goes along with significant mistakes. First of all, praxis often shows an increase in burst pressure during the first years of operation. Second of all, relevant regulations and standards do not describe the parameters for burst tests in a way that they can be reproduced reliably: there are limits for the maximum pressure increase rate only, and these are set so high that most test facilities are anyway incapable of coming into conflict with those requirements.

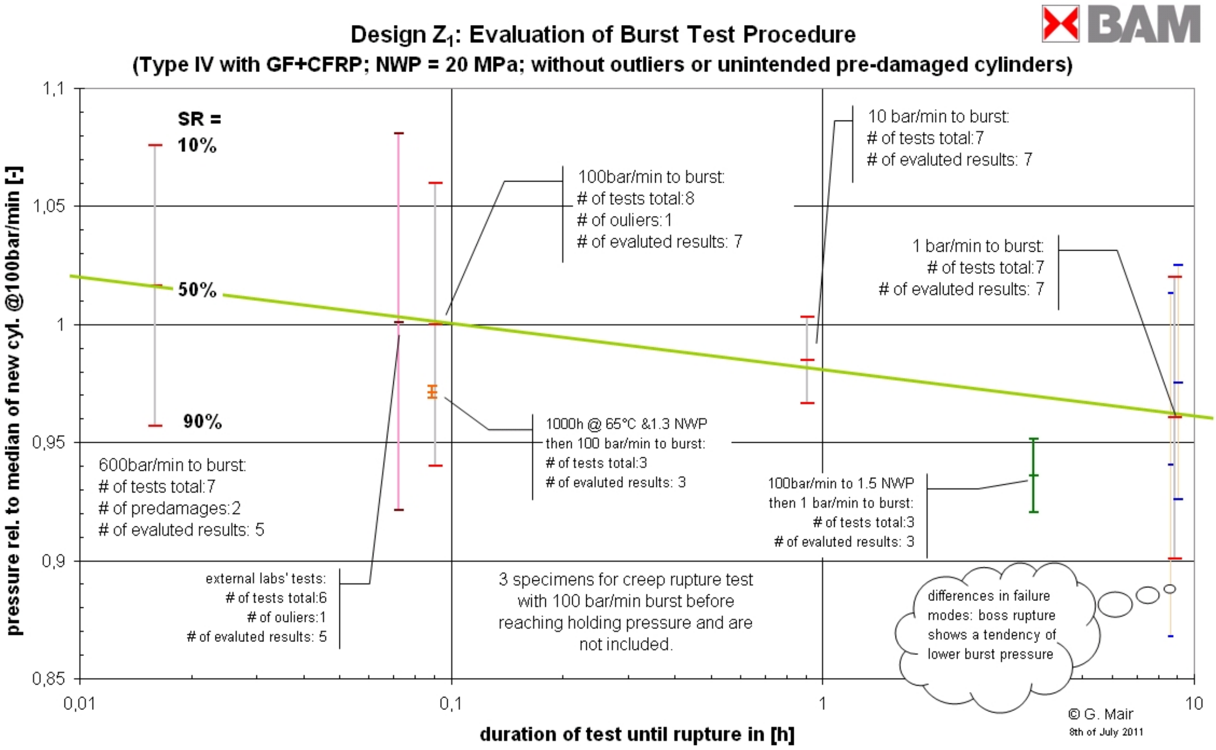

Figure 5: Influence of constant pressure increase rate on burst pressure for one specific design Z_1

As shown in **figure 5** concerning burst tests, the pressure increase rate shows significant influence on the test result. Thus the pressure increase rate should not be specified inside a wide range between minimum and maximum values. For the indispensable comparison of test results elaborated at different test laboratories, the pressure increase rate has to be specified exactly within a small tolerance. In order to achieve universal test procedures this pressure rate should be expressed in relation to the design pressure. The most common design pressure is the test pressure (PH). The test pressure historically comes from the still mandatory hydraulic tests before putting on the market and is also used for retesting. While the test pressure for TDG and onboard storage PH equals 150% of working pressure (PW; TDG) or nominal working pressure (NWP; vehicles), this ratio is lower for stationary use. The most common pressure level for pressure receptacles is still "PH300 PW200bar". A pressure increase rate of 100bar/min means in this case 33% PH/min. Thus the time to burst in relevant tests can be kept within narrow limits and the real strain rate per time does not vary too much.

But what is a good basis for later comparison with residual strength values? As a basic idea for rupture under static loads, it is generally assumed that the creep of the matrix plays an important role. Thus sustained load tests (creep rupture tests) would represent the real loads ideally. But those tests have significant disadvantages. Firstly the time to failure is not known and may vary in dimensions of less than 1/10 to more than 10 times or 100 times of the originally expected time frame. On the other hand, for testing short time periods the employed pressure level has to be so high (80 – 98% average burst pressure) that a significant part of the sample will fail in the phase of pressure increase. In addition, the effort for protective measures (test chamber) and holding pressure constant is high. Thus BAM has been searching for alternatives.

It has been a self-evident idea to mix aspects of burst tests and of sustained load tests such that the "slow burst test" has been created. For the purpose of high reproducibility the pressure increase rate should be kept uniform, at least within periodic time steps. Secondly the slow burst test to rupture should be repeated so that the decrease of residual life time can be quantified and compared with the data on new cylinders as has been described above for the ambient cycle tests on type III cylinders. The slow burst test attempts to achieve a failure mode which introduces failure aspects of matrix creeping. Thus there might be a limit for the pressurisation rate that may depend on the material and design. First tests – as shown in **figure 6** – show acceptable results at a maximum pressure rate of ⅓% PH per minute which is equal to 20% PH per hour.

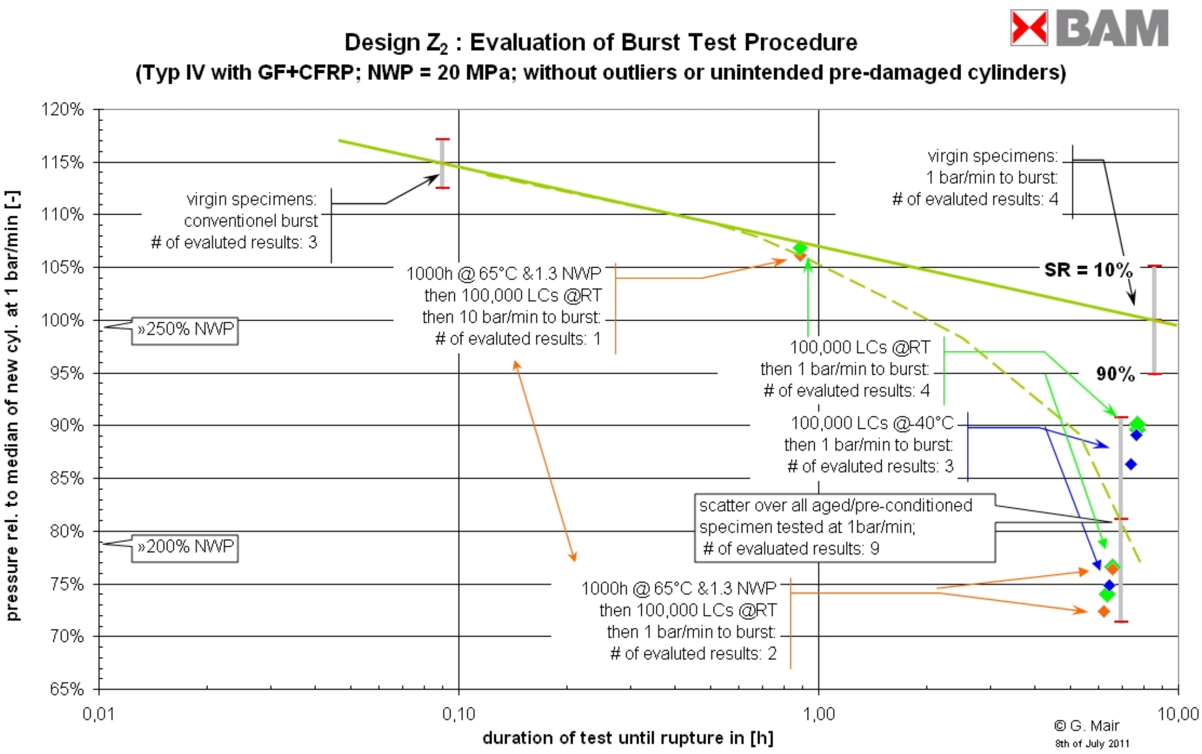

Figure 6: Strength of artificially aged cylinders in comparison with new cylinders of design Z_2

One gets the impression that a rate of 200% PH/h is too fast and 20% PH/h may be sufficient or may still be too fast, but this has not yet been substantiated. Since neither 200% PH/h nor 20% PH/h is currently being assessed in a statistically sufficient manner for different materials and designs, research as to this question at the BAM is ongoing. The aim is to develop a test procedure that allows measuring and comparing the residual strength for all issues that need quantification for containments artificially aged during design type tests or for containments naturally degraded during operation.

8.0 PROPOSAL FOR THE TESTING PARALLEL TO OPERATION AND RELEVANT DESIGN TYPE TESTS

Both main concepts, the approach of testing parallel to operation (TPO) and the probabilistic approval approach (PAA), mean a lot of effort. Onboard storage systems are going to be manufactured in much high numbers, and neither are they subject to manual handling nor are they required to get retested (with the exception of visual inspection as part of the vehicles inspection). In contrast, composite cylinders and tubes for the transport of compressed gases are manufactured in smaller numbers than onboard storage systems and have to be retested. In addition TDG-cylinders are being handled manually. Thus the advantages and disadvantages of the approaches described above are different.

But for both applications a combination of both approaches, PAA and TPO, is expected to yield most efficient results. While manual handling and mandatory retesting limit the advantage of the probabilistic approach, a strictly limited time of operation limits the advantage of destructive tests with regard to real degradation. Nevertheless the combination of both approaches allows a reliable check of the accuracy of the original life time prediction for both applications. Simultaneously the effort for life time prediction as is necessary in line with the probabilistic approval approach can be reduced if the quantification of degradation results in statistically assessable data. It is expected that the focal point for safety surveillance of onboard storage containers should be set on the approval, and destructive tests after years of operation may give additional confidence with regard to life time prediction.
Since the operation parameters for TDG-cylinders can not be predicted as accurately as for onboard storage systems, the design optimisation has to follow design requirements with higher safety margins related to the expected average loads. Therefore the probabilistic approach will be less efficient than for onboard storage. The focal point of safety assessment will shift a little more towards destructive tests on operated cylinders for the quantification of residual safety. These would even cover the uncertainties on operational loads and may substitute hydraulic retesting.

Thus in the following a concept for the quantification of residual strength for transport cylinders will be outlined.

8.1 Concept

Since composite materials show degradation under static loads (static fatigue), there is no composite vessel with an unlimited life. Therefore design standards and standards for retesting should include a certain life time limitation for every composite vessel. The major deficiency with respect to this circumstance lies with the design standards. Thus the approach for UN cylinders to always dictate a certain life time (with the possibility of extension / a second life) is a concept which reflects reality (see [22]).

Most experts have recognised that beside the internal and external visual inspection, hydraulic retesting seems to be an expensive tool with unclear functionality concerning the limitation of life time. Consequently, a tool with respect to fatigue aspects is needed which allows limiting the life time to an appropriate end of service – not too conservative and not too careless. It is thus recommended to use an approach which allows combining retesting with the assessment of residual life time.

Since relevant approaches based on acoustic emission are not sophisticated enough, this leads to a concept of destructive testing of cylinders parallel to operation.

Reflecting the outcome of the discussion on the harmonisation of the 15 years interval for steel cylinders for LPG in Europe, some measures should be taken to reduce the impact of gas filling on aging of containments and to intensify the effort for the inspection before filling (scratch and impact damages).

The first step of the proposal is to limit all composite cylinders to a limited life of not more than 25 - 30 years – as long as the requirements of the design standard for a relevant number of years are fulfilled. Then, after about 10 years, some tests on randomly picked cylinders should be conducted for an ongoing assessment of the end of life. Finally there should be a good data basis for the safe end of life taking into account the operational loads and relevant degradation effects. A prediction based on two statistically sufficient samples (brand new and 10 years old) should be reliable for a confirmation of the end of service life at about 30 years. Nevertheless a test campaign after 20 years for purposes of surveillance could be made mandatory for all cases with very small safety surplus.

No parallel hydraulic pressure test for periodic inspection would be necessary. The periods for internal inspection would have to be correlated with the periods for the exchange or retesting of valves (not more than 15 y). External inspection should be performed at the filling station accompanied by random tests by a third party inspection body (X(a) body – see [4]).

If there is an interest in a period of more than 30 years, further assessment by a further destructive test campaign should be performed in the same way again for another 5 to 30 years' life time, but not more than 60 years in total.

8.2 Test methods

Secondly it is necessary to analyse the test methods. Therefore we have to take into account the basic information on cylinders, which means the results of design tests. Thus we have to differentiate between cylinders with metallic liners and those without load sharing liners – which current standards do.

In addition, we have to ask whether the scatter of fibre material does allow demonstrating the safety level (survival rate of 99.9999% against burst, see [23]) by cycling tests within an acceptable frame of load cycles. This leads to further differentiation based on fibre types. In [18] it is expected that for some composites an average of 250 times the addressed number of fillings (1,000 fillings → 250,000 load cycles) would have to be demonstrated, which is simply an unacceptable effort for design type testing. Thus the differentiation as practiced with respect to burst ratios should be used for other aspects, too.

In conclusion, the relevant set of tests on new cylinders should start with burst tests and with cycle tests to leakage at constant -40°C and +65°C (gases for TDG) or +85°C (hydrogen onboard storage).

If the cycle tests of a sample of three new cylinders show at least two leakages or bursts before e. g. 50,000 LCs, the sample exhibits cycle sensitivity and we should follow a). If not, the sample is called "non-cycle sensitive", see b).

a) If we can be certain that the statistical evaluation of cycle tests is apt to demonstrate a sufficient safety level (see [3]), the cylinders should undergo ambient cycles tests to leakage after preconditioning by static loads as e. g. PH@65°C for 100 h per design life time (type III and eventually type IV with GFRP).
These tests should be performed on new cylinders plus operated cylinders with 1/3 (and perhaps 2/3) of the specified time of operation. Relevant results should be compared such that the decrease of residual life time can be quantified and compared with the original expectation. To this end the size of samples should guarantee an acceptable statistical basis.

b) If the modified extreme temperature cycle tests on a sample of three new cylinders show no leakage or burst after 50,000 LCs (expected for type IV made of CFRP), the slow burst test as described above should be used as alternative test protocol.

The alternative use of both tests would guarantee a reliable surveillance and prediction of safe operational life time for existing cylinder populations from the start. Thus the deterioration caused by hydraulic tests would not be necessary any longer and the effort needed for internal inspection and removing valves every 5 years could be used to gain a higher level of knowledge on cylinders in use. Since this concept would require changes in regulations, in the mean time we could consider the use of this concept for the determination of a design type specific retesting period up to 15 years by the competent authorities.

9.0 SUMMARY

There are some historical reasons for differences in design type test procedures for vessels for the transport of gases and for onboard storage of gases as fuel. The critical points in both fields are similar. The figures gained from current test results can neither be assessed statistically nor used for risk control. But the quantification of risk is necessary for an objective safety assessment.

The need for quantification of strength properties shows the problems of current test procedures. There is no sufficient reproducibility, the minimum requirements are sometimes too low, sometimes too stringent and inflexible; and the obligatory cycles test does not allow obtaining strength data for all design types. Currently a minimum strength is demonstrated based on one to two prototypes per test only.

The proposed way out of this dissatisfying situation is to implement test procedures that address probability assessment of relevant designs at design type testing as well as destructive tests parallel to operation for an optimised survey of safe service life. This requires a test procedure which enables us to quantify strength of new and of used composite pressure containments – even when cycle fatigue tests do not work. For this purpose the test procedure of slow burst tests has been proposed in combination with a pre-test procedure that allows differentiating "cycle fatigue sensitive" designs from those that are not cycle fatigue sensitive.

Based on this it will be possible to restructure the current concept of combining periodic retesting with conservative life time prediction during the design type approval procedure such that hydraulic retesting can be substituted, and life time prediction could be adjusted by probabilistic evaluation of tests parallel to operation.

REFERENCES

[1] Mair, G. W.: Hydrogen Storage Systems for Automotive Application (StorHy): Pre-Regulatory Research Results Related to Public Safety. Presentation at the Session on RC&S of the HFC-Review Days 2007; 11th October 2007 in Brussels

[2] Mair, G. W.: **Hydrogen Onboard Storage – an Insertion of the Probabilistic Approach into Standards & Regulations?** Paper and Presentation at the „International Conference on Hydrogen Safety ICHS".Pisa: TU; Conference Proceedings 2005 on CD

[3] **Druckgasverordnung** (DruckgasV) vom 20. Juni 1968 (BGBl. I S. 730)

[4] **RID/ADR** European Agreements concerning the International Carriage of Dangerous Goods http://www.unece.org/trans/danger/publi/dg_publications.html

[5] **Gefahrgutverordnung Straße, Eisenbahn und Binnenschifffahrt** (GGVSEB), promulgated on 2009-06-17 (BGBl I, p. 1389);

[6] **Council Directive 1999/36/EC of 29 April 1999 on transportable pressure equipment** (Official Journal L 138 , 01/06/1999 p. 0020 – 0056, No. 399L0036) http://www.netinform.net/Vorschriften/DG/tped_en/directive_1999_36_ec.htm

[7] **International Maritime Dangerous Goods Code (IMDG-Code)** Verkehrsblatt 2009, S. 102ff., Dokument Nr. 26 http://www.bmvbs.de/-,1827.933629/Gefahrgut-Recht-Vorschriften-S.htm

[8] **Gefahrgutverordnung See (GGVSee)**, as amended and promulgated on 2007-12-03 (BGBl I, p. 2815); in particular chapter 6.2 and 4.1.6 IMDG-Code – Amdt. 34-08

[9] **IATA-Gefahrgutvorschriften (IATA-DGR)** 51. Ausgabe; valid for the year 2010; http://www.iataonline.com

[10] **Recommendations on the Transport of the Dangerous Goods - Model Regulations** Geneva: United Nations 2007: 15th rev. edition; http://www.unece.org/trans/danger/publi/unrec/rev15/15files_e.html

[11] **DIRECTIVE 2010/31/EU OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL** of 19 May 2010 http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2010:153:0013:0035:EN:PDF

[12] Moretto, P.; Mair, G.: **Overview of Requirements for Destructive Hydrogen Container Tests. Workshop on International RCS: Gaps and Adaptations**; 3rd June 2008 during the 5th Annual StorHy-Meeting 2008 in Paris

[13] Mair, G. W.: **Generals on a Probabilistic Approach. Workshop on International RCS: Gaps and Adaptations**. 3rd June 2008 during the 5th Annual StorHy-Meeting 2008 in Paris

[14] Mair, G. W.: **Fatigue Testing and its Operational Relevance**. Workshop on International RCS: Gaps and Adaptations. 3rd June 2008 during the 5th Annual StorHy-Meeting 2008 in Paris

[15] Mair, G. W.: **Highlights of SP SAR within StorHy related to RC&S.** Brüssel: CDMA 2005. Internet publication at: http://www.harmonhy.com/harmonhydocs/HarmonHyWP0-012.ppt

[16] Mair, G. W.: **Advantages of Reliability Analyses of Hydrogen Storage Receptacles** Brüssel: EUCAR 2004. Internet publication at: http://www.StorHy.net

[17] Mair, G. W.: **The Low-Cycle Fatigue of Fibre-Metal-Hybrid Structures.** Proceedings of the „Fourth International Conference on Low Cycle Fatigue and Elasto-Plastic Behaviour of Materials (LCF 4)“. Oxford: Elsevier Science 1998.

[18] Mair, G. W.; Schulz, M.: **Fundamental Examination of a New Concept of Safety Surveillance and Interactive Determination of Safe Service Life for Composite Pressure Vessels by Destructive Tests Parallel to Operation.** Paper and Presentation at the 16th INTERNATIONAL CONFERENCE ON COMPOSITE STRUCTURES, University of Porto; 28-30 June 2011.

[19] EN 12245:2009, **Transportable gas cylinders - Fully wrapped composite cylinder.**

[20] Bohse, J., Mair, G. W., Novak, P.: **Acoustic Emission Testing of High-Pressure Composite Cylinders. Conference on** Advanced Materials Research / 27th European Conference on Acoustic Emission Testing (EWGAE 2006), September 20-22, 2006, Cardiff, Wales, UK (Proceedings) 13-14 (2006), 267-272; Trans Tech Publications, Switzerland.

[21] Mair, G. W.; Erhard, A.: **Recognized technical code (Anerkanntes Technisches Regelwerk, ATR) for the construction, equipment, test, approval and marking as transportable pressure equipment of composite tubes with a non-load sharing plastics liner with a working pressure not exceeding 500 bar and a water capacity not exceeding 450 L** (see VkBl. Heft 23-2010; p. 611-622) (ATR D 4/10); BAM 2010: official announcement for the use by German inspection bodies. http://www.bam.de/en/service/amtl_mitteilungen/gefahrgutrecht/druckgefaesse.htm

[22] USA and CAN: UN/SCETDG/35/INF.50; **Life of Gas Cylinders of Composite Construction Bearing the UN Mark** - Comments on ST/SG/AC.10/C.3/2009/7 (ISO) and INF. 7 (EIGA); Information Paper for the Sub-Committee of Experts on the Transport of Dangerous Goods

[23] ISO/TC 58/SC 3/WG 17: **Rationale: High Pressure Cylinders for the On-Board Storage of Natural Gas as a Fuel for Automotive Vehicles**; ISO July 1995.