# THE SLOW BURST TESTING OF COMPOSITE CYLINDERS

# PART I: SLOW BURST TESTING OF SAMPLES AS A METHOD FOR QUANTIFICATION OF CYLINDER DEGRADATION

**Mair, G. W., Hoffmann, M., Schönfelder, T.**
**Department 3.2, BAM Federal Institute of Materials Research and Testing, 12000 Berlin, Germany, georg.mair@BAM.de**

## ABSTRACT

The current practise of focusing the periodic retesting of composite cylinders primarily on the hydraulic pressure test has to be evaluated as critical - with regard to the damage of the specimen as well as in terms of their significance. This is justified by micro damages caused to the specimen by the test itself and by a lack of informative values. Thus BAM Federal Institute of Materials Research and Testing (Germany) uses a new approach of validation of composite for the determination of re-test periods. It enables the description of the state of a population of composite cylinders based on destructive tests parallel to operation.

An essential aspect of this approach is the prediction of residual safe service life. In cases where it cannot be estimated by means of hydraulic load cycle tests, as a replacement the creep or burst test remains. As a combination of these two test procedures BAM suggests the "slow burst test SBT". On this a variety of about 150 burst test results on three design types of cylinders with plastic liners are presented. For this purpose both, the parameters of the test protocol as well as the nature and intensity of the pre-damage artificially aged test samples are analysed statistically. This leads first to an evaluation of the different types of artificial ageing but also to the clear recommendation that conventional burst tests be substituted totally if indented for assessment of composite pressure receptacles.

## 1.0 INTRODUCTION

Composite materials are subject to service-related changes of their properties. Therefore, there is a need to implement residual strength determination procedures in order to survey safety of a population of pressure receptacles or fuel gas storage cylinders made of composite materials. The procedure published in [1] by the Federal Institute for Materials Research and Testing (BAM) as the responsible composite authority in Germany considers the safety of composite pressure vessels throughout the service life by the assessment of survival probability and derives appropriate inspection intervals are derived. This is based on an idea, already published during the 1st ICHS conference in 2005 [2] and ICHS 2011 [3].
Data resulting from such a concept of degradation surveillance are supposed to give very important input to the extensive discussion safety margin as being mandatory in regulations and standards.

As demonstrated in [4], it is essential to distinguish each design type in terms of their material related behaviour according to its cycle-fatigue-sensitivity. This sensitivity is assessed and confirmed as “cyfas” by the failure rate of a sample hydraulically tested to at least 50,000 hydraulic load cycles; which is intended to be the first step of each design type approval process.

The criteria applied in [1] were established in [5] to [8] by experience of the first repetition of load-cycle tests as key test for “cyfas” composite gas cylinder. These test samples have been in service for many years. In order to develop the burst test as an analogous key test for non-cycle-fatigue-sensitive (non-cyfas) composite gas cylinders results from relevant test campaigns and their findings will be presented. The slow burst test (SBT) will be introduced as a variation of the burst test with a constant pressure rate of not more than 20% of the test pressure (PH) per hour. A variation of test parameters in comparison to to conventional burst tests will be demonstrated. It has to be mentioned that in principle the SBT is relevant for cyfas-designs, too. The assessment of the degradation of the composite wrapping on metal liner ensures the residual strength that is necessary for a reliable leak-before-break behaviour.

The concept of a parameter variation at burst tests is based on two dedicated aspects of experience for cylinder designs that cannot be assessed by hydraulic cycle tests. At first the negative experiences of the authors with statistic assessment of creep rupture tests to failure as e. g. described in [9] and at second the micro mechanical model for failure processes under sustained loads as presented by A. Bunsell et al [10]. Creep rupture tests have the intrinsic uncertainties of an open time frame and of the statistical evaluation of the specimens of a sample that fail during the phase of pressure increase. The model based on clusters of broken filaments by Bunsell explains why the duration of a quasi-static load test is important and supports the concept of creep rupture testing in principle. This lets assume good results by combining the long term aspect of a creep rupture test with the advantages of pressure increase at the slow burst test (SBT).

## 2.0 ASPECTS OF THE PROBABILISTIC ASSESSMENT OF DEGRADATION BEHAVIOUR

Degradation is a term that derives from the Latin (origin of meaning "reduction in rank"), which in the context of fibre composites describes the change in material properties over time. More generally it is also used for the ageing behaviour of components. In the following, degradation is an empirically ascertainable ageing behaviour in the sense of the loss of required and measurable safety characteristics. This means that the decrease in the probability of survival, i.e. reliability against failure during use as described in [1], is also an aspect of degradation.
However, the criterion of load-cycle-sensitivity as shown in [8] implies that material specific degradation has to be considered as depending on the configuration of the gas cylinders respectively of the used materials. This becomes more apparent the more the designer understands the material and is able to use the maximum properties of his design.

If the degradation of residual strength of a cylinder cannot be described directly in a technical appropriate frame, using load cycle strength as primary criterion for fatigue, there is a procedural disadvantage. Especially since this is the desired property of material because of its low degradation under cyclic loading. The cause of non-load-cycle-sensitivity in service between working pressure at 15°C (WP) and test pressure (PH = 150% WP) can also be result of too low stresses due to high wall thickness. But this seems not to be the case for weight minimised composite gas cylinders, because this is the dominant reason to use these expensive containments.
The high fatigue strength of carbon fibres hints at another property that is unknown for metallic gas cylinders: the limited creep rupture strength, scientifically not accurate described as “static fatigue strength”. The knowledge of this effect justifies a test method that can detect the state of degradation by applying a quasi-static load: The burst test. Therefore it has to be clarified what influence the parameters of the burst test, in particular the pressure rate, have on the results and what aspects of the sample can characterise statistically the state of the degradation respectively the residual strength. This is followed by the question: Is it allowed to get conclusions about the probabilistic residual service life out of this description of degradation behaviour?

Figure 1 is based on more than 60 tests and shows the burst strength of two similar design types Y (blue) and Z (green), both with plastic liner and with a significant amount of glass fibres in the load bearing fibre volume.

A lower pressure rate reduces the burst strength of both design types. However, due to a significant ratio of glass fibres in the composite, these design types have a borderline load-cycle-sensitivity in accordance with the criteria as shown in [1]. Therefore, the degradation behaviour as explained in [2] should better be assessed with an extended load-cycle test. Furthermore Figure 1 shows that the samples with pre-condition (red), pressurised with 1 MPa/min, show no visible differences to the new samples (blue). With a pressure rate of 0.1 MPa/min there is a significant decrease of strength regardless the pre-condition compared with virgin test samples (0.1 MPa/min = 20% PH/h). This way of determining the pressure rate as constant in relation to the test pressure is based on the intention to keep the resulting strain rate in the fibre material as specific as possible. These test results have already been assessed by numerical simulation based on a micro-mechanic model [11].

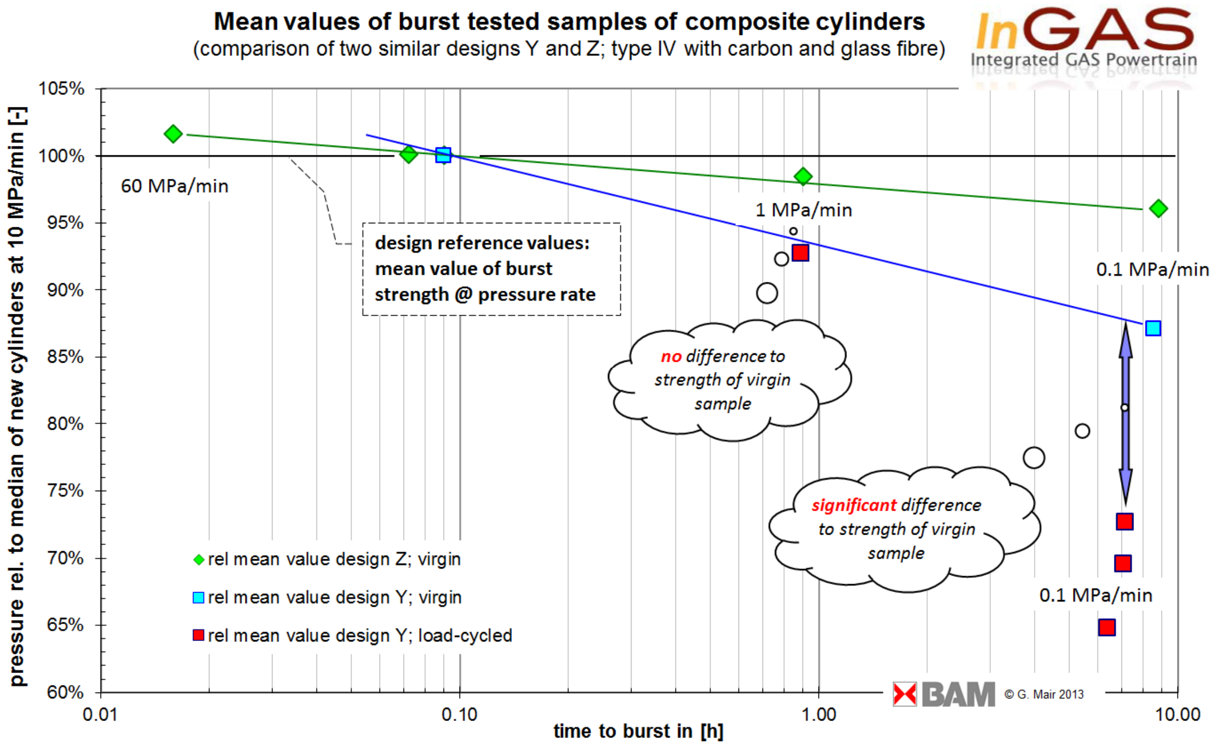


*Figure 1: Influence of pressure rate on burst pressure: GFRP/CFRP-design types with plastic liner*

## 3.0 SYSTEMATIC TEST PARAMETER VARIATION WITHIN HYCUBE

Some effects in [12] were unexpected first and it was decided to test another 75 specimens of one batch of this design D within the European project “HyCube” [13]. The test concept with a systematic variation of pre-conditioning and burst parameters for these 75 burst tests is shown in Table 1. The total effort for pre-condition of all samples shown in Table 1 and explained in the following was in summary more than 3 Mio hydraulic load cycles and about 20,000 h of sustained pressure loads at 65°C.

Pressure-time-curves of burst-tests as varied in Table 1 and coded by numbers 1….5* are shown in Figure 2. There and in the following the relation of burst pressure to test-pressure is represented by the symbol Ω. The only of the double stepped time curves marked by blue in Figure 2 that have been operated and are therefore listed in Table 1 are “4*” and “5*”.
The results of the 75 burst tests with the parameters of Table 1 are shown in Figure 3. Available data on conventional production batch tests with a mean value of 2.7-times of test-pressure (Ω = 2.66) cannot be illustrated due to their high pressure rates.
The virgin samples of the CFRP-cylinders in Figure 3 show no clear influence of the pressure rate on mean value Ω (blue diamonds: A1 – A4*). The samples of GFRP-cylinders - as shown in Figure 1 - display in contrast to this a clear influence of pressure rate on the virgin specimens.

Table 1: Test program for 2nd test campaign on cylinders made of CFRP and plastic liner

| PH = 45 MPa<br>pre-conditioning<br>burst procedure of finale testing | A: no pre-conditioning/ "virgin" | B: 100 000 LC; 2 MPa …PH 10 LC/min; RT | C: 100.000 LC; 2 MPa …PH; 10 LC/min; 65°C | D: 50.000 LC; 2 MPa …PH; 5 LC/min; 65°C | E: sustained load 2000 h; PH @ 65°C | F: 935 h @ PH @ 65°C → 50.000 LC; RT; 2 … PH; 5 LC/min | G: 50.000 LC; RT; 2…PH; 5 LC/min; → 935 h @ PH @ 65°C |
|---|---|---|---|---|---|---|---|
| **1:** 10 MPa/min<br>**11 min** | **A1** | **B1** | | **D1** | | | |
| **2:** 1.5 MPa/min<br>**1.2 h =** 72 min | | | | **D2** | | | |
| **3:** 0.15 MPa/min<br>**12 h** | **A3:** | **B3** | **C3** | **D3** | **E3** | **F3** | **G3** |
| **4*:** 10 MPa/min →<br>→ 0.015 MPa/min<br>**64 h =** 2.7 d | **A4*** | | | | | | |
| **4:** 0.015 MPa/min<br>**128 h =** 5.3 d | **A4** | | | **D4** | | | |
| **5*:** 10 MPa/min →<br>→ 0.0015 MPa/min<br>**780 h =** 32 d | **A5*** | | | | | | |

75 specimens tested: 72 burst tests after pre-conditioning by 3 Mio LC and 19,350 h sustained load

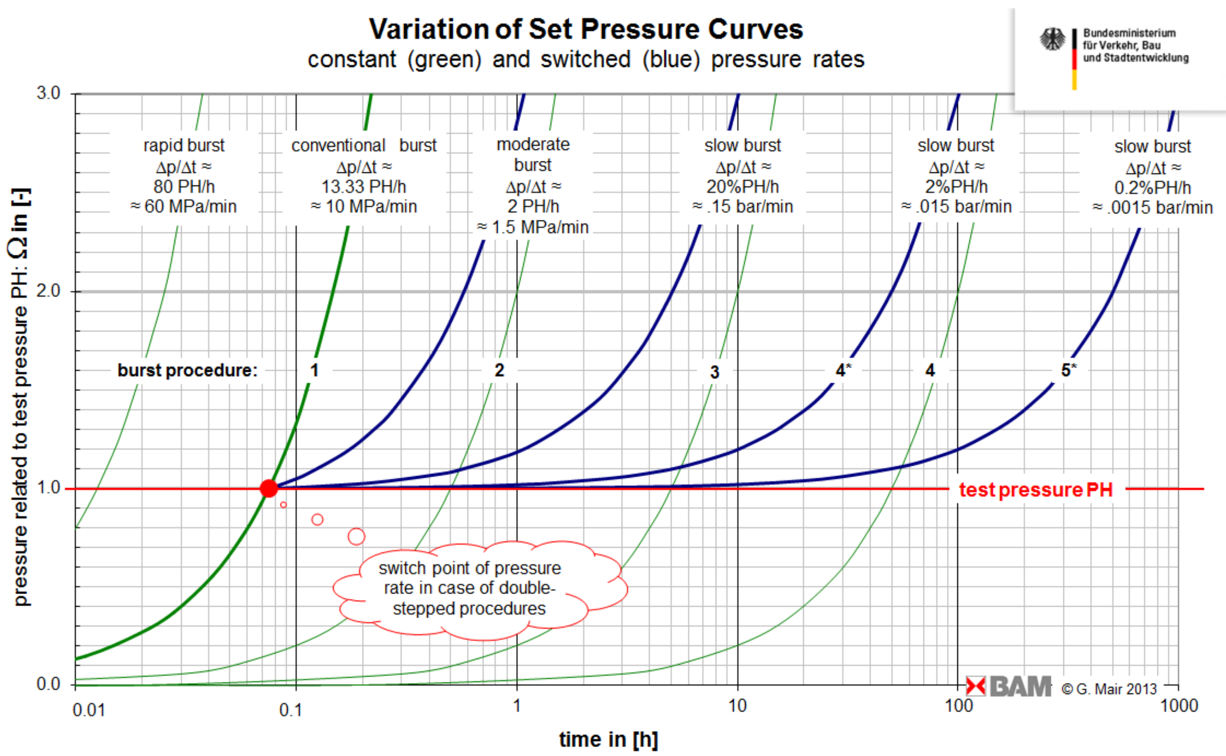


*Figure 2: Representation of different pressure-time curves as a test parameter*

The mean values of the sustained pre-loaded sample (E3) show no significant difference to the unloaded samples. The samples previously damaged by hydraulic cycling show, however, at all pressure rates a significant loss of strength. Here it seems that the influence of ambient temperature and of pressure fluid temperature on the relative mean value Ω is surprisingly low. The mean strength of the test sample (D3) that has been exposed to 65°C for 50,000 LC with 5 LC/min before burst with 20 % PH/h is higher than the strength of the sample pre-conditioned with twice the number of LC (C3: 100,000 LC) with 10 LC/min.

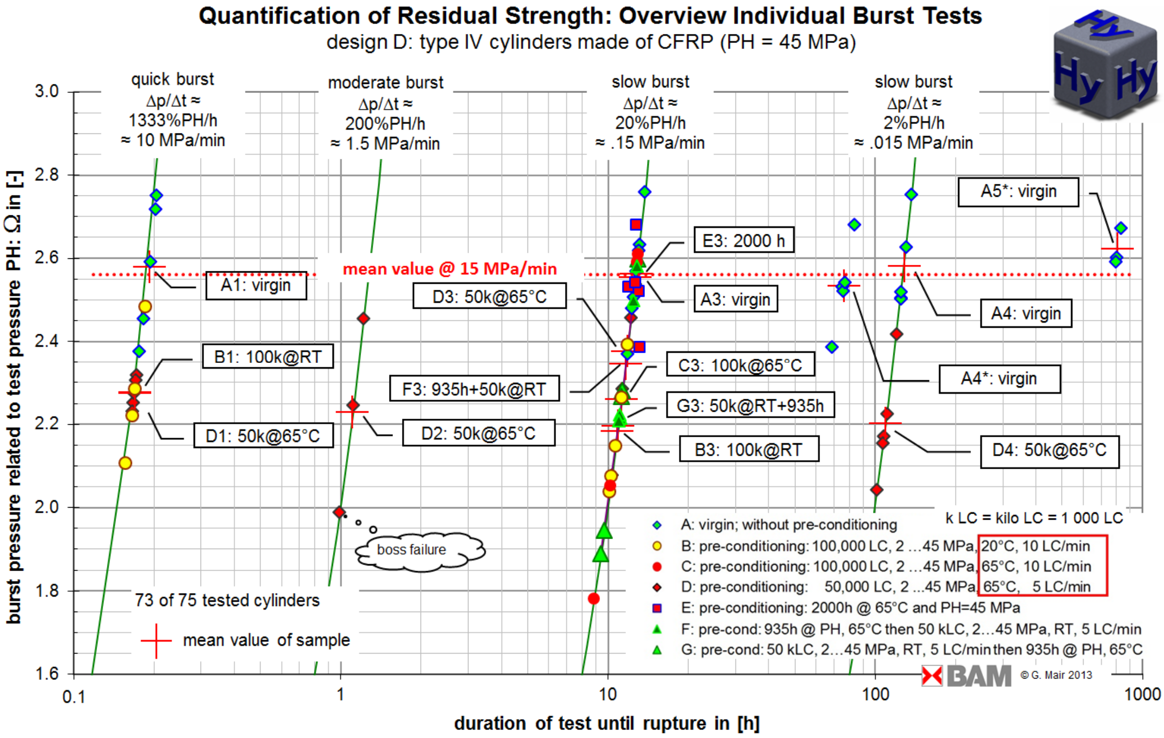


*Figure 3: Results with systematic parameter variation based on Fig 2*

It is however unexpected, that the test specimens with the same hydraulic load at room temperature (B3) do not show a better performance than (C3). Surprising are the results at conventional burst rate with 10 MPa/min. The pre-conditioned samples bursted conventionally after 100,000 LC (B1) and the slower cycled ones with 50,000 at 65°C (D1) in average show the same residual strengths.

## 4.0 CONSIDERATION OF RELIABILITY AND SAMPLE SCATTER VALUES

### 4.1 Ranking of pre-conditioning procedures

As stated in [2, 3, 4 and 13], the reliability R is – cognisant or unknowingly – the essential criteria of all safety evaluations. Accordingly Figure 4 shows the burst test results of Figure 3 related to samples by mean value (y-axis) over scatter of samples (x-axis). The parameter of the scatter is the spread $\psi$. It is defined as the difference between burst properties with a survival rate (SR) from 10% and 90% and is proportional to the standard deviation.
The work chart from [1] is in Figure 4 used into evaluating the results from Figure 3. For a better comparability the mean value of each sample in Figure 3 (red crosses) are shown in Figure 4 with the symbols of pre-conditioning as introduced in Figure 3. The information about the burst parameter in Figure 3 (x-axis) is indicated by the surrounded colour of the symbol.
In Figure 4 the areas of test results of the samples not pre-conditioned by cycling (top) and the hydraulically cycled samples (below) are clearly distinguishable. The area with non-cycled samples appears to be small, while the various pre-conditioned samples show significant differences. Due to

absence of test data of relevant production batches further 28 available test results from [10] can unfortunately not taken in consideration in the following.

The aspect of reliability $R$ will be discussed in particular in the following. Therefore the difference of test pressure (PH) and mean values of burst pressure $m_p$ is divided by the corresponding standard deviation of the burst pressure $s_p$ of a sample. The survival rate $SR$ of a sample is given applies to:

$$R \sim SR \sim x_{ND} = \frac{m_p - PH}{s_p} \tag{1}$$

Equ (1) corresponds to the definition of the normalised deviation rate $x_{ND}$ in the GAUSSian standard distribution. Due to the limited intention of comparing values without discussing absolute reliability values it is not necessary to assess the distribution density function of test results.

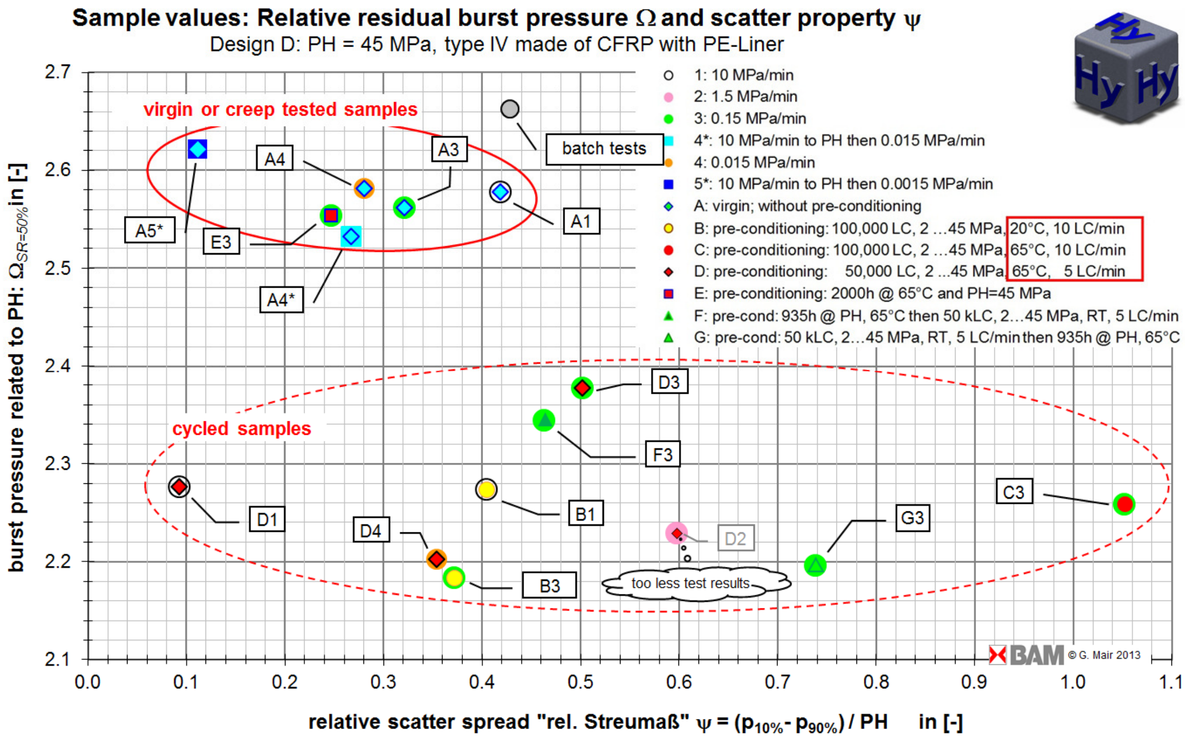


*Figure 4: Sample assessment: Mean burst values over spread*

Based on this Figure 5 graphically shows a ranking of the impact of pre-conditioning procedures on the degradation of samples. Therefore the test results of all samples tested with burst procedure 3 (0.15 MPa/min; dark line) are ranked according to their quantified standard deviation according to Equ (1) as a qualitative placeholder for reliability.

This deviation is shown as a margin between sample values and a reference value of the virgin sample tested at a pressure rate of 20% PH/h with $x_{ND}$ = *12.5*. Following effects are noticeable:

a) Pure load cycle pre-conditioning with 100,000 LC at RT (B3) lead to a reliability loss.

b) The reduction of burst strength of sample (C3), which was pre-conditioned with 100,000 LCs at 65°C, is higher than sample (D3) which hat 50,000 LC at 65°C with the double duration per cycle.

c) Residual reliability decreases with the load cycle temperature from A3 → B3 → D3 → C3.

d) The specimens of the sample (E3), which were loaded with sustained test pressure (PH) for 2000 hours at 65°C, show a significant higher reliability than all groups of virgin samples.

e) The sample (F3) with pre-conditioning for 935 h under PH at 65°C before load cycle test at RT shows a higher residual reliability as the sample (G3) with reverse order of pre-conditioning phases.

f) Sample (F3) shows in relation to (D3) that the improvement due to sustained load as seen in load case (E3) is not significant any more. Nevertheless load cycles after a pre-conditioning by sustained load (F3) seem to be less critical than in the other sequence (G3).

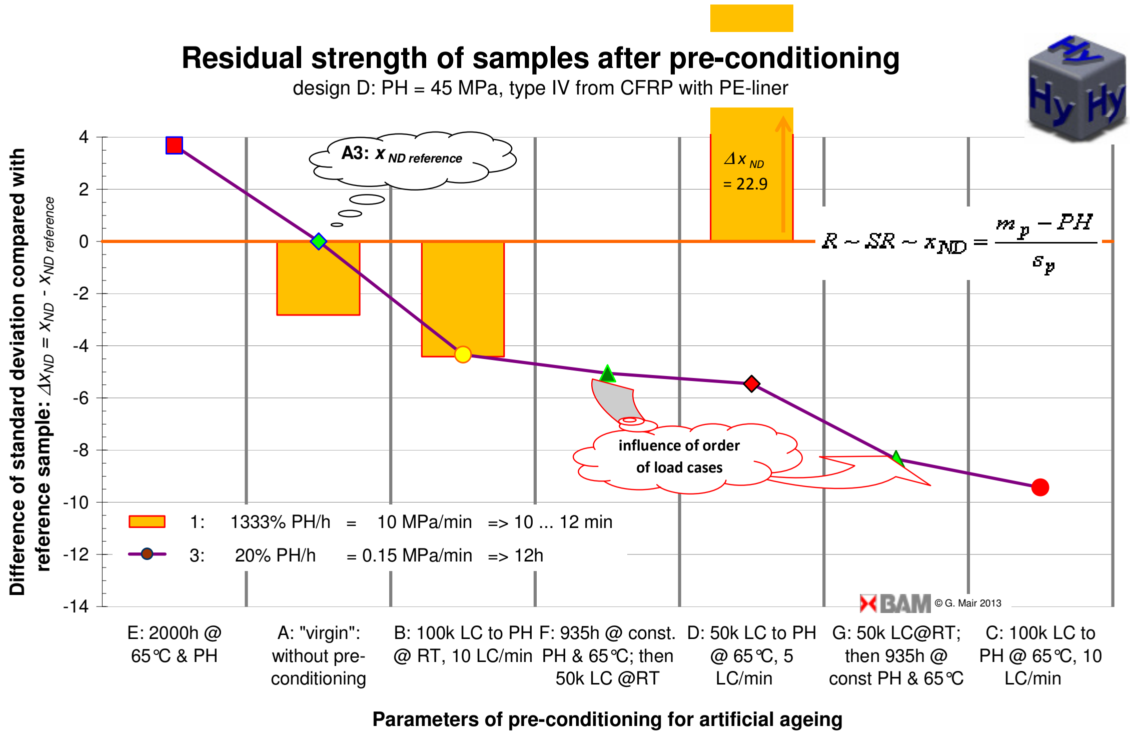


*Figure 5: Ranking of reliability according to pre - conditioning*

### 4.2 Assessment on influence of pressure rate

The intention of the ranking in Figure 6 is to identify which pressure rate of burst test or SBT is the best to determine the level of degradation or damage.
For this purpose in Figure 6 mean values of different burst procedures of non pre-conditioned samples (A) are compared with those of load cases (B) and (D). The comparison is presented in columns of discrete pressure rates. To consider the deviation in a better way the test results of all samples are related to the reference result of a virgin sample at pressure rate of 20% PH/h.

At all slow pressure rates the samples without pre-conditioning (A1, A2, A4, A4*: blue; “virgin”) show a marginal deviation compared to the reference sample (A3). The slight decrease of burst pressure is an expected but not significantly confirmed influence of double-stepped SBT (4*). One explanation for this may be found on a micromechanical scale: in setting processes by micro cracks and matrix creep which cause a positive influence by rerouting stresses due to slow passage of the uncritical load region. This means positive “relaxation effects” between filaments and matrix and an increasing rate of filament breaks according to the pressure take place during the burst tests. But these positive effects contribute little at the fast pressurisation to test pressure at the test protocol with double-stepped pressure rate.

As shown in Figure 6 by using SBT (procedure 3) the sample pre-conditioned at 65°C (red line) achieves a higher mean burst value than the sample conditioned at room temperature (yellow lines). This observation speaks against an exclusive consideration of the mean value as a measure for the degradation (red arrow). At the same time a high pressure rate lets the mean value of the red line decrease to the yellow line. This behavior contradicts the results of the yellow line (green arrows) and is no argument for the use of high pressure rates.

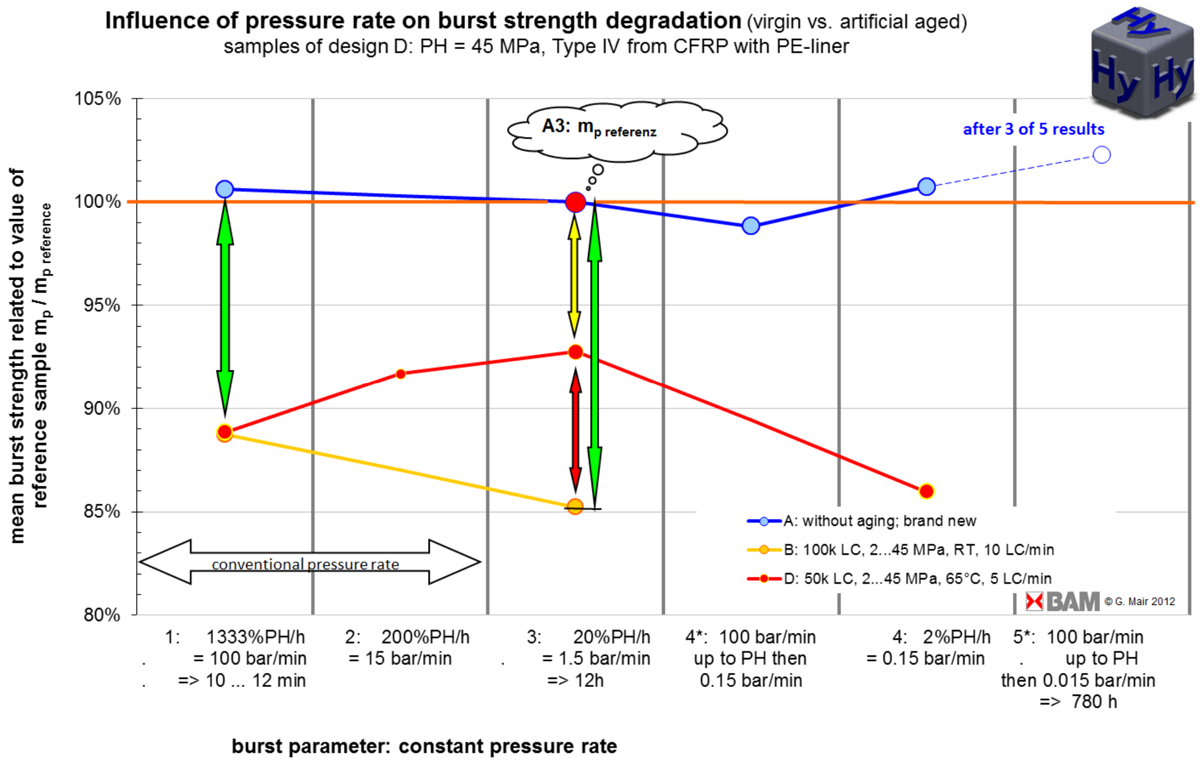


*Figure 6: Mean values of burst pressures over pressure rate*

Addition to this Figure 7 shows the same context for the standard deviation of samples. Again the values of the samples are compared to the reference sample. At a conventional burst pressure rate (procedure 1) the standard deviation of the samples pre-conditioned by cycling at RT remains the same as the one of the virgin samples. In contrast to this the standard deviation of the conventionally bursted sample after pre-conditioning at 65°C (red line) is unexpectedly much lower (red arrow).
Using SBT (procedure 3) the scatter value of the sample pre-conditioned the 65°C is significantly higher than the ones of both other samples (green arrow). The virgin samples show a tendency of change of the distribution that is similar to the tendency of the mean values: smaller mean value equals small scatter. None of these descriptions show a consistent appearance. Therefore a further evaluation according to Equ (1) is performed.

Figure 8 presents the influence of pressure rate on the burst pressure results by applying Equ (1). Here the differences compared to the reference value $x_{ND} = 12.5$, virgin sample, pressure rate of 20% PH/h, are shown:

i) The virgin samples (blue lines) show increasing test time results in an upward trend of reliability. It seems that the time saving double-stepped burst procedure has no impact on the test results.

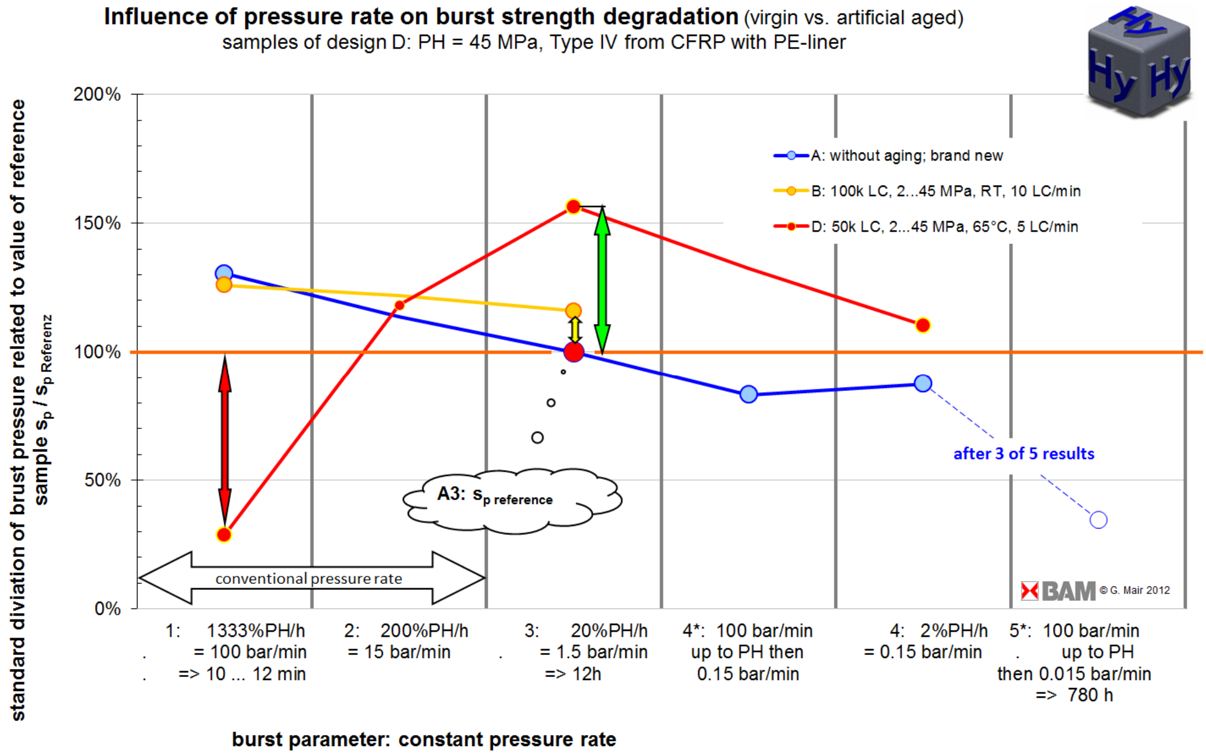


*Figure 7: Standard deviation over pressure rate*

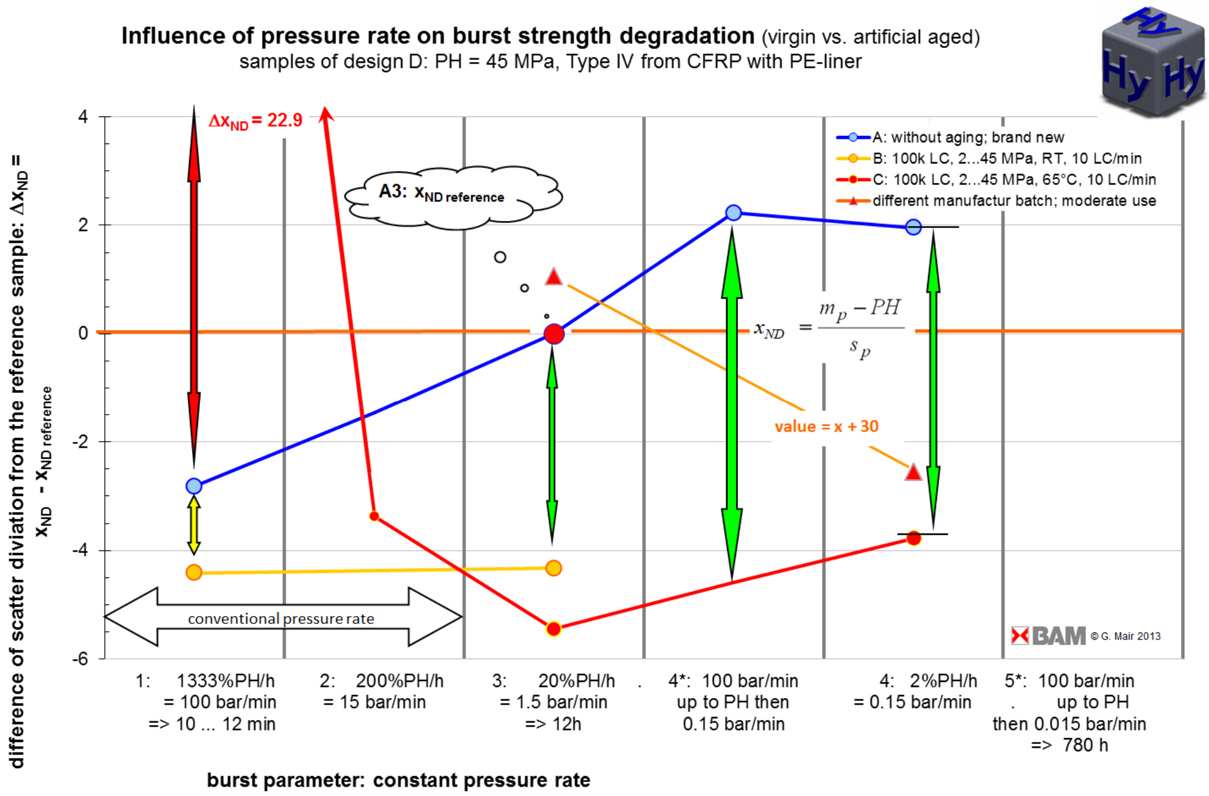


*Figure 8: Normalised deviation rate as indicator of reliability over pressure rate*

ii) For the load cycle-test at room temperature (yellow line) no influence of the pressure rate on reliability is evident.

iii) The small samples - degraded during operation (orange line) or cycled at 65°C (red line) - show a decrease of their reliability corresponding to the test duration. This corresponds with the expected tendency of increased detectable degradation with increased test duration.

iv) In tests with the currently common test duration of less than 10 minutes (left column) it seems that the reliability of the sample C1 (pre-conditioned by LC at 65°C) is enormously higher than all virgin samples despite of high pre-damage. On this scale this cannot be explained and must therefore be regarded as an artifact of the combination of pre-conditioning and quick burst testing, which argues against conventional burst procedures.
One explanation for this artifact could be that the damage processes initiated during pre-conditioning cannot increase further during burst test due to the fast load rate. It appears that at high burst rates different processes are initiated where the aging at 65°C might have a positive effect.

It is assumed that differences in burst pressure between virgin and pre-conditioned samples are an indicator of the degree of degradation. As part of the projects [12 and 13] among others the pressure rate was looked for at which this difference is most evident. With reference to the arrows in Figure 8 this is not the case at 10 MPa/min (yellow arrow). This tests parameter even can be dangerously deceptive (red arrow). The green arrows however show that a pressure rate of 20% PH/h (here 0.15 MPa/min) or even slower allows an optimal gain in knowledge and offer a maximised differentiation. With regard to the practice this is adequate because filled gas cylinders have to keep their service pressure for hours or months until they are unloaded at the customer again. It is be permissible to speculate about possible reasons for these partially surprising phenomena without verifying any theses. Therefore (I) positive effects of temperature, (II) characteristics of damage evolution in fast burst tests and (III) aspects of matrix hardening as well as (IV) relaxation processes of individual filaments as a homogenisation etc. have to be considered when analysing these phenomena. Also the consideration of competing damage processes probably leading to significant increase of scatter value has to be involved.

## 5.0 SUMMARY ON THE PERFORMANCE OF SLOW BURST TESTING

Some unexpected results from burst tests of "virgin" and pre-conditioned samples of composite gas cylinders have been shown and explained. Basically it must be checked which phenomena are expected in general or valid for a type of construction (e.g. CNG-3) or are only specific to an individual design type.

The results already show that a quantitative determination of initial- and residual strength of non-cycle sensitive receptacles, especially with a plastic liner, has to be performed instead of load cycle tests until failure. In contrast to today's rapid test procedure the slow burst test (SBT) with duration of at least 10 hours (pressure rate not higher than 20% of PH per hour) seems to be the most useful method for this quantitative strength measuring because of:

1. The results of slow burst tests (SBT) performed with a pressure rate of constant 20% PH/h or less show a significantly more coherent performance compared to the results of conventional burst tests. Moreover SBT with increased test time (which means reduced pressure rate) come closer to the real behaviour of cylinders under operational load. It seems that the SBT is the better choice for the purpose of reproducible and consistent performance.

2. Test results fives the impression that SBT allow a more robust evaluation of the real state of degradation, then initial long duration sustained loads on specimens of this design type have a positive effect while fast load-cycles especially at high temperatures have a negative effect on the residual strength.

3. The differentiation of usual damage from critical damage status of a non-cycle fatigue sensitive specimens is hardly to assess. Some damage processes seem to be noticeable by a decreasing mean value or by a rising scatter value. A combination of both shall be used to describe reliability. So it seems that the measure of degradation is not suitable by sole consideration of exclusive mean burst pressure or exclusive scatter values.

It has been shown that burst tests may lead to false safety assessment results if the pressure increase rate is too high. Therefore burst parameters in relevant standards should be changed in general to SBT that is defined here as hydraulic burst test with exactly controlled pressure rate and test duration of not shorter than 10 to 20 hours per test. This is most important in case of cylinder designs with an expected first failure of the composite based on the growing and accumulation of clusters of broken filaments. For a reliable safety assessment based on SBT it is recommended to study the mean values and scatter values of samples in combination as reliability. The validation of only one of these aspects is expected to lead to false conclusions often.

Thus for each design type that has been individually assessed as non-cycle-fatigue-sensitive (non-cyfas) there are strong hints that SBT is the best choice as a test procedure that enables an interpretation, which is qualitatively comparable to hydraulic cycling of cycle-fatigue-sensitive designs. This means that the SBT is recommend herewith for non-cyfas design types as basic test for the determination of residual strength of samples. This recommendation is valid for samples artificially aged by pre-conditioning during design type approval process or naturally aged during years of service. An assessment of degradation is only manageable as a comparison of different SBT results of a dedicated design type – without design variations. At its best a SBT result of a virgin sample of the same production batch is available.

## REFERENCES

[1] Federal Institute of Materials Research and Testing BAM: official publications (online): http://www.bam.de/en/service/amtl_mitteilungen/gefahrgutrecht/druckgefaesse.htm

[2] Mair, G. W.: Hydrogen Onboard Storage – an Insertion of the Probabilistic Approach into Standards & Regulations? Presentation at the „International Conference on Hydrogen Safety ICHS“, Pisa: TU; Conference Proceedings 2005 on CD; ID 22001

[3] Mair, G. W. and Pöschko, P.: Regulations and research on RC&S for hydrogen storage relevant to transport and vehicle issues with special focus on composite containments; presentation at the „International Conference on Hydrogen Safety ICHS 2011“; San Francisco, USA: Conference Proceedings 2011 on CD; ID 244

[3] Mair, G. W.; Pöschko, P.; Schoppa, A.: Verfahrensalternative zur wiederkehrenden Prüfung von Composite-Druckgefäßen; Technische Sicherheit (TS) Vol.1 (2011), Nr. 7/8 (Juli/August), p. 38 - 43, ISSN 1434-9728

[4] Mair, G. W.; Duffner, E.; Schoppa, A.; Szczepaniak, M.: Aspekte der Restfestigkeitsermittlung von Composite-Druckgefäßen mittels hydraulischer Prüfung; Technische Sicherheit (TS) Vol. 1 (2011), No. 9 (September), p. 50 - 55. ISSN 1434-9728

[5] Mair, G. W.; Pöschko, P.; Hoffmann, M.; Saul, H; Spode; M.: Betrachtung von Grenzwerten der Restfestigkeit von Composite-Druckgefäßen, Teil 1: Kriterien der hydraulischen Lastwechselprüfung; Technische Sicherheit (TS) Vol. 2 (2012), No. 7/8 (Juli/August), p. 30 - 38. ISSN 1434-9728

[6] Mair, G. W.; Hoffmann, M.; Saul, H; Spode; M.: Betrachtung von Grenzwerten der Restfestigkeit von Composite-Druckgefäßen, Teil 2: Extrapolation der Lastwechsel-Degradation; Technische Sicherheit (TS) Vol. 2 (2012), No. 10 (Oktober), p. 38 - 43. ISSN 1434-9728

[7] Mair, G. W.; Hoffmann, M.: Assessment of the residual strength thresholds of composite pressure receptacles - Criteria for hydraulic load cycle testing; MP Materials Testing, Volume 55 (2013) 2, p. 121 – 129; ISSN 0025-5300

[8] Reeder, J. R: Composite Stress Rupture: A New Reliability Model Based on Strength Decay NASA/TM–2012-217566 Langley Research Center, Hampton, Virginia

[9] Scott, A.E.; Sinclair, I.; Spearing; S.M.; Thionet, A.; Bunsell, A.: Damage accumulation in a carbon/epoxy composite: Comparison between a multiscale model and computed tomography experimental results; Composites Part A 43 (2012) 1514-1522

[10] Research project: Ermittlung des Langzeitverhaltens und der Versagensgrenzen von Drückgefäßen aus Verbundwerkstoffen für die Beförderung gefährlicher Güter; Bundesministerium für Verkehr, Bau und Stadtentwicklung (BVMBS) AZ UI 33/361.40/2-26; supported by Industriegasverband e. V. (IGV).

[11] Chou, H. Y.; Bunsell, A. R.; Mair, G.; Thionet, A.: Effect of the loading rate on ultimate strength of composites. Application: Pressure vessel slow burst test. Composite Structures 104 (2013) 144–153; http://dx.doi.org/10.1016/j.compstruct.2013.04.003

[12] Research project: HyCube – HYdrid HYdride HYdrogen pressure storage, EU: KIC InnoEnergy Innovation Project KIC-CCAV – 02_2011_LH02_Hy3…

[13] Mair, G. W.: Die betriebsbegleitenden Prüfung als Methode der Sicherheitsüberwachung und interaktiven Lebensdauerfestlegung an Composite-Druckbehältern: Teile 1, 2 und 3; Technische Überwachung (TÜ) Vol. 50 (2009), No. 7/8 (July/ August), p. 46 – 49; No. 9 (Sept.), p. 41 – 45; No. 10 (Oct.), p. 46 - 49. ISSN 1434-9728

# THE SLOW BURST TESTING OF COMPOSITE CYLINDERS

# PART II: STATISTIC EVALUATION OF SAMPLE TEST RESULTS

**Mair, G. W., M., Scherer, F.**
**Department 3.2, BAM Federal Institute of Materials Research and Testing, 12000 Berlin, Germany, georg.mair@BAM.de**

## ABSTRACT

It is common practice to certify pressure receptacles or hydrogen storage cylinders based on the test results of a small number of specimens. In the following a statistic assessment is described for analysing results of sample testing by (slow) burst test of composite pressure receptacles. The results of each sample are represented by just two parameters as a pair of values (means (x; y)). The intention is to make this description as physically understandable as possible for various types of composite pressure receptacles. Therefore one chapter is based on the normal distribution ND for burst issues and applied to non-fatigue sensitive design types. In any case the average burst strength and scatter are determined, analysed and drawn as a pair of parameters. Combined with the introduced diagrams a first rough estimate of survival rate can be created for every sample.

## 1.0 INTRODUCTION

Composite Materials show an extraordinary change of properties depending on service life. This creates the necessity to find tailored methods to determine strength and residual strength of pressure receptacles. Having appropriate knowledge would allow validating and possibly reducing current safety margins for design type approvals. The BAM Federal Institute of Materials Research and Testing as competent authority published a procedure in German and English [1] in 2012 which investigates the failure and survival rate of composite pressure receptacles as scale of safety during total lifetime. These results have to be assessed with respect to expected safe life time and the determination of retest periods.

As demonstrated in [2] to [5] design types should be treated depending on their fatigue behaviour. This permits most efficient use of testing resources. Fatigue sensitivity is determined by the occurrence of significant damage during cycle testing. If no critical damage emerges during cycle testing at 50 to 100 times of the expected service load cycles the design type is deemed non-fatigue sensitive. In this case the receptacle will be judged by its burst pressure, in all other cases by its cycle strength.

The loss of strength can sometimes be determined by mean values, but more often only by the combination of mean value and scatter as shown in [4], [5] and especially [6]. In the following we show how to derive describing parameters of a sample and analyse them through a graph as described in [1]. Describing parameters have to be as generic as possible to be able to compare samples of one design type with varying service history or samples of various design types with equivalent service history. In this paper methods for the statistical assessment of burst tests are described which satisfy those demands. The results are concentrated in a twin parametric description (x; y) and plotted. Two different types of graphs are used to be more physically understandable: The normal distribution for (slow) burst tests and the logarithmic normal distribution for cycle test results. Cycle test aspects will be explained in [7].

## 2.0 EVALUATION OF QUASI STATIC STRENGTH TESTS

The foundation of a simple strength assessment or of an even more sophisticated strength evaluation is the strength test under quasi static loads. That means a test procedure to be performed within a set time frame, which shows results independent from the loading rate. The acceptable time frame depends on the kind of specimens, their materials and other aspects. For pressure receptacles burst tests (“BT”) are commonly accepted as quasi-static strength tests. The independence of burst results from loading rate is – within the relevant range- well proven for receptacles made from metal.

Fibre-plastic composites however show a significant sensitivity regarding time influence during burst tests. So actually there is no loading rate which has no influence on strength. As found in [6] there is a range for loading rate which has no significant influence on strength during slow burst test (“SBT”; procedure described in [8]) – at least for carbon fibre composites. This loading range appears to be in-between 0.1% and 10% of the test pressure PH per hour depending on material and design type. Due to the non-linear strain-behaviour of gas cylinders those values cannot be directly transferred to material strain. Depending on the design type they can be estimated to 0.05% to 10% per hour of the failure strain of the fibres.

The assessments in the following are based on a highly reproducible pressure rate of 20% of the test pressure per hour according to [8]. This results in 10 to 12 hours of test time. For determining test cycles according to [1] the methods according to [8] are mandatory as well as the assessments introduced in this paper.

The data used in this paper regarding slow burst tests (SBT) were derived from carbon fibre composite cylinders for breathing air during an EU-funded project “HyCube” [9].

Column 1 and 2 of Table 1 show SBT results from a sample of 7 specimen of the design type used in [9]. The pressure increase rate as main parameter was constantly 20% of PH per hour according to [8].

Table 1: Slow burst test results of a CFRP-type IV cylinder for breathing air tested within [9]

**Steps for the statistic assessment of test results**

$$SR = \frac{3 \cdot j - 1}{3 \cdot j_{\max} + 1}$$

MCS; virgin

| chronologic order of testing | | ascending order of | | | | |
|---|---|---|---|---|---|---|
| 1. | 2. | 3. | 4. | 5. | 6. | 7. |
| ID of specimens | Burst Pressure PB (test results) "$p_B$" in [MPa] | ranking place "j" | Burst Pressure PB (test results) "$p_B$" in [MPa] | survival rate SR in [%] | mean-value of burst pressure $m_{pB}$ | standard deviation of burst pressure $s_{pB}$ |
| 11800 | 111,6 | $j_{\max}$ = 7 | 106,6 | 91% | | |
| 11802 | 112,8 | 6 | 111,6 | 77% | | |
| 11806 | 115,6 | 5 | 112,8 | 64% | | |
| 11809 | 118,5 | 4 | 115,6 | 50% | 115,28143 | 5,621840 |
| 12295 | 117,8 | 3 | 117,8 | 36% | | |
| 12273 | 124,1 | 2 | 118,5 | 23% | | |
| 12334 | 106,6 | 1 | 124,1 | 9% | | |

The working steps for the evaluation are the ranking of results (3 and 4), the calculation of individual survival rate (5), the calculation of mean strength value (6) of the sample and the calculation of standard deviation (7) as shown in Table 1. The equations relevant for columns (6 and 7) are:

$$m_{pB} = \frac{1}{n} \sum_{i=1}^{n} p_i \qquad \text{[MPa]} \qquad (1)$$

$$s_{pB} = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} \left(p_i - m_{pB}\right)^2} \qquad \text{[MPa]} \qquad (2)$$

Finally the individual test results can be drawn in combination with the mean value and the standard deviation, as shown in Figure 1. The gradient of the best fit straight line visualizes the scatter of burst pressure of the various specimens. The intersection of the fit line with the survival rate of 50% is the mean value per definition. The difference between mean value and the points referencing to survival rates of 50% ±34% equates to the standard deviation $s_{pB}$ .

As soon as the test results are evaluated in the way shown in Figure 1 the mean value and the standard deviation are known.

The concept of destructive pressure cylinder tests is limited by the usually very small number of specimens. For this reason there is no point in discussing the sufficiency of the ND. A fit with WEIBULL distribution or other more intricate fits need a much better data base (WD needs ≥ 13 … 21 test results). As a first step an increase of the minimum sample size of 5 would primarily improve the fit using ND before an assessment of distribution character becomes relevant.

Since the intention of such an analysis is the assessment of safety the available strength values (load carrying capacity) has to be related to the load values (load to be carried). The easiest and best way of interpretation is the division of burst pressure by occurring pressure. This division of load carrying capacity by the load is well known as load factor or safety factor.

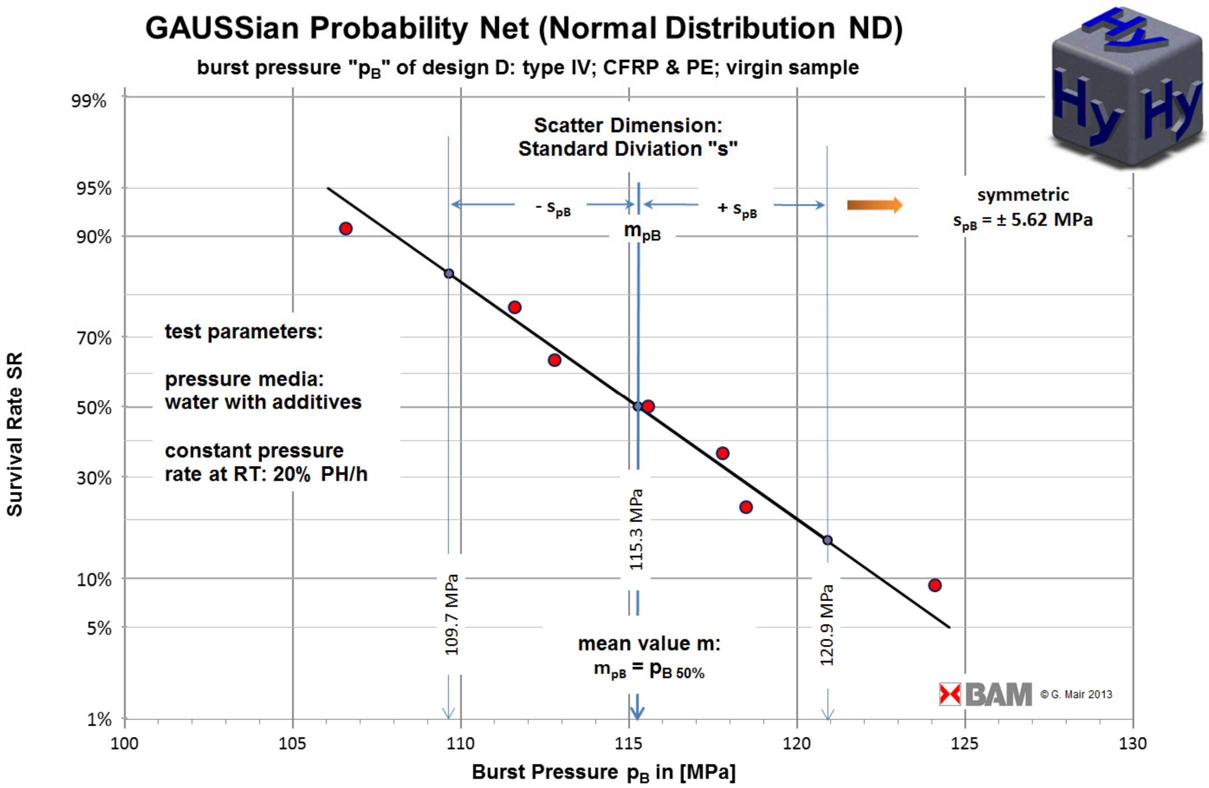


Figure 1: Plot of individual test results, mean value and standard deviation from Table 1

The first choice for determining the load value of pressure receptacles for the transport of gases as dangerous goods is the test pressure PH. In general gas services it describes the maximum pressure that can be achieved by gas within the temperature range up to 65°C. Thus PH can be interpreted as MAWP (maximum allowable working pressure). For dedicated gas services it can be more accurate and helpful to avoid unnecessary safety factors to use the gas specific pressure that develops at maximum temperature as shown in [8]. This approach is common for tube trailers, breathing apparatuses or even on-board storage systems for automotive application (CNG or $CGH_2$).

By relating the values - as shown in Fig 1 - to test pressure PH (= 150% NWP or 150% PW) Fig 2 develops. Further down in this paper all test and assessment results are expressed as percentile of test pressure or as load multitude of PH.

The description of the mean value $m_{pB}$ as a multitude of the reference pressure (here: test pressure) is called $m_{rel}$. For the purpose of safety assessment it equals $\Omega_{50\%}$:

$$\Omega_{50\%} = m_{rel} = \frac{m_{pB}}{PH} = \frac{1}{n}\sum_{i=1}^{n}\frac{p_i}{PH} \qquad [-] \qquad (3)$$

Relating standard deviation $s_{pB}$ to reference load (PH) creates $s_{rel}$. This is already very useful for general description:

$$s_{rel} = \frac{s_{pB}}{PH} = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}\left(\frac{p_i - m_{pB}}{PH}\right)^2} \qquad [-] \qquad (4)$$

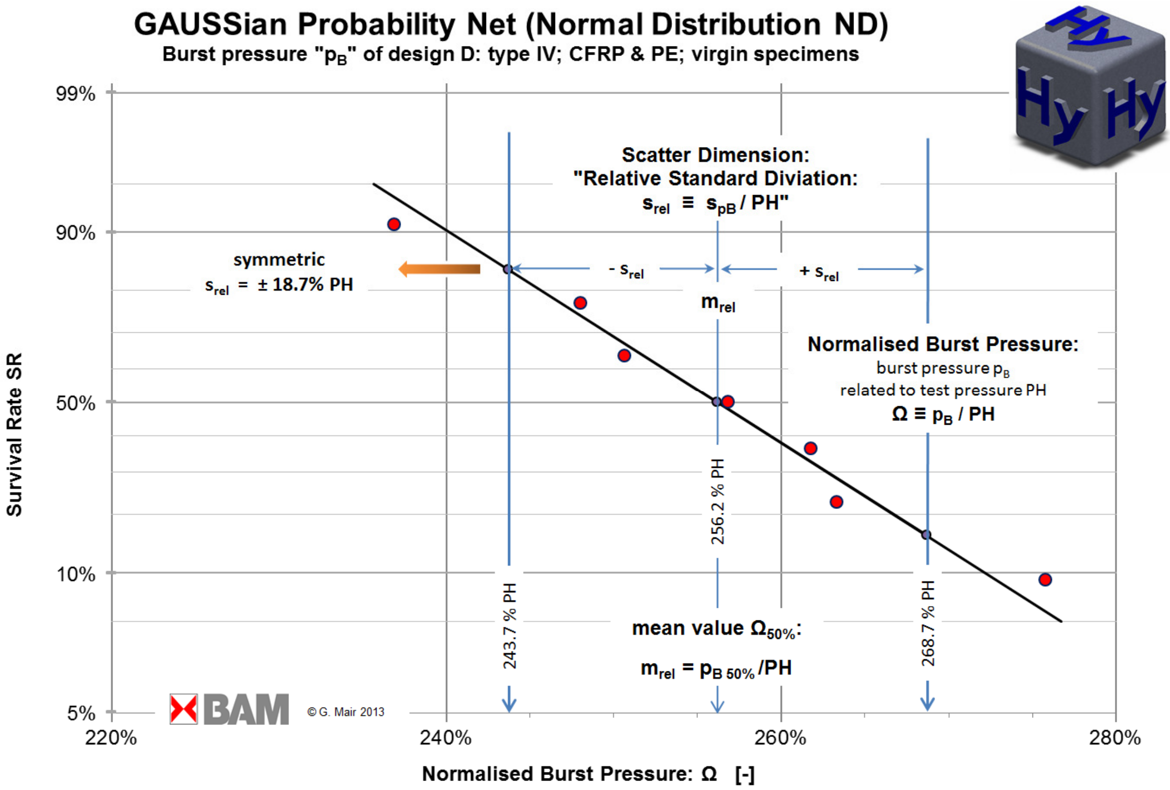


Figure 2: Results of Fig 1 after normalisation of data (Division by test pressure PH)

More commonly found in literature (as e. g. [9]) is the so called "scatter spread" (DE: "Streumaß"). It is used normalized with PH in Fig. 3 as "relative scatter spread" or "relatives Streumaß" in combination with the normalised burst pressure Ω as introduced in Fig. 2.

The outcome of this combination is the "relative scatter spread ψ". It is the difference of the strength value with a survival rate of 10% minus the strength value with a survival rate of 90% related to the reference load pressure (here: PH):

$$\psi \equiv \Omega_{10\%} - \Omega_{90\%} \qquad [-] \qquad (5)$$

By using the definition of Ω the "rel. Streumaß" relates to the relative standard deviation as follows:

$$\psi = \frac{p_{B10\%}}{PH} - \frac{p_{B90\%}}{PH} = 2.563\,\frac{s_{pB}}{PH} = 2.563\,s_{rel} \qquad [-] \qquad (6)$$

Based on this it becomes possible to compare different test samples – even of different design types of different pressure levels – directly in one diagram. Additionally it is possible to derive the strength of the most interesting survival rates of 90% and 10% directly out of the diagram.

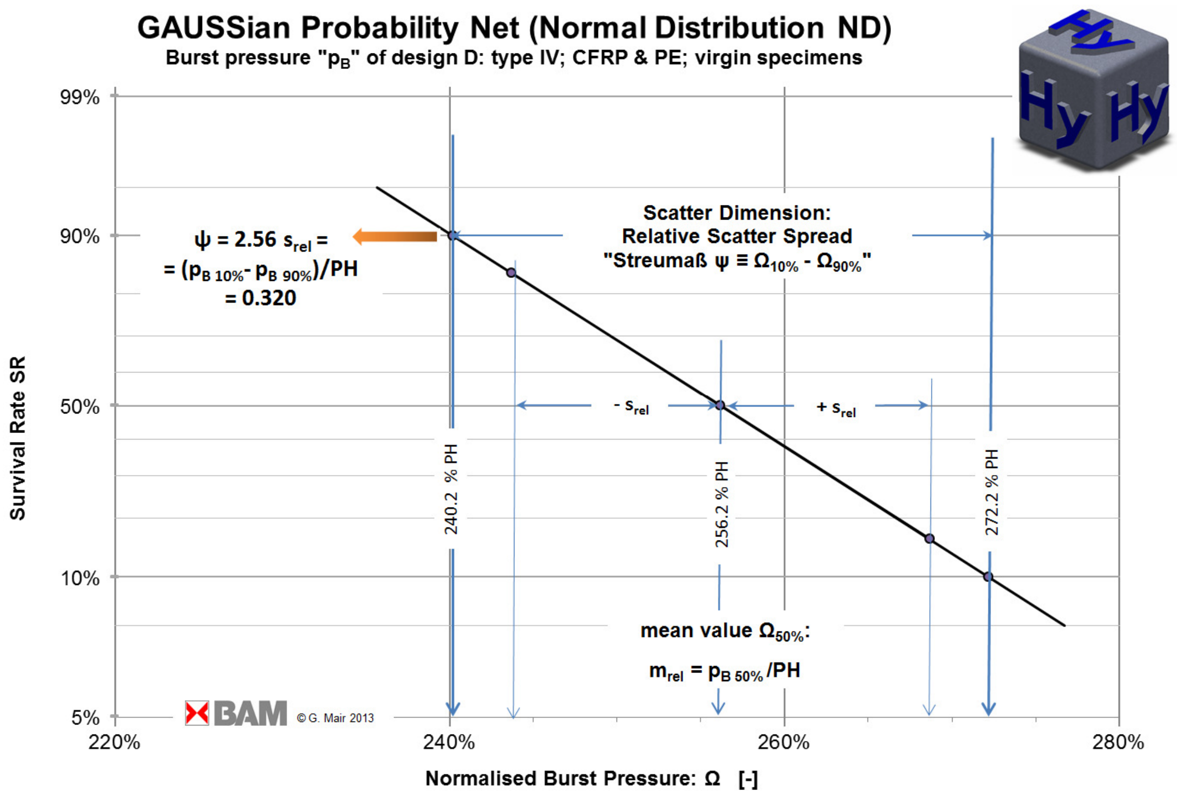


Figure 3: Values of Fig 2 by using "Streumaß" for quantification of relative scatter

The original intention of the assessment is to present the strength of a sample by a pair of values (x; y) to enable the comparison of sample properties. Following this approach the test results of a sample become a single point as shown in Fig. 4.

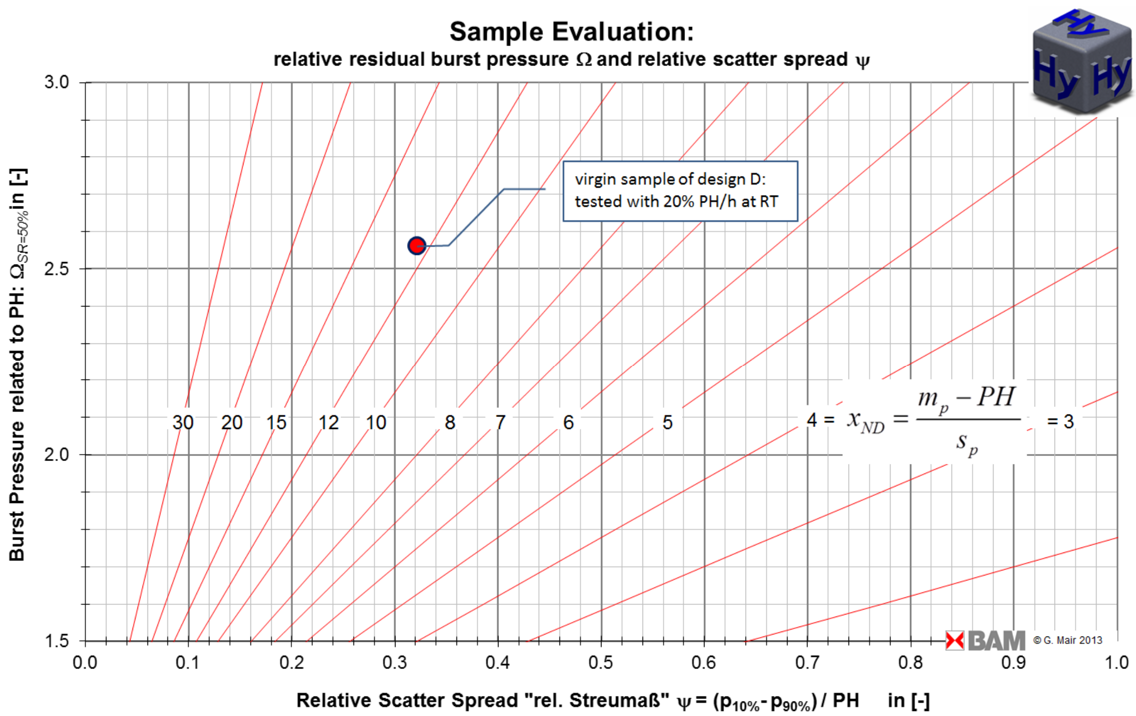


Figure 4: Result of evaluation of the slow burst tested sample of virgin specimens of design D

This kind of diagram is suited for the comparison of several samples of a certain design type concerning virgin samples' strength (as e. g. batch testing), its degradation in service (parallel to operation) or for the long term surveillance of manufacturing quality (parallel to manufacturing) by assessment of batch tests. Each elaborated strength point can be compared directly with lines of tentative reliability requirements:

$$reliability \sim \frac{strength - load}{scatter} \tag{7}$$

The lines of constant reliability ("isoasfalia"), as shown in Figure 4, are described by the standard deviation of ND. The standard deviation value x of ND is used here in the case of a dedicated load case with on $p_{reference}$ or for general service with PH as:

$$x_{ND} = \frac{m_p - p_{reference}}{s_p} \xrightarrow{p_{reference} = PH} x_{ND} = \frac{m_p - PH}{s_p} \quad [-] \tag{8}$$

This graph and its graphic assessment can be used generally – independent of the pressure level. This is the most important advantage of this kind of evaluation.

If the scatter is known for an adequate size of samples a further estimation can be elaborated. By using the correlation data of ND and the two parametric WEIBULL-distribution (WD with $\varepsilon = 0$) as shown in Table 2 it is possible to search for dedicated values of standard deviation $x_{ND}$. So lines of constant survival rates or reliability - even for higher reliability levels - can be allocated. The survival rate estimates are based on a spread ND. An example is shown by the red lines in Fig 5.

Table 2: Adjustment factor $\chi$ for the point-by-point spread of ND to WD (from [10])

| Survival probability (rate) $P_Ü$ (SR) = 1-$P_A$ | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| $P_Ü$ | 50% | 90% | 99% | 99,9% | $1\text{-}10^{-4}$ | $1\text{-}10^{-5}$ | $1\text{-}10^{-6}$ | $1\text{-}10^{-7}$ | $1\text{-}10^{-8}$ |
| Value of standardised deviation x of normal distribution (ND) | | | | | | | | | |
| X | ± 0 | - 1.282 | - 2.33 | - 3.10 | - 3.72 | - 4.27 | - 4.76 | - 5.20 | - 5.67 |
| Adjustment factor $\chi$ for spreading to WD | | | | | | | | | |
| $\chi$ | - | - | 1.53 | 1.79 | 2.01 | 2.21 | 2.29 | 2.56 | 2.72 |

From a safety standpoint relative minimum burst pressures are only sufficient if scatter is small. The design type shown only achieves its acceptable starting reliability by overly elevated burst pressures.

Taking into account the intended use of the investigated cylinders in a breathing apparatus it is worthwhile to revaluate the load level. By filling cylinders with compressed air up to 300 bars at 15°C the resulting pressure at 65°C raises to only 84.8 % of test pressure PH (see Table 4 in [10]). This reduced maximum pressure increases the safety through an increased numerator of equation (8). An enlarged distance can be observed of the sample's point in Fig 5 to the blue lines for each reliability value.

Some further assessment of the evaluation of test results shown here is described in [6] and leads to the recommendation to improve the burs tests procedure. A similar evaluation of sample test result as shown here will be given in [7] for load cycle tests based on [10].

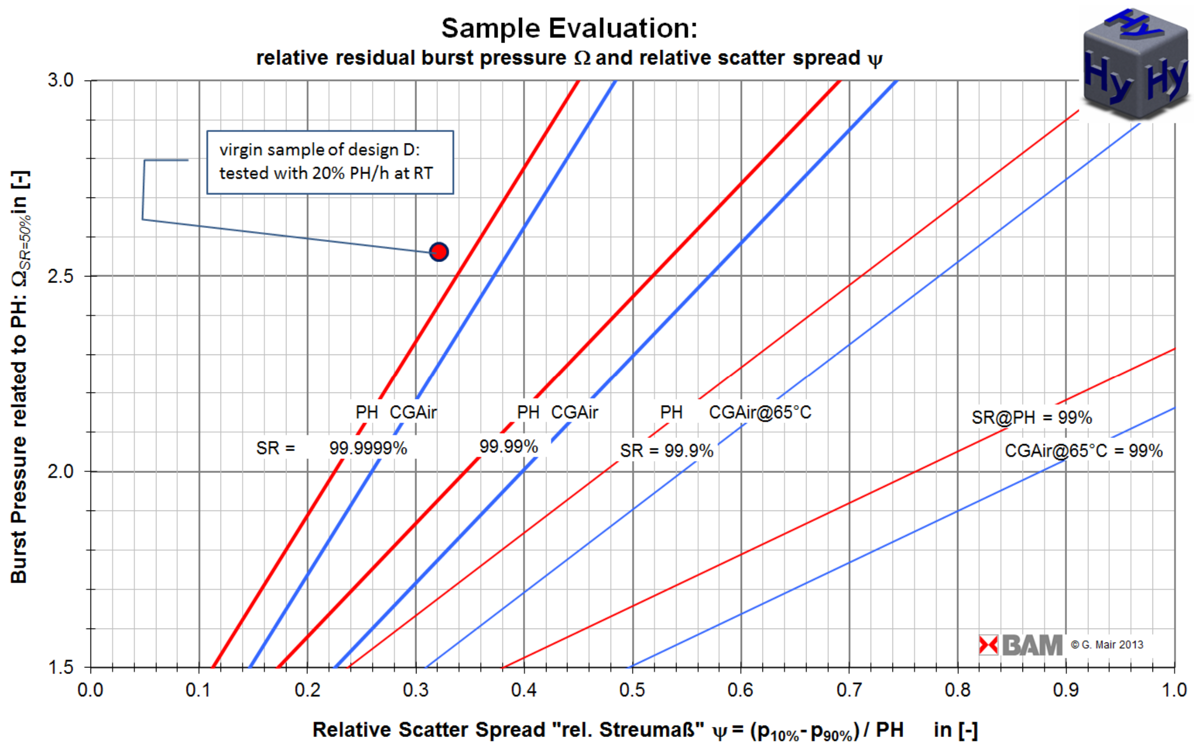


Figure 5: Comparison of tested sample properties with lines of reliability

## 3.0 CONCLUSION ON THE EVLAUATION OF SAMPLE TEST RESULTS

For assessing various specimens of a design type as one sample they must have had a comparable previous service history. Under this condition test results can be analysed and plotted with two parameters (mean strength and scatter) using the applicable method for the respective test procedure. Using the introduced diagram any parameter set of a sample can be rated approximately regarding its survival rate.

It has to be emphasized, that the narrow data base is a weak point of this assessment. The comparably high cost for each specimen and the accompanying burst or cycle tests currently limit the size of the samples for economic reasons. However a larger data base could lead to improved design standards which would allow for material saving or longer test cycles. The manufacturer or operator providing sufficient data should be provided a chance to benefit from this, while a limited statistical foundation should result in additional safety margins. This could justify increased spending on testing.

The introduced assessment is a tool for creating rough estimates, not precise predictions, of uncertainties from degradation, service life estimates and safety margins. Small samples, down to just five specimens are sufficient for this. In a first step a symmetrical Normal Distribution is used. Assuming a twin parametric WEIBULL distribution WD is describing extreme values more conservative, ND is used in a second step, spread by adaptation factors on the WD towards the safer side.

If the effort for larger samples can be justified, the resulting improved data should be used in the described manner to create more reliable estimates. One way to achieve this with acceptable cost could be to include batch tests during manufacturing into data base. Those would only require some minor adjustment in the testing methods and continuous statistical analysis. Investigations if a more sophisticated distribution like 3 parametric WD would better characterize the sample behaviour should start if 11 …. 21 specimens or more are tested per parameter set. This will have to be discussed later.

Regardless of the obvious weakness of the sample tests the introduced method of assessment permits more qualified estimations than the current standard tests. The improvements are very limited if sample size is smaller than 5 specimen though. Referring to Figure 5 samples can be compared using their average strength and scatter. Even single samples can be evaluated rudimentarily regarding their reliability.

For this reason BAM demands assessment of a virgin sample right from production for approving the common test period of 5 years in [1]. For even longer retest periods it is required for the manufacturer or operator to track the degradation of strength during service life with accompanying sample testing.

## REFERENCES


[1] Federal Institute of Materials Research and Testing BAM: official publications (online): Determination of Retest Periods of Composite Pressure Receptacles by the competent authority including the re-assessment of service live after 15 years for UN-composite pressure receptacles (PDF);
http://www.bam.de/en/service/amtl_mitteilungen/gefahrgutrecht/druckgefaesse.htm

[2] Mair, G. W.; Pöschko, P.; Schoppa, A.: Verfahrensalternative zur wiederkehrenden Prüfung von Composite-Druckgefäßen.
Technische Sicherheit (TS) Bd. 1 (2011), Nr. 7/8 (Juli/August), S. 38 - 43. ISSN 2191-0073

[3] Mair, G. W.; Duffner, E.; Schoppa, A.; Szczepaniak, M.: Aspekte der Restfestigkeitsermittlung von Composite-Druckgefäßen mittels hydraulischer Prüfung.
Technische Sicherheit (TS) Bd. 1 (2011), Nr. 9 (September), S. 50 - 55. ISSN 2191-0073

[4] Mair, G. W.; Pöschko, P.; Hoffmann, M.; Schoppa, A.; Spode; M.: Betrachtung von Grenzwerten der Restfestigkeit von Composite-Druckgefäßen, Teil 1: Kriterien der hydraulischen Lastwechselprüfung.
Technische Sicherheit (TS) Bd. 2 (2012), Nr. 7/8 (Juli/August), S. 30 - 38. ISSN 2191-0073

[5] Mair, G. W.; Duffner, E.; Schoppa, A.; Szczepaniak, M.: Betrachtung von Grenzwerten der Restfestigkeit von Composite-Druckgefäßen, Teil 3: Phänomene der Berstprüfung.
Technische Sicherheit (TS) Bd. 2 (2012), Nr. 111/12 (Nov./Dez.), S. 43 - 50. ISSN 2191-0073

[6] Mair, G. W.; Hoffmann, M. and Schönfelder, T.: The Slow Burst Testing of Composite Cylinders Part I: Slow burst testing of samples as a Method for Quantification of Cylinder Degradation; Presentation at the „International Conference on Hydrogen Safety ICHS“, Brussels: Conference Proceedings 2013; ID 102- Part I

[7] Mair, G. W.; Hoffmann, M. and Scherer, F.: Statistic Evaluation of Sample Test Results of Composite Cylinders.
MP Materials Testing, Volume 55 (2013) Nov/Dez., p. N.N.; ISSN 0025-530

[8] Federal Institute of Materials Research and Testing BAM: official publications (online): SBT (Slow Burst Test): Procedure and assessment of hydraulic slow burst tests on Xy-composite pressure receptacles (Xy-CPR) (PDF);
http://www.bam.de/en/service/amtl_mitteilungen/gefahrgutrecht/druckgefaesse.htm

[9] Research Project HyCube - Hybrid HYdride HYdrogen pressure storage EU: KIC InnoEnergy Innovation Project KIC-CCAV - 02_2011_LH02_HY3…

[10] Mair, G. W.; Hoffmann, M.: Assessment of the residual strength thresholds of composite pressure receptacles - Criteria for hydraulic load cycle testing;
MP Materials Testing, Volume 55 (2013) 2, p. 121 – 129; ISSN 0025-5300