

Chapter 2

Test Procedures for Hydraulic Sample Testing

Abstract Currently, in particular static strength, determined by a burst test, as well as operating strength, obtained via load cycle test, is seen as central safety-related properties. According to current norms and standards, the determination of these strength properties is done on single test specimens for verification. As mentioned below, strength properties may vary from component to component as well as from material specimen to material specimen, too. This makes it essential to quantify statistically and to assess the respective characteristic of strength on the basis of several test specimens. For the statistical calculation of strength properties, there are central aspects which are discussed in the following. On the one hand, in Sect. 2.1 arguments are explained why the reproducibility of tests is essential, and according to which aspects, reproducibility of the load cycle test can be increased. On the other hand, in the following Sects. 2.2–2.4 the question of the appropriate test procedure is pursued. Whenever the load cycle test does not lead to the desired knowledge the creep rupture test has to be discussed alternatively. In this context, the slow burst test is developed and described step by step in order to substitute the creep rupture test (see Sects. 2.2 and 2.3). First experiences are shown in 2.4.

All common procedures for the evaluation of safety and service strength of components are based on strength property testing. These are usually determined through destructive testing on individual specimens, i.e. test specimens. Non-destructive test procedures are usually applied to each product additionally in order to survey the quality and absence of defects of the material or joints. Under very restrictive conditions and in a few cases, it is possible to roughly estimate the utilization factor under a specified load through procedures of non-destructive testing, such as acoustic emission analysis and X-ray refraction. The utilization factor is defined by the ratio of material stress to capability. As it is currently difficult to apply such estimations on whole components (e.g. composite cylinders; abbreviated CCs) under laboratory conditions and certainly not in the field, only destructive tests can be used to determine essential safety properties. This does not ignore the great development of non-destructive tests (NDT) in the last years, but the interpretation of their test results is still mainly based on destructive reference tests.

Despite of this, there are a lot of non-destructive test (NDT) methods under development for composites and CCs. These are, e.g., acoustic emission testing AT, ultrasonic testing UT, porosimetry, X-ray, computed tomography, X-ray refraction, tangential radiography, guided wave testing GWT, eddy-current testing ECT, metal magnetic memory, infrared thermography IRT, thermal imaging, optical 3D deformation and elongation measurement and some other very promising NDT methods with potential for CCs.

Some of these could be developed to a reliable tool for black-white decisions about the current condition of a CC within the frame of the periodic 100%-testing. Others might become test procedures for the check of current properties, but might be treated as destructive methods if in future there is still a requirement to reduce the weight of CCs. But the most promising NDT methods might be developed to a tool that allows the measurement of the individual residual strength without any destruction. This NDT tool could enable the creation of statistics on residual strength and finally to substitute the destructive tests, such as the slow burst test or the load cycle test, used here for the probabilistic monitoring of degradation of safety.

But on the way to this aim, it is recommended to validate the functionality and reliability of each NDT method intended for safety assessment by the probabilistic approach as described in the following in combination with the proposed, destructive tests.

Today, static strength in the meaning of a burst test and the service strength in the meaning of the load cycle strength are seen as essential safety relevant features. As explained later on, strength properties scatter from component to component as well as from material specimen to material specimen. Hence, it is essential to quantify and evaluate the particular strength characteristic on the basis of several specimens statistically in order to serve the increasing interest in cost/weight-saving optimisation.

For the statistical assessment of strength properties, there are central aspects, which will be discussed in the following. On the one hand, in Sect. 2.1 it will be explained why the reproducibility of tests is essential and how it can be increased when performing load cycle tests. On the other hand, the question of the appropriate test procedure will be treated in the subsequent Sects. 2.2–2.4. If the load cycle test does not result in the intended knowledge, the creep rupture test will be discussed as an alternative. In this context, an approach of using a slow burst test instead of creep rupture test is deduced step by step in Sect. 2.2 as well as described in Sect. 2.3. First experiences will be shown in Sect. 2.4.

The aspects of interpretation and evaluation of results are not taken into account here as a part of the determination of strength properties and will be considered separately later on.

2.1 General Aspects Regarding Reproducibility of Test Procedures

The results of the tests serve different purposes. Often, minimum properties should be demonstrated through general practice with individual tests. More often, test results are used to get conclusions out of direct comparison. Principally, this counts also for those tests, which are applied as test series to a statistical evaluation. In all these tests, it must be ensured that the test results provide comparable values independently of the test centre or the test facility. However, this is a vague requirement. Within an evaluation concept of tests demonstrating minimum values, this requirement makes an impact exclusively in the rare case of borderline properties. Nevertheless, its importance should not be underestimated according to its practical importance in critical cases.

In tests, where results shall be evaluated quantitatively (i.e. determination of the actual property instead of a pure yes/no assessment) or even on which basis a statistical analysis will be made, a significantly increasing aspect according to the comparability of the values has to be mentioned.

In the context of the following statistic of properties, this means that the obtained scatter of these properties increases significantly when using changed or inconstant test parameters. In doing so, the obtained scatter increases even more than the actual scatter of the property of the sample. To avoid this for statistical evaluations, it is essential to specify the test requirements in much more detail than usually defined and currently known. This also includes that the specification of minimum or maximum values for parameters is not sufficient anymore. Instead, a preferably small range of test parameters has to be specified and bindingly arranged by setting minimum and maximum values.

Before focusing on burst and creep rupture test procedures in the following Sects., it should be shown through the discussion of the load cycle test at which points an improvement of the currently standardized test parameters is desirable in order to reach a sufficient degree of reproducibility for statistical purposes.

2.1.1 *Reproducibility of Cycle Tests*

Tests, which simulate frequently recurring load conditions, in materials science often called vibration tests. This name comes from the clear swing of the clamping jaws, e.g. flat test piece at the usual load cycles frequencies from 1 to 300 Hz, i.e. vibrations per second.

Such a high load cycles velocity is not feasible for testing components. This applies in particular if—in the case of the pressure vessels—for the simulation of the operating load pneumatic or hydraulic pressure must be applied.



Fig. 2.1 Hydraulic load cycling system with its test chamber shown in the *right* picture and its pressure generation in the *left*. The hydraulic pump is on the *left side* on the *left* picture while the pressure intensifier is in the *centre*.

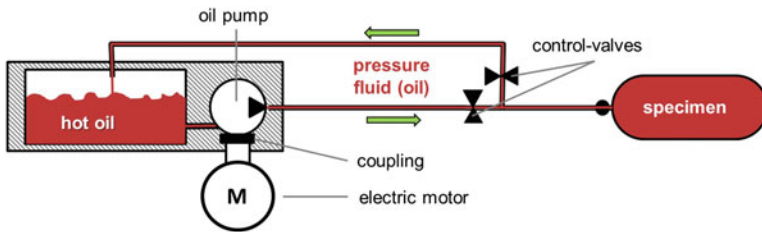
In cycling facilities such as the one shown in the Fig. 2.1, a load cycle velocity (frequency) up to 0.5 Hz can be implemented for small gas cylinders under the best conditions.

Often the neck threads limit the volume flow of the test medium so that depending on the boundary conditions only 3 up to a maximum of 15 load cycles per minute (0.05–0.25 Hz) can be driven. The expenditure of energy for such tests is high. It depends on the method for pressure generation, load cycle velocity and test specimen volume. The required electric power demand can go up to 200 kW and more.

Against the background of high energy consumption, the energy efficiency in running the facility is often a major issue. The energy efficiency depends very much on the type of pressure generation. Basically, the types of pressure generators can be distinguished between two principles as shown in Fig. 2.2: the pump operated (open) single-circuit system (upper part) and the pressure intensifier-based dual-circuit system (below).

The direct drive pump is a low energy efficiency type of pressure cycling. The test fluid is here mostly hydraulic oil. The oil gets compressed while its flow, and thus, the pressure in the CC-test specimen gets controlled by a valve (throttle control). In the discharge phase, (pressure relaxation) the test fluid flows back into the reservoir while it often gets decompressed by a diaphragm. In this process, the entire compression energy is converted into heat. For the next cycle, the pump takes the test fluid from the reservoir again, and so on. These systems require significant cooling effort and are basically hardly used for tests at defined narrow limits of room temperature. Since the pumps require a certain operating temperature, this group of equipment is not suitable for low temperature tests. It would require an enormous cooling capacity to heat up or cool down the test pressure medium to the required temperature after the pump before it goes into test specimen; and then to condition it back to the temperature required by pump.

Open one-circuit hydraulic system



High efficient dual-circuit hydraulic system

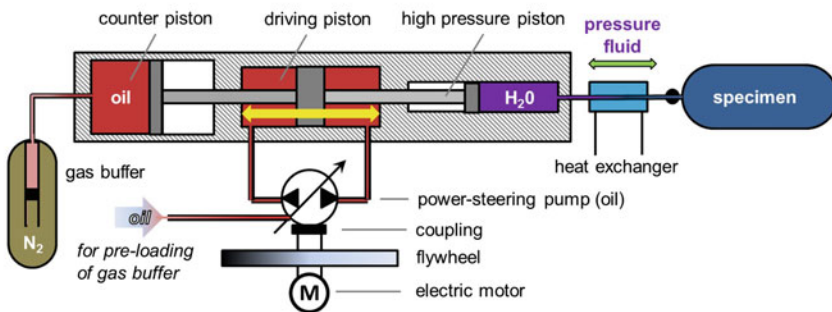


Fig. 2.2 Schematic sketches on the principle of operation of hydraulic load cycle facilities: simple single-circuit facility with open oil system (*top*) and dual-circuit system with complex elements to maximize the energy efficiency (*bottom*)

The counterpart to this is the group of facilities with pressure intensifier-based systems, like this is shown for example in Fig. 2.1 and the in lower part of Fig. 2.2. In these systems, there are a primary and a secondary fluid circuit. With the oil-based primary circuit (throttle valve or displacement control), a pressure intensifier is driven here instead of a CC-test specimen. The pressure intensifier then acts via the second hydraulic circuit with water or a water–glycol mixture on the test specimen (CC). A major part of the test fluid cyclically moves as liquid column between the pressure intensifier and the test specimen. Thus, the precondition is created to regulate the test fluid efficiently and to adjust wide areas of the secondary circuit to a specified temperature.

At the bottom of Fig. 2.2, the principle of a very complex but energy-efficient displacement control system is shown. It has an additional third hydraulic circuit, which acts on a preloaded gas buffer, and a flywheel mass for further improvement of energy efficiency. In this type of plant to which also the system shown in Fig. 2.1 belongs, the pressure medium in the primary circuit operates in both directions on the primary piston. Accordingly, the depressurizing phase can also be controlled accurately and operated much more quickly, as this allows discharging by a pure relaxation of the test specimen.

In [1, 2], inter-laboratory load cycles tests (round robin test) are set out that have been arranged in the framework of the EU project StorHy [3] by test laboratories from four different European countries. Amongst other issues, they focused on the resulting pressure and temperature curves of different, specified in detail variations of the hydraulic load cycle test.

The shape of the pressure–time curve was set to a trapezoidal curve with upper and lower hold phases of 1 s like it is partially demanded from vehicle manufacturers for testing onboard storage systems (cf. [4–6]). The upper pressure level was set to 87.5 MPa (125% of 70 MPa), the lower one to 2 MPa. In this case, it was important to ensure that the real pressure extrema are outside of the red marked pressure range, shown in Fig. 2.3.

The results of the tests series on contour accuracy, as shown in Fig. 2.4, had to be rated differently:

- One partner featured maxima with additional pressure peaks, meaning that consequently, in addition to the number of cycles per minute, the minimum holding phase at the required pressure level is deemed to be not fulfilled.
- The next partner took the liberty, to increase the stop phase significantly while he met the initially specified load cycle number per minute, but he exceeding the lower pressure level systematically.
- One partner got much closer to the target curve, but had to struggle with his nonlinear increase and decrease of pressure.
- The last partner ensured the upper and lower pressure specifications well but did not meet the determined frequency of load cycles.

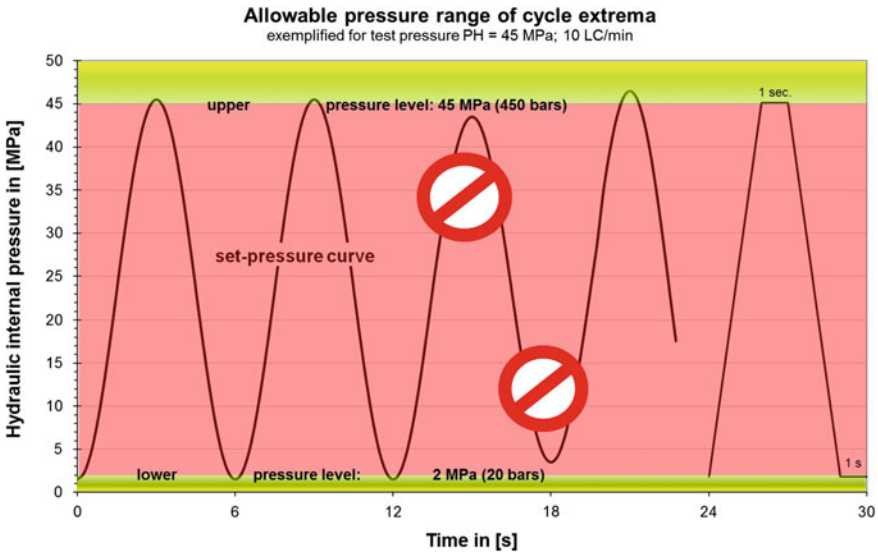


Fig. 2.3 Display of pressure cycle curves and the range of unaccepted extreme values (cf. [2])

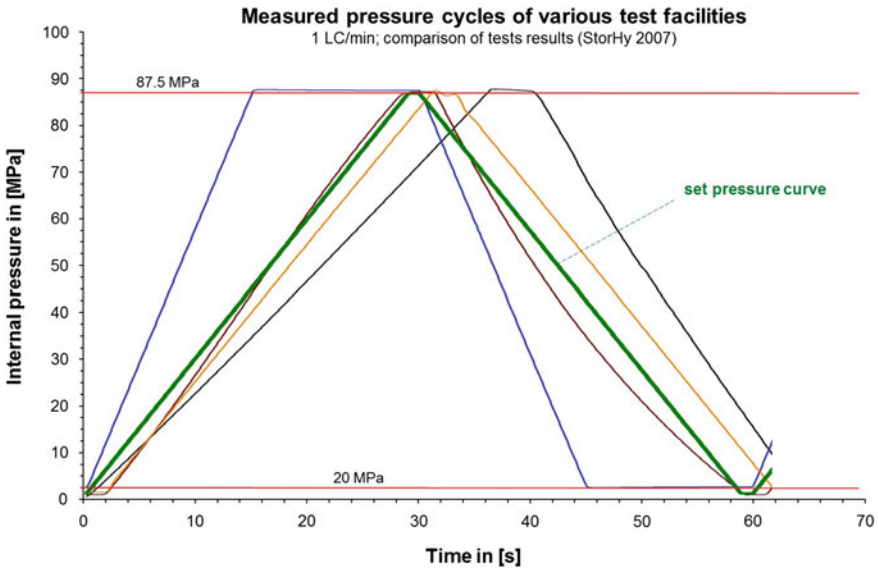


Fig. 2.4 Pressure curves of various hydraulic cycle facilities (cf. [1])

Furthermore, the reproducibility of cyclic tension or internal pressure loads is of essential interest as represented by the extrema (per mean value $\pm$ standard deviation) in Fig. 2.5. Here, the results of the tests done with 1, 2, 3, 5 and 10 LC/min are displayed for each partner, viewing from the left to the right side.

The results show:

- Facility A had a scatter of the upper values that are much larger than the scatter of minima values. Strictly speaking, only the tests with 3 and 5 LC/min fulfil pressure specifications. In all other cases, one of the extrema is located in the not accepted area.
- Facility B showed, particularly at 5 and 10 LC/min, a very small scatter of the pressure extremes. Big drawback is that all the values of the pressure extrema are shifted to higher pressures. The subsequent analysis showed that a pressure offset in the measuring chain was present, which can be seen as systematic measuring error. In order to indicate this kind of measuring error, not the real but the defective externally measured values are shown. In any case, it will be comparatively easy to explore this error and correct it, if control measurements within inter-laboratory test campaigns are done.
- The next facility C met the requirements only when more than 2 LC/min were done and showed a low scatter when starting from 3 LC/min.
- The fourth test system in the comparison (facility D) fulfilled the requirements to the pressure compliance in each case but shows an unnecessarily high strain for the test specimen because of the relatively large scatter and the large

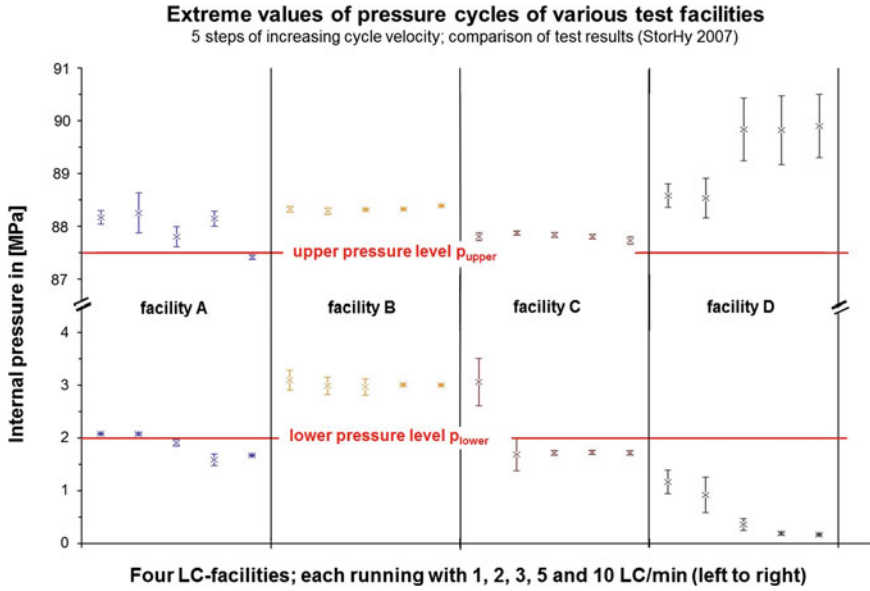


Fig. 2.5 Statistical view on the pressure minima and maxima (mean value with $\pm$ standard deviation; cf. [1])

distances to the set value. This means, it is on the one hand conservative, but on the other hand it constrains the reproducibility.

The results showed that the tests at room temperature with increasing load cycle frequency feature speed-dependent scatter widths between the pressure extrema (peak pressure and pressure minimum). On the whole, the cycle facilities worked relatively well with 10 LC/min. The preoperational tests with pressure sensors at both openings of the reference cylinder had demonstrated that the final test set up with measuring the pressure extrema at the adapter (i.e. pressure inlet) worked effectively even at higher frequencies. Nevertheless, higher load cycle frequencies were not tested. The tests would not have been possible for at least one of the involved test facilities.

Due to the partially clearly reprehensible deviations from the pressure specifications, one example is chosen to demonstrate what consequences are to be expected resulting from such “inaccuracies”. Figure 2.6 gives a first impression of the influence of pressure deviations on the extremum of the set point values. For the example analysis shown in Fig. 2.6, material data and calculation procedures taken out of the guideline “FKM-Richtlinie” [7] for the material 34CrMo4 are used.

In Fig. 2.6, the deviation of the lower pressure level is shown in contrast to the deviation of the upper pressure value. Parameter of the set of curves is the relative lifetime. Consequently, e.g. when speaking of a systematic deviation of the upper pressure level of approximately 0.7 MPa on top to the set pressure, in average a reduction of 10% of the expected load cycle strength compared to an ideally

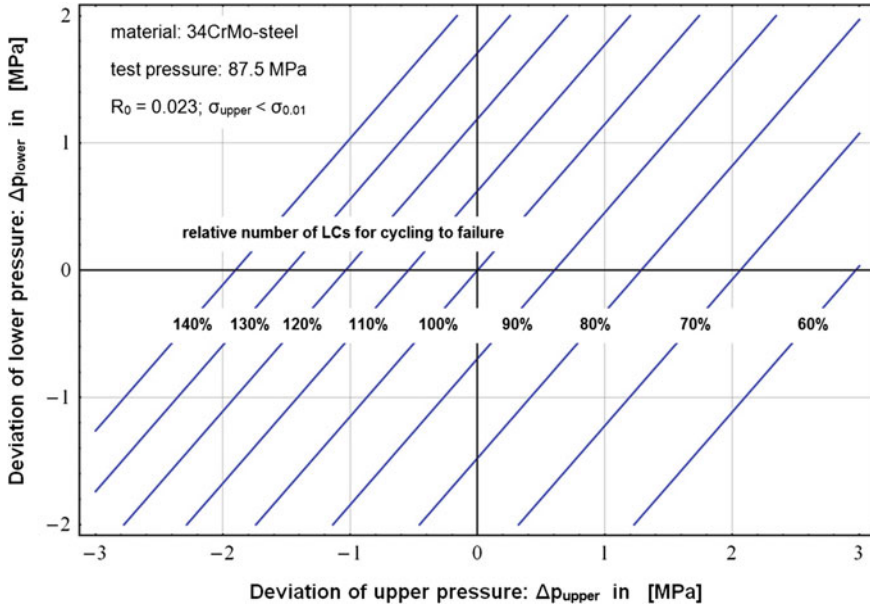


Fig. 2.6 Variation calculations on the influence of pressure deviations on the load cycle strength (cf. [2])

performed test can be noticed. As the lines of constant cycle life are inclined with an angle higher than 45° , it is obvious that the influence of the upper pressure inaccuracies is slightly larger than the influence of the deviations of the lower nominal pressure level.

In practice, high attention is given especially to the upper pressure levels. Hence, when using very low pressures the compliance of the lower pressure level can often not be set as accurately as the higher one due to control problems. Knowing this fact, it is surprising that the importance of both pressure limits is seen as almost to be the same as shown in Fig. 2.6. Consequently, it is necessary to pay more attention to a detailed description and exact control of minimum values below the lower set point than is currently common.

Since this influence is a material and design-specific aspect, the quantitative information of Fig. 2.6 can be used for indication only.

Furthermore, in [1, 2] the issue of possible harmonic components (right curve, green in Fig. 2.7) of the pressure in the test medium compared with the reference pressure curve (left, red; phase-shifted) was taken up. The result was defined to have an unexpectedly small impact on the overall test result.

Furthermore, the temperature behaviour within extreme temperature load cycles was observed. In this case, the two facilities, which were able to perform these test (out of the four ones which were used within the inter-laboratory tests) showed the following differences between the temperature curves as displayed in [1, 2].

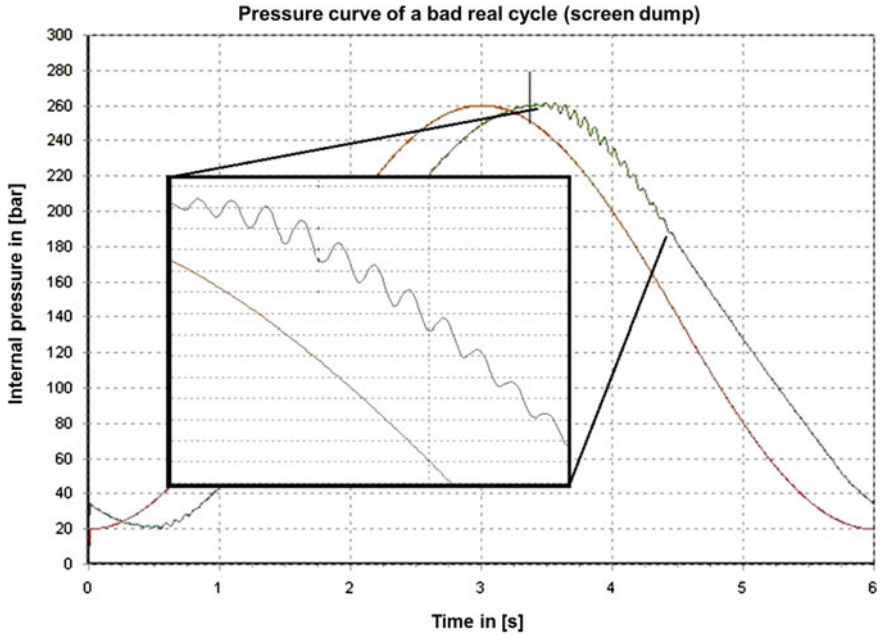


Fig. 2.7 Example of a harmonic component (*green*; shifted to the set-pressure curve in *red*); provoked by a wrong tuning of the control parameters (cf. [2])

An essential aspect of the comparability of the media temperatures in the test sequence is the question how the cooling of the pressure media is done, where measurements are done and whether the whole volume of needed test medium is pre-cooled or not. Especially, it is important to ask if at least that amount of fluid is pre-cooled, which is necessary to be pumped in the test specimen for generating the pressure level and if the pre-cooled medium is mixed significantly with the warm fluid which is pumped into the system cycle by cycle.

One concept which is able to ensure this is displayed in Fig. 2.8. It shows a modular unit based on a bundle of pipes working as a “cooling line” which conditions the medium and also prevents the usual mixing effects when being set to an appropriate adapted fluid capacity. Details can be found in the patent [2, 8].

In Fig. 2.9 various measurements are shown which were done at relevant positions according to Fig. 2.8.

In the tests shown here, the media within the “boss” (thread neck piece of metal of the CC) was cooled down to a temperature of $-45\text{ }^{\circ}\text{C}$ within several hours. As shown by the initial bend of the measured curves in Fig. 2.9, the medium reached an average temperature of just about $-20\text{ }^{\circ}\text{C}$ due to the good isolation properties of the composite material. These large temperature differences between boss and test medium within the test specimen result amongst others from the different thermal conductivities of the different materials. The boss and the little bit containing fluid cool down faster than the relatively large amount of the fluid within the

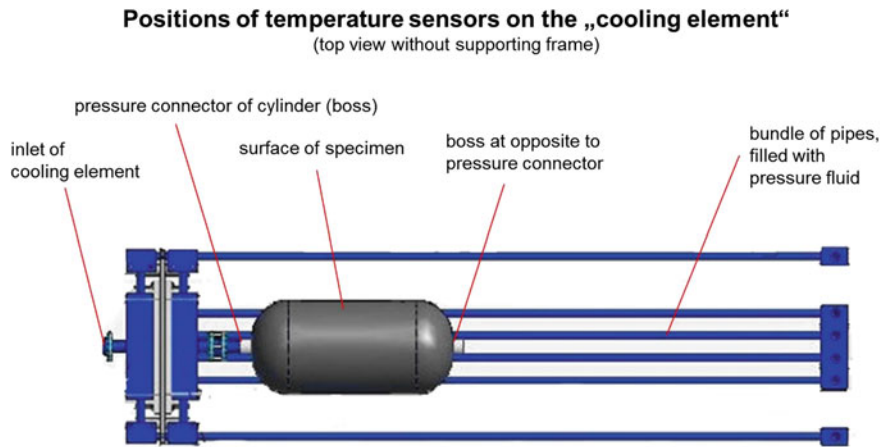


Fig. 2.8 Cooling unit based on a pipe bundle for the pre-conditioning of the pressure media with a mounted CC-test specimen; top view (cf. [2])

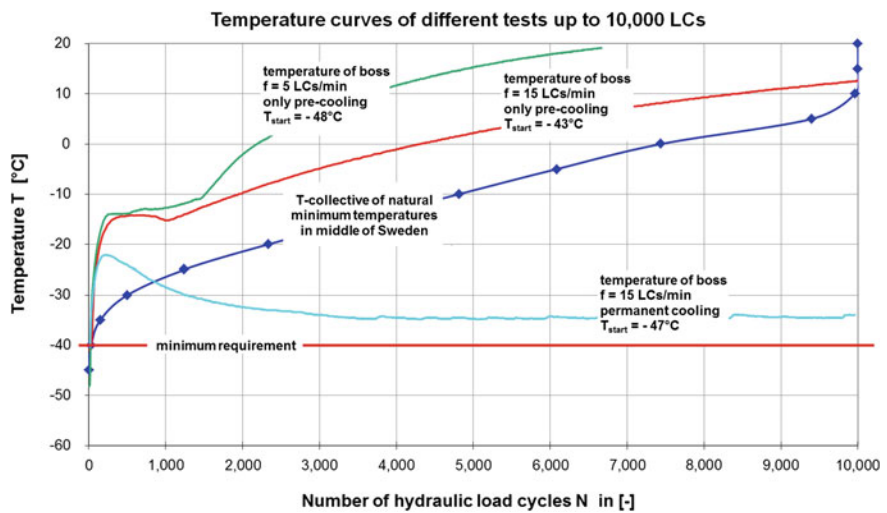


Fig. 2.9 Development of temperature during hydraulic cycle testing versus number of load cycles

(well-insulated) CC does. Therefore, the actually inside temperature of the CC can only be indirectly derived from the temperature curves of the boss just after having started the cyclic tests. Hence, experience is needed in order to reach the right temperature constantly.

In the case of the used hydraulic dual-circuit system, which is used here, the pressure cycles are realized by the cyclic oscillation of a liquid column (test medium) in the connecting pipes. The test medium is mixed at both ends, where the pressure media flows in a relatively large volume of test fluid. The filled CC is

situated at the one end, while at the other end, the pressure amplifier (piston with a defined swept volume) is working.

All analyses to Fig. 2.9 are based on the satisfied conditions of a sufficient quantity of pre-cooled test medium and a low mixing ratio of the pre-cooled fluid with the warm test medium in the cycle facility. To pre-cool the necessary quantity of test medium, which has at least to be equal to the used swept volume of the pressure intensifier, the cooling unit explained in [2, 8] and shown in Fig. 2.8 is used.

The patented design of this mobile cooling system (cooling line) was first built in 2005 and expanded for a larger volume capacity later on. The first installation of the originally implemented design is shown in Fig. 2.10.

The lower line of test results in Fig. 2.9 represents a test with a continuous cooling of the climate chamber down to a set temperature of not higher than $-40\text{ }^{\circ}\text{C}$. Due to the mixing of the (in this case still) too warm pressure medium in the test specimen with the sufficient pre-tempered test medium in the cooling unit, initially the temperature at the boss rises. This is followed by a turning point that shows that the cooled pipe system dissipates more heat as is produced by compression, flow friction and heat flux from the warm pressure amplifier. Consequently, the temperature of the medium drops until the whole system within the permanently cooled test chamber reaches a stable temperature level. If the temperature sensor is directly placed in the flowing test medium at the boss, the effect of the cycling fluid column will be observed during each cycle as shown in Fig. 2.11. Due to the (low) compression-related heat generation in the relatively well-isolated CC and the cooled

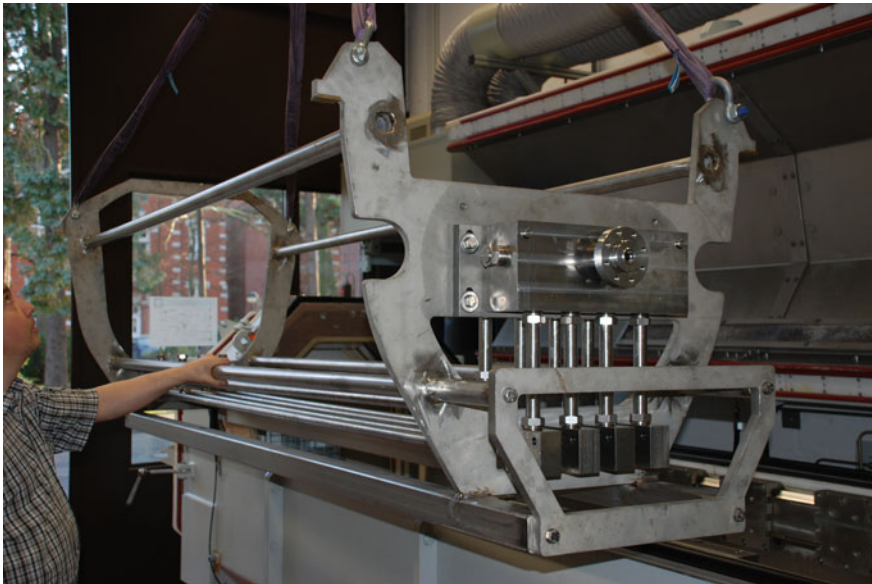


Fig. 2.10 Cooling line on the crane hook during the first installation

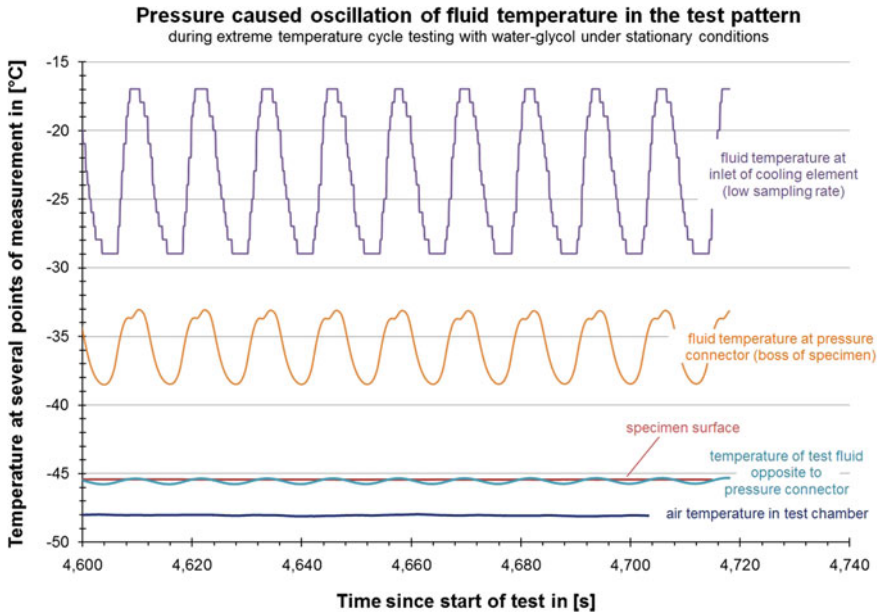


Fig. 2.11 Influence of the location of the temperature sensor (cf. [9])

pipe, the temperature difference never fully disappears even after having reached the temperature balance of the overall system.

An impression of the effectiveness of the cooling line is given by Fig. 2.12. The surface temperature of the test sample of approximately -40°C results in immediate generation of hoarfrost when opening the test chamber.

Furthermore, Fig. 2.9 shows a curve which is based on the statistical analysis of more than 30 years of continuous weather statistics in a place in the middle of Sweden (Jokkmokk; see [10]). Within this, the frequency distribution of the minimum temperatures is adapted to the 10,000 cycles as 100% of cycle life. This means a temperature curve that corresponds to this line would be adequate to the minimum of the expected minimum temperatures in Europe, shown in Fig. 2.13.

In order to realize this temperature profile, two different tests are shown in the upper half of Fig. 2.9. In both tests, the cooling of the test chamber and the pressure medium was switched off with the start of the cyclic loading. The curve increasing quicker in terms of LCs belongs to the cycle speed of 5 load cycles per minute (LC/min). The other of both curves describes a speed of 15 LC/min. Since the time per load cycle between the two curves differs by a factor 3, the upper curve shows a slower temperature increase in terms of time. Over the time, the general warming of the test chamber occurs in addition to the heat entry of the sample caused through compression and friction of the medium. Nevertheless, the slower testing increases only approximately twice as fast per cycle as the faster testing. This means a faster temperature increase for the fast testing method. Therefore, the hydraulic processes in

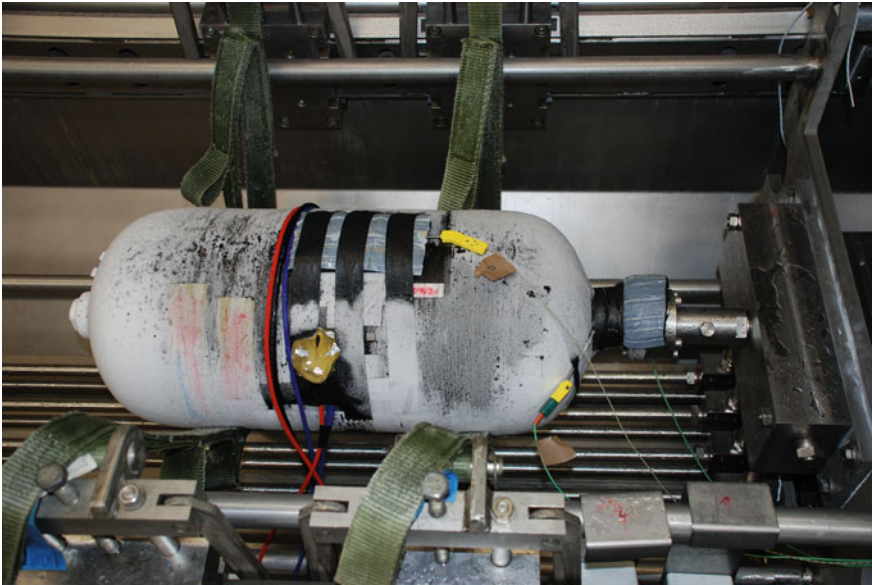


Fig. 2.12 Hoarfrost on a CC (type III, AL & CFRP) with temperature sensors

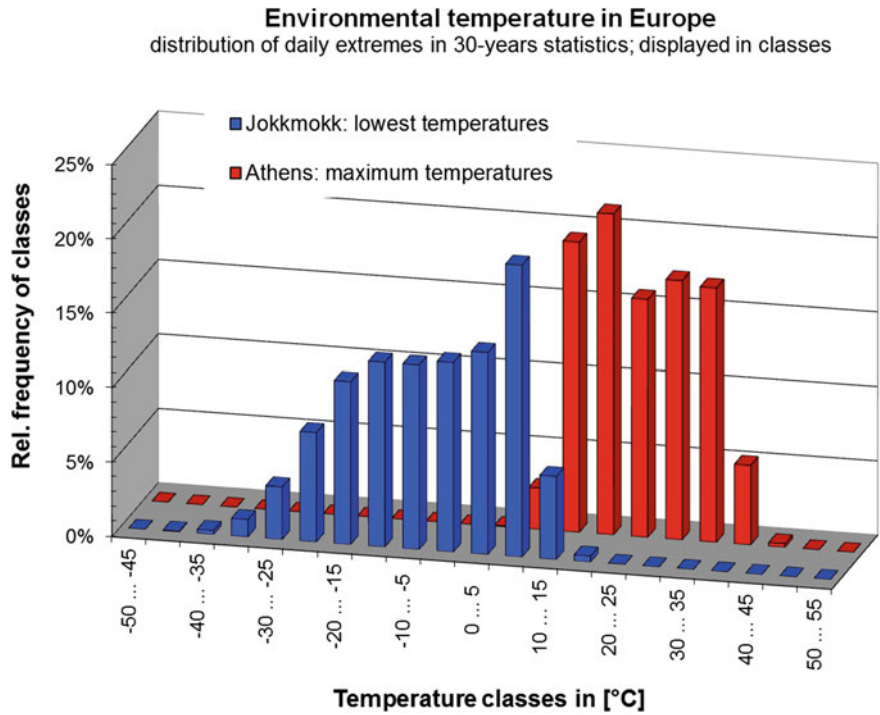


Fig. 2.13 Climatic differences in Europe: extreme values of the temperature

the test specimen and the pressure connector have a greater influence on the heat input than the average temperature loss caused by an averagely isolated, robust test chamber. However, this hydraulic influence depends on the ratio of the flow resistance in the pressure pipe to the test sample size. Hence, the requirements of temperature control of the test system increase depending on the adaptation of the test specimen.

For the reproducibility, this means that the specification of a constant temperature is generally better to hold and to check with basic measuring equipment than to rely on the specification of and control to a time–temperature set curve. The last mentioned method would require much higher control effort as well as the capability of a cooling/heating-control of the pressure medium and the test chamber. In case of uncontrolled temperature development of the pressure media, as it can presently be found in [4], it can be expected that the tests are run specifically to the equipment. This consequently means no reproducibility of test boundary conditions. Furthermore, a sufficient reproducibility will not be reached if a not tempered pressure media is used during the tests (as shown in [11, 12]), if the surface temperature is exclusively measured or if the temperature has to be kept in a range of 10 °C.

Overall, it must be noted that in particular the differences in reaching the extreme pressures but also the control of extreme temperatures are likely to influence the scatter of the results of cyclic load tests significantly. In particular, the temperature influences the behaviour of metallic parts (boss or liner) in connection with composite materials and plastic liner. Consequently, the scatter of the properties of the test specimen within a test sample would appear larger than it actually is. Therefore, it seems to be necessary to describe test procedures in more detail as well as to refine parameters and to specify them more restrictively. However, as the relevant test systems would have to be retooled in order to meet the requested requirements more precisely, this will probably cause some initial problems. But overall, this is a necessary step in order to create the preconditions for reducing the influence of the test facility and consequently to realize the statistical assessment of sample properties.

2.1.2 Reproducibility of Burs Tests

Relying on the historical beginning of early technical testing of pressure vessels, besides the non-destructive proof test the burst test can be seen as the simplest form of strength testing of internal pressure loaded components due to its elementary construction. Hence, within succeeding considerations focus is set on this testing method.

Experiences of the technique for pressure storage were made with steam boilers (“vessels”) and gas cylinders (“cylinders”), which have been made of steel. In case of this combination of material and design, it is irrelevant how fast the burst pressure is reached. Moreover, the question by which way the pressure level is reached was of little importance. Accordingly, the basic standard for vehicle storage

(ISO 11439 [4]) does not show any statistically sufficiently detailed specifications for the burst tests, too. Just the new GTR #13 [13] on hydrogen vehicles (cf. [5, 6]) applies a first and very limited statistical evaluation aspect, which will be discussed in Chap. 5. But also standards [11, 12, 14] for composite pressure receptacles (gas cylinders for industrial gases) rarely feature any specifications. They only feature a maximum burst rate which is almost impossible to reach when using usual composite pressure receptacles.

According to the standards, this maximum allowable pressure rate is shown on the left-hand side in Fig. 2.14. It can only be achieved with test equipment for the batch testing of large series of relatively small CCs. In most cases, the real pressure rates rather look like the green line shown in the centre. After a warm-up period, the pump works and charges the test specimen until, e.g., the metal of a steel bottle or e.g. the metallic liner in a CC begins to yield and the increase rate of volume per pressure becomes significantly higher. Despite the aspect of pressure control, the volume flow performance of the hydraulic pumps is usually not good enough to ensure the required increase of volume when working in the upper pressure range.

Considering these practical boundary conditions, a test featuring a constant rate of pressure increase of, e.g., 10 MPa/min cannot exactly be run on most test equipment. The wide range of the test specimen-dependent volume flow increases the problem. But as shown later on, the total test duration to failure of composite cylinders (CCs) does have a recognizable type-dependent influence on the burst test result. It is also likely that not only the time until the end of the tests but also the

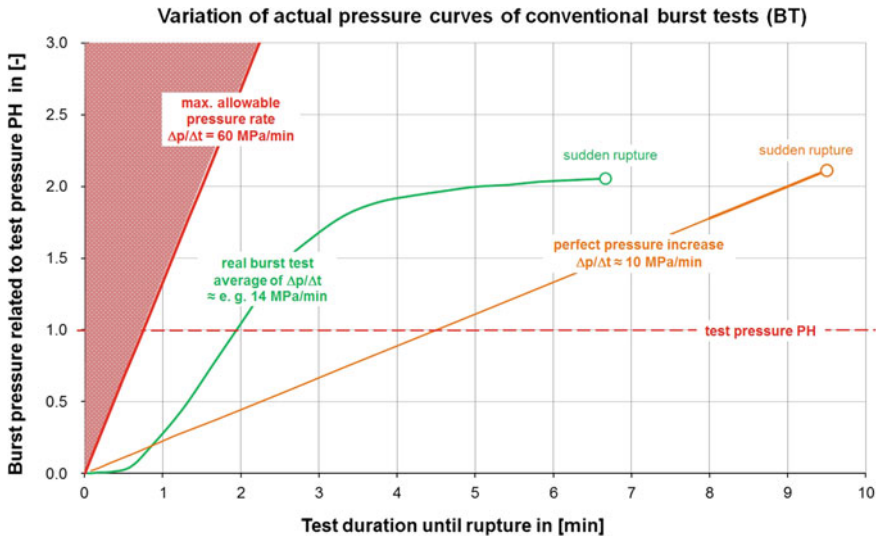


Fig. 2.14 Examples of the pressure–time curves for burst tests

form of the pressure curves do have an influence on the result of the test. Thus, the pressure rate has to be provided as a constant set value in order to maximize the reproducibility of the test results. Furthermore, it can be expected that additional boundary conditions such as temperature and moisture influence the test result, too. However, these influences are currently not captured or systematically investigated. Hence, as long as the contrary is not proved and in contrast to the metallic materials, specifications for the permissible range of those test parameters have to be made.

2.2 Influence of Time on Quasi-Static Test Procedures to Rupture

The essential value of the fatigue strength of CCs is the fatigue or cycle strength, determined through a pressure cycle test. In some cases, the cycle fatigue strength is derived from the static strength (burst pressure) within strict design specifications (e.g. [7]). This was (and partly still is) usual for steel vessels made of low-alloy steels. With increasing opportunities in the test engineering and equipment availabilities, design specifications were reduced, and on the way towards to a performance-based test approach, fatigue strength tests were introduced more and more.

At the end of the 1990s, the first international test standards for CCs were introduction. This was connected to a period where so-called performance-based standards started to be preferred. That means that tests focused on the service loads. This results in requirements for demonstration of appropriate minimum strength values and other properties. In the meantime, there is a dilemma has been detected with respect to CCs made of carbon fibres: on the one hand, almost none of the test specimen fails before reaching the minimum criterion, which usually means the abort of a cycle test. On the other hand, due to their high fatigue strength it becomes almost impossible to determine the load cycle reliability at room temperature. The effort for testing beyond a dimension of 100,000 load cycles is too cost- and time-consuming. This is valid for design type testing and in particular for the much higher number of production batch tests.

If the later described need requires a statistical determination of strength properties, a medium-sized test effort beyond the 100,000 and possibly even from 50,000 load cycles should be classified as unacceptable. However, as the necessity to determine the service strength in a statistical manner exists independently from the test effort, alternative test methods have to be included. This idea of an alternative test method refers to the task of the identification and quantification of strength values or residual strength values, only. The determination of the residual strength initially has nothing to do with the later discussed issue of the simulation of operating loads in the sense of an artificial ageing.

2.2.1 *Burst Testing of Composite Cylinders*

The short-term strength determined via burst testing is not a property directly related to the service strength. It basically shows the resistance against a singular overload. In contrast to metals and according to [9, 15, 16, 17] in the case of CCs, the more or less direct relation between the bursting strength and service life does not exist. In the 1970s, NASA did considerable efforts to determine sustained load strength (time to creep rupture) over 30 years. In the initial phase of the 1980s, the first results were published inter alia in [18, 19]. In the 1990s, these results were considered by the working group WG 17 of ISO/TC 58 (see [20]) and influenced the requirements for the design, construction and testing of CCs in succeeding standards.

References [18–21] show that the tensile strength of material samples decreases under ageing. But there is also a wide feedback from practice, supported by the investigations made in [22], which say that the burst strength of CCs increases during the first years of service due to static load. At the same time, the load cycling capability of CCs featuring a metallic liner decreases during service. Further investigations in [22–24] indicate that for individual cases the burst strength according to the probability of failure shows a higher reliability after 50,000 load cycles and under extreme temperature than a new sample of CCs does. This fact is initially seen as rather implausible. Consequently, it is an indication of a reasonable belief that the burst test performed in the usual manner is not an appropriate procedure for the check of service strength. Therefore, it cannot be used for assessing the degradation of service strength.

Looking back not that far in history of technology, one comes to the testing procedure of the creep rupture test (sustained load test). This testing procedure precisely shows the one aspect of the fatigue strength which is complementary to the cycle load test. Figure 2.15 shows the principle of a filling cycle of a fuel tank for gas. At the fuelling station, the onboard storage system inside the vehicle is filled up. Since temperature is increasing during the filling process, temperature and, thus, pressure decreases again after having finalized the fuelling. In case of a fuelling in the evening, this mostly overlaps with the subsequent short journey home (or to the office in the morning). After that the vehicle is parked overnight, then is used for driving to work, stands still again and is driven again—and so on.

This change of phases of movement and rest (parking) of the vehicle continues until a fuelling station is reached again, which starts a new filling cycle. While the filling and driving phases (red sections) are connected to changes of the gas pressure, the total lifetime dominant parking phases do always feature a roughly constant pressure. The reason why the consideration of these long phases does not have a dominant role in the current test requirements up to now can be certainly traced back to the experiences with metallic pressure vessels. Within the relevant temperature range, the metallic materials used are not subjected to any limiting degradation process or fatigue effects under sustained load. But referring to e.g. [25], when speaking about composite materials their visco-elastic-plastic properties

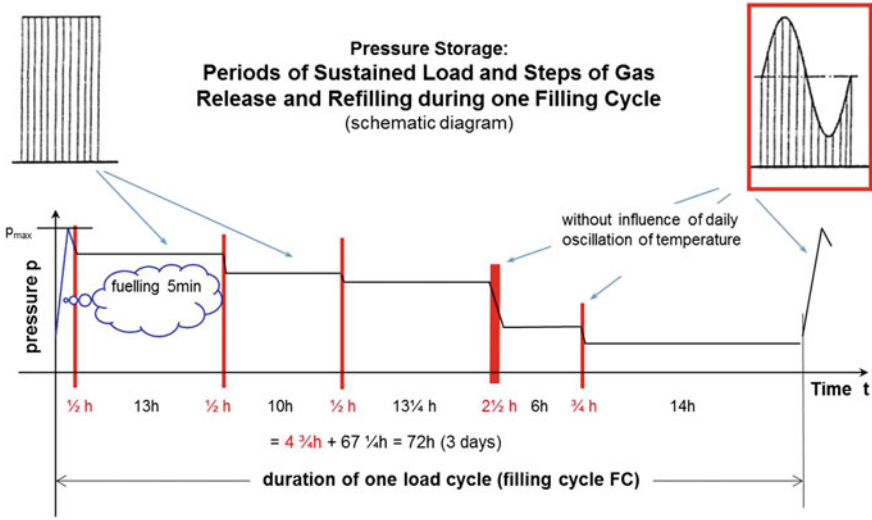


Fig. 2.15 Schematic sequence of a complete fueling cycle

means time-limiting factors with respect to service strength. Hence, the creep rupture strength can be seen as the counterpart of the load cycle strength.

An interesting micro-mechanical model describing the behaviour of composite material under sustained loading in the direction of the fibre has been created by BUNSELL and his colleagues. It has been explained in a series of papers ([26–31]), which will be referenced in the following later. This model is based on the principle of local accumulation (clustering) of filament breaks due to statistically distributed failures and statistically distributed strength behaviour in combination with a matrix-driven and therefore time-dependent load shifting. This principle is shared by others (cf. [32]) and explains a lot of effects.

This concept concentrates on sustained loads. But in service, it is always the combination of sustained and cyclic loads, of creep rupture strength and load cycle strength that is responsible for the failure process. On this basis, Fig. 2.16 displays four stages of an idealized degradation and failure process (A to D) up to the total failure in accordance with the above-mentioned model.

Especially, Fig. 5 in [30] shows that the relevant model visualized in Fig. 2.16 is appropriate for explaining a lot of effects. This is supported by the series of 4 pictures showing computed tomography experimental results. This series has been provided by ROSINI and is shown in Fig. 2.17.

For a better understanding of the dimension of the above-mentioned filaments in comparison with usual production influences, Fig. 2.18 displays an optical fibre for strain measurement embedded in a multi-layer winding composite of a CC. The optical fibre with a total diameter of about 250 μm (glass fibre with coating) makes it clear how small the individual fibre filaments are (approx. 6 μm). In comparison, the matrix enrichments with the non-distinctive air bubble inclusions are relatively

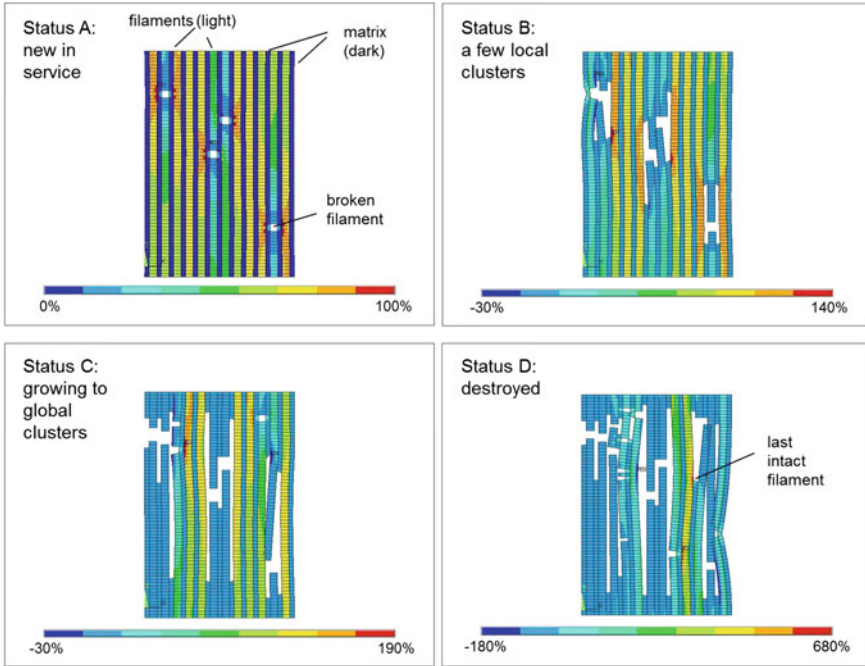


Fig. 2.16 Simulation of the degradation of composite with the increasing number of broken filaments (cf. [9, 17])

large. The transected filaments can be identified as bright spots. The more oval a bright point is, the more aslope the fibres are cut (top of screen) and the more acute the angle between the cutting and winding direction is. The winding layers of different directions are separated by stripes of resin enrichment, which are visible in the picture as dark lines. Each unidirectional winding layer has a thickness of approximately 0.3 mm.

If the effort for running a load cycle test to leakage is too high for a certain design type, a creep rupture test might be the right choice in order to determine the loss of fatigue strength (degradation). This is not just about the evaluation of new test specimens, but also for the determination of the residual fatigue strength of CCs, subsequent to either a part or the whole service life.

2.2.2 Sustained Load Testing of Composite Cylinders

In the creep rupture test, material specimens or components are exposed to a sustained load—in this case, internal pressure. The strength criterion is set to be the time up to the point when a previously defined failure occurs. Within this, failure

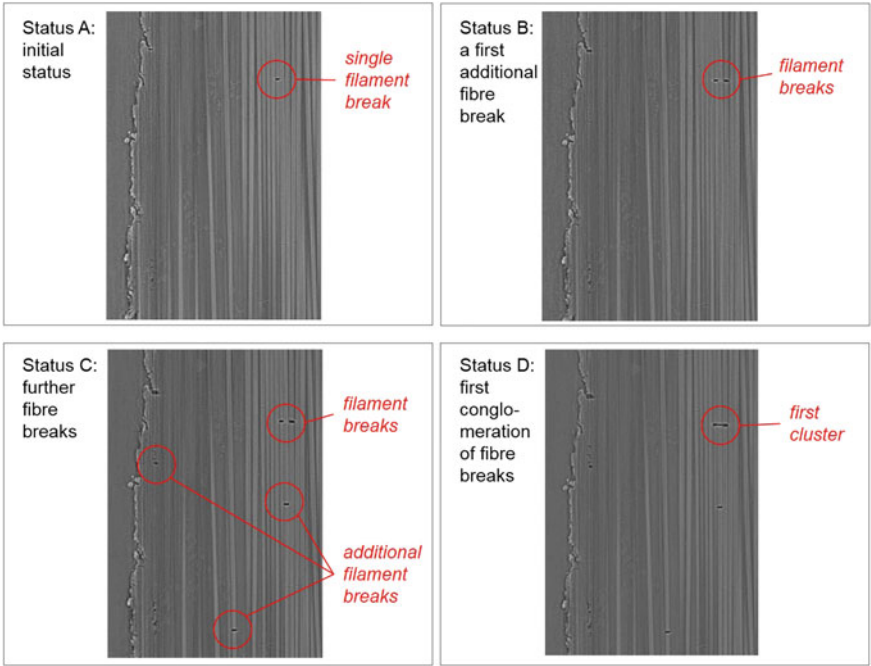


Fig. 2.17 Series of pictures from computed tomography (by courtesy of ROSINI, MAVROGORDATO, SINCLAIR, SPEARING; muVIS X-ray Imaging Centre at the University of Southampton; HELFEN from 19 beamline at ESRF)

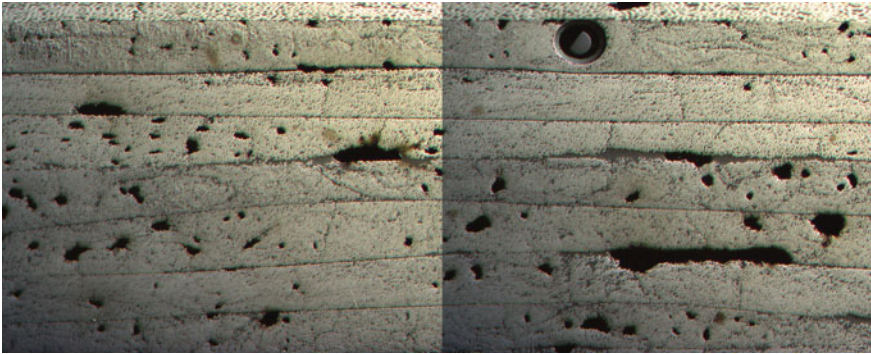


Fig. 2.18 Micrograph of a wound carbon fibre laminate (CFRP) with an integrated optical glass fibre (*top*, to the *right* of the image centre; composed of two separate images)

definition, value of load, kind of loading, test specimen geometry, load introduction, temperature and air humidity, etc. are the main influencing factors on the result. Hence, these parameters are defined as test parameters.

The test is built up of two phases. The first one is the load increase, the second one the holding phase, defined as the period of waiting for failure under sustained load. Usually, the increase phase is so short in relation to the holding phase that the time ratio of the load increase phase can be neglected when viewing the whole test duration “time until rupture”. This is principally based on Fig. 1.7 and shown in Fig. 2.19: while an ideal burst test means a constant pressure rate up to rupture, in case of a creep rupture test the pressure increase phase ends at the desired level of pressure and is ideally kept constant over the desired pressure level. Otherwise, when reaching the failure during the way shown in Fig. 2.16, a pressure drop might occur due to composite creeping, yield effects of the liner or an internal pressure increase might happen caused by rising temperatures.

Bringing the results from the burst test and two different creep rupture tests together based on statistical analysis results in the idealized scheme of Fig. 2.20. Through the three points presenting the three mean values of a sufficiently large number of samples, a straight line can be constructed depending on the scaling of the time axis. This could be supplemented by parallel lines of constant survival probability. Theoretically, the critical time of creep rupture is reached, if the line with the required survival probability crosses the level of maximum internal service pressure. So far, this is the idealized theory.

However, in reality, there is the following conflict: on the one hand, the test duration is intended to be kept as short as possible by maximizing the pressure level. On the other hand, a reliable statistic is needed. This means that the strength of as many of the test sample as possible should be determined. For such kinds of tests where the measured variable is the time up to failure at a defined load or

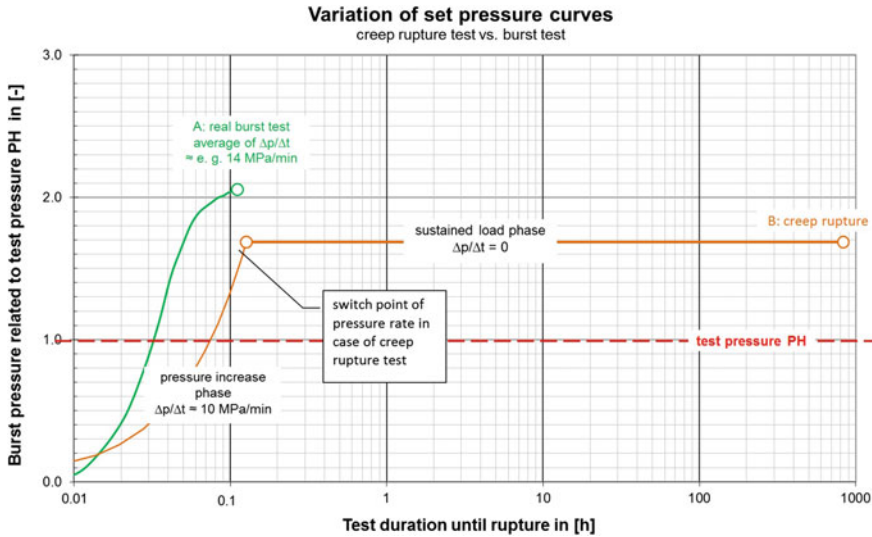


Fig. 2.19 Schematic display of pressure–time curve for burst and creep rupture tests as two different quasi-static tests until rupture

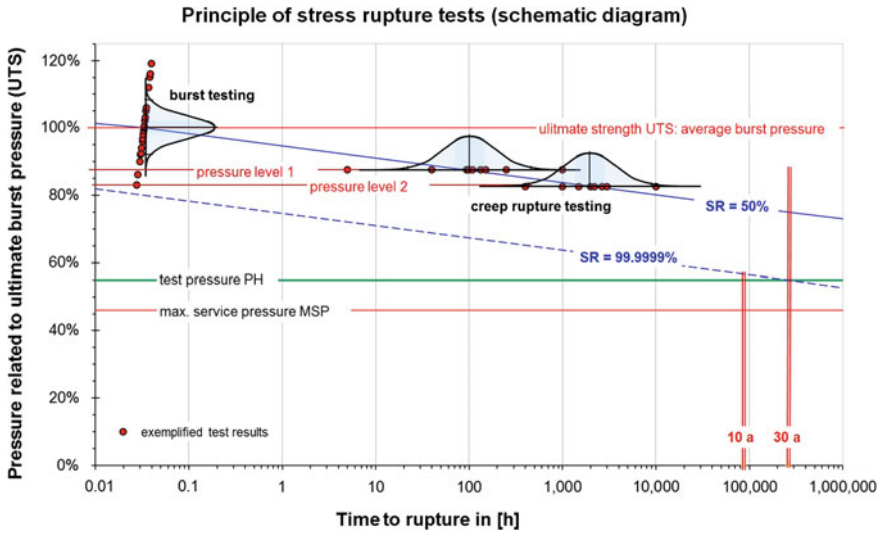


Fig. 2.20 Evaluation scheme of fictitious results of test duration until rupture of three samples; cf. [17]

pressure level, this means that as few as possible of the weakest test specimens should fail before the set pressure is reached. Also, it is not acceptable to abort the test since otherwise the best test specimens would not be considered by the statistics.

For capturing statistically even the so-called early failures within the selected strength criterion “time to failure”, the pressure level is not allowed to be higher than the claimed value of the results of the initial burst test. This means when accepting to lose only one out of 100 test specimens during the phase of pressure increase, the determined load level has to remain below the level which is equal to a failure rate of 1% of the relevant burst strength distribution (survival rate $SR = 99\%$).

Regarding the creep rupture strength of carbon fibre strands, first of all a very small reduction in strength can be observed. As a result according to [19], it is assumed that a carbon fibre strand degrades linearly in the double-logarithmic diagram. More in detail, it means that in seven dimensions of time the strength loses 10% of its initial value on average. Knowing this, the conflict mentioned above is actually impossible to solve. It can be seen as a big problem, even without considering other additional early failures, which occur frequently in the moment when switching from “increasing pressure” to “holding pressure”. This means when doing an initial burst test of 6 min with a load of 95% of the average strength, we would have to wait for more than 100 h (4 dimensions) until just the half of the samples have failed.

If you compare this degradation rate with burst pressure results, such as, e.g., for a CFRP breathing air cylinder of type IV (used in so-called self-contained breathing

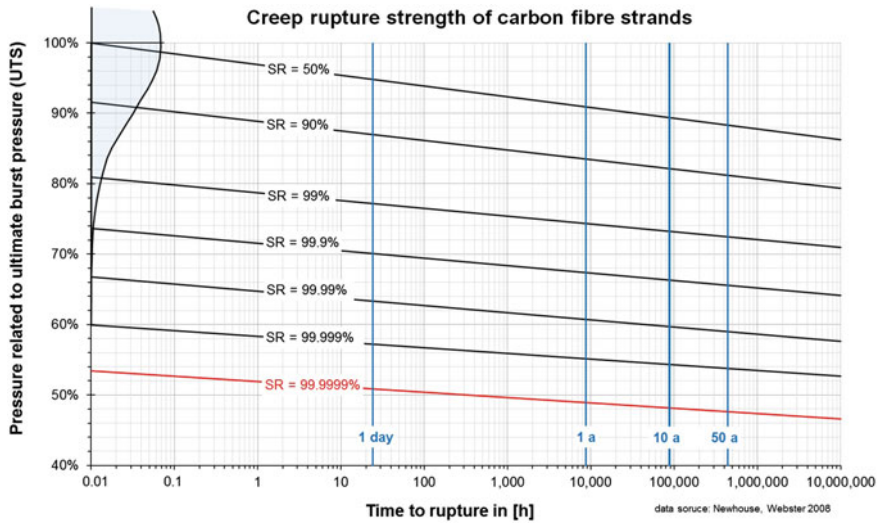


Fig. 2.22 Time -strength-curve from creep rupture tests up to failure, according to [21]

Knowing that 1000 h is a very critical time frame for prototype tests already, the creep rupture test is not applicable for the general determination of strength properties within the frame of prototype testing and production batch testing.

2.3 The Slow Burst Test (SBT)

In order to escape from the dilemma mentioned above, an idea was created to modify the time–pressure curve of static strength tests. As shown below, the procedure for pressure increase has been modified in order to develop a manageable replacement for the creep rupture test.

2.3.1 Development of the Slow Burst Test

While during creep rupture tests the hydraulic pressure was kept constant as much as possible, as an alternative a precisely defined set-pressure curve is driven until leakage occurs. Above the point of changeover “switch point”), this time–pressure curve corresponds to the principle of a burst test. However, it is still the idea that time is the strength value which has to be determined. This means that the pressure, similar to a creep rupture test, has to follow a precise time–pressure curve in order to be used as a significant measured variable. Such a time–pressure curve is

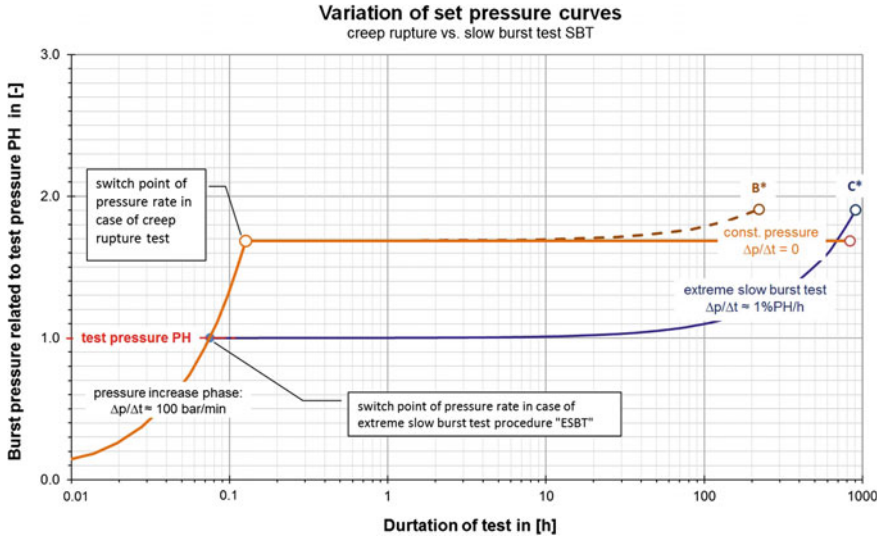


Fig. 2.23 Time–pressure curves of different quasi-static test procedures based on time to rupture

presented in Fig. 2.23 and marked with the letter “B*”. Consequently, “B*” shows no modifications until the switch point is reached (between phase of rapid pressure increase and the “holding phase”).

The specification of the pressure rate stands in contrast to the practice of a burst test. Within the standard specification of a burst tests, a very high maximum pressure rate is defined as limited specification, orientated on the yielding behaviour of metals. Hence, it is obvious that the authors of these standards did not consider a major influence of the test duration on the test result above a minimum test time of one to three minutes.

Due to the fact that the increasing pressure rate above the switch point is higher than zero (curve “B*”), it is not necessary anymore to choose the pressure level for the switch point as high as possible. This has been done before for the limitation of the test duration when performing the creep rupture test (cf. Fig. 2.19; curve “B”). In order to avoid early failures with respect to strength statistics based on time or pressure, it has become possible to shift the switch point to lower pressure even without risking a multiplication of the test duration. For the area of transportation of dangerous goods, the so-called test pressure PH was considered as a good choice ($PH = TP = 150\% \text{ NWP}$), as this value has to be withstand by each CC in any case. This option is shown in Fig. 2.23, marked with “C*”. Since the intended time frame is much longer than the one of a conventional burst test, it has been called “slow burst test”.

Following the idea of the creep rupture testing and other tests, which are already requested in regulations and standards for CC testing, a virtually limited test time frame of up to 1000 h is assumed.

Assuming a moderate pressure increase rate for the lower pressure range (which is suitable even for large design types), a fixed starting pressure rate of, e.g., 10 MPa/min to be run up to PH might be adequate. In this case, the time to reach the switch point is between 3 min (NWP = 20 MPa) for natural gas (methane) and 10 min (NWP = 70 MPa) for hydrogen. When choosing such long overall test durations (1000 h) and using these specifications, the relative duration of the initial pressure increase phase remains short. Consequently, the measuring error of time, which is defined as strength criterion is also very small. This would always be negligible even without having made any differentiation between both phases in case of using identical approaches. For the measuring error in time measurement and thus for the time of the increasing pressure phase, a limited value of approximately 0.1‰ is assumed.

Following this initial idea, a test procedure can be described by two steps: a pressure increase rate of approximately 10 MPa/min up to the switch point at 150% of the work pressure (NWP). The switch point is followed by a phase with an exactly controlled pressure increase rate. Following the idea of a limited proportion of the pressure increase phase of 1‰, minimum test duration of approximately 100 h has to be concluded. In the following, the test method described by curves “C*” in Figs. 2.23 and 2.24 is defined as “extreme slow burst test” (ESBT).

If shorter durations for test periods are needed, the time of the pressure increase phase has to be taken into account and must be defined as an independent

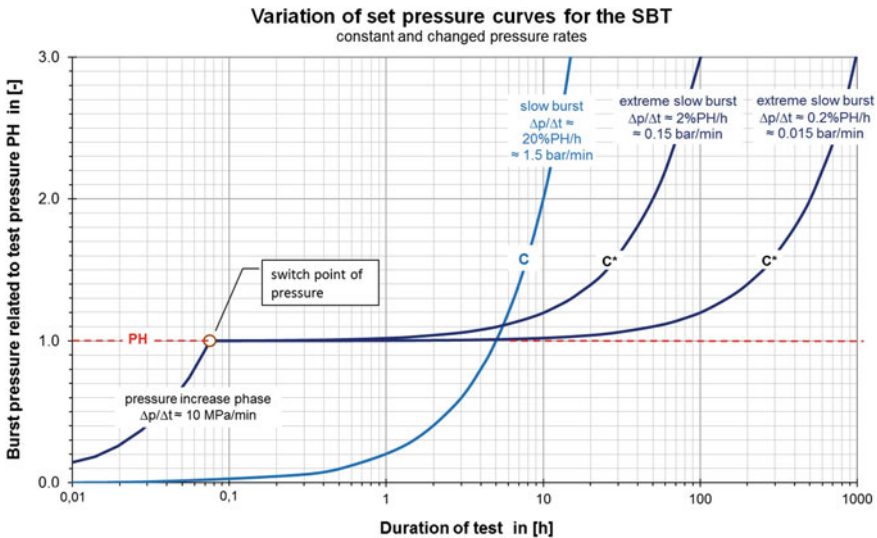


Fig. 2.24 The p – t curves of different variants of applicable slow burst tests

parameter, always. Alternatively, the phase of the constantly slow pressure increase can be set from the beginning onwards, i.e. starting from a very little internal pressure. This comparatively easily controllable variant of the test procedure (marked with “C”) mentioned above is described as “slow burst test” (SBT) in the following. It features a total test time of at least 10 h in case of a minimum strength of 200% PH.

Test duration below 100 h is also available as a variation of the extreme slow burst test when performed in two steps. This means a rapid pressure increase to the switch point combined with a relatively short phase of increasing pressure above the switch point. In this case, the influences on material behaviour (shown in Sect. 2.3) as well as the not negligible time component within the pressure–time correlation have to be taken into account. However, this option will not be considered in the following and will not be subsumed under the term “slow burst test”.

If, within the procedures of the ESBT, the time proportion of the rapidly increased first phase of pressurization up to the switch point is negligible or if a unique time pressure correlation is given as shown in the SBT, the following will apply: each time value can be correlated to a pressure value—and vice versa. For these variants of the burst test, this allows that both the “time to failure” as well as the “pressure at failure” can be interpreted as a characteristic value of strength.

In contrast, creep rupture tests and conventional burst tests usually feature unspecified and test facility-dependent pressure rates in praxis. Accordingly, these test procedures rely on one of the two values of strength characteristics only. Hence, the test procedures SBT and ESBT are derived from a mixture out of creep rupture test and restrictive burst test.

As a next step, it has to be clarified which effect test parameters will have and how test results can be evaluated and certain effects can be interpreted in particular. According to [31, 33, 34] and, e.g., [35] in ultimate strength testing (burst test), there is a connection between the elongation velocity (strain) of a material test piece and its elongation at ultimate tensile strength (strain to rupture). Furthermore, this thematic can be referred to [36], too. According to this, it is assumed that the time–pressure curve of a slow burst test SBT influences the result. If this influence is essential or not depends on many influences, which might affect creep rupture properties, too. Hence, it is initially assumed that an ideally manufactured material test piece and the shell of an equally ideally manufactured CC feature strength properties, which values decrease with increasing test duration. Consequently, the pressure increase rate can be seen as the central parameter of the SBT.

As already shown, it is assumed that a phase of rapid pressure increase will stay without an undue influence on the universality of the interpretation of test results, if several conditions are ensured. These are:

- (a) The rapid increase of pressure is operated until test pressure, which means up to 150% of the NWP.
- (b) During the phase of rapid pressure increase the pressure rate is equal to 10 MPa/min.
- (c) If the duration proportion of the phase of rapid pressure increase is not greater than about 1% of the total test duration (ESBT), the duration of the whole test can be replaced by the failure pressure to be the essential strength value.
- (d) The pressure increase rate of pressure increase has to be controlled very precisely above the changeover point to ensure the reproducibility of results.
- (e) If the total test time is less than 100 h (which means that (item c) is not fulfilled), the slow pressure increase shall be driven from the very beginning (SBT).

Picking up again the aspect of the dependency of strength values from strain rate, it is probably of advantage, if the pressure rate is defined comparably to the strain rate. This means to interpret the strain rate as fibre strain to rupture divided by the test duration. As this is not practicable with respect to CC testing, at least approximations can be made. It seems of less practical use to relate the pressure rate on an initially unknown burst pressure of CC. Due to the current legislation, the CC has to feature a determined fibre specific minimum burst pressures. Since in regulations, the minimum burst strength values are defined as a multiple of the nominal working pressure NWP or of the specified test pressure PH respectively MSP. Accordingly, it is obvious to define the pressure increase rate in analogy to the switch point to be multiple of the test pressure. Since for most of the pressure receptacles, the minimum burst pressure is determined as twice the value of the test pressure PH and as most of the fuel storages for gas (automotive onboard storage) are below (cf. Sect. 5.2.1), it is recommendable to use pressure rates with the double value of the test pressure PH. Consequently, when, e.g., operating a pressure increase rate of 20% PH/h i.e. 30% of the NWP per hour, test duration of around 10 h is assumed.

The associated variability of test durations may make small difference for the direct comparability of different design types. For the comparison of different states of degradation of one design type, this difference should not be of relevance at all.

Summarizing the above, the pressure time sequences (SBT: "C"; ESBT: "C*") shown in Fig. 2.24 are of primary interest. The pressure–time curves displayed in Fig. 2.24 differ significantly from the curve of an unspecified burst test ("A") referring to Fig. 2.14 or the creep rupture test ("B") in Fig. 2.23.

The most important aspects of the current experience made within this test procedure are listed in the following with focus set on the influence of the test parameters.

2.3.2 Experience with the Slow Burst Test

The key point for the examination of the impact of the pressure increase rate or test duration in practice is accuracy. This means the test facility has to govern the

specified set-pressure curve very precisely. For this purpose, an electro-hydraulically driven 3500-bar dual-circuit system was used for the most of the test results shown in the following figures. The volume flow of this facility available at maximum pressure amounts to 3000 ml/min. This concept of a burst facility does not differ much in principle from a displacement controlled dual-circuit load cycle system. The most obvious difference is the second pressure intensifier (i.e. piston) in the secondary circuit, the anticyclical run of both pressure intensifier and their small volumes. The second piston enables continues pressure increase despite the small piston volumes. Since a controlled pressure release is not part of a burst test, it is not useful to provide the necessary amount of fluid by the piston volume of one pressure intensifier, i.e. by one stroke of a piston.

In the case of test durations of more than one hour, the use of hydraulically driven systems is not efficient. Already the energy consumption for maintaining a certain pressure level is very high. With the longer test duration, the required delivery volume flow decreases and consequently so does the physically necessary power for pressure increase. Thus, it is obvious that the performance of slow tests calls for a different type of facility: the electrically driven spindle piston pump. The heart of this system is seen in Fig. 2.25. This piston is designed for a volume of 1000 ml and a maximum pressure of 230 MPa. Due to the step motor control, this drive can be controlled very precisely; its energy consumption is very low.

The measured pressure values of a test with this facility are shown in Fig. 2.26. The pressure curve stands for an idealized and perfect constantly increasing pressure rate. The required increase in fluid volume is not linear and shows two



Fig. 2.25 Spindle-driven piston pump in its frame work

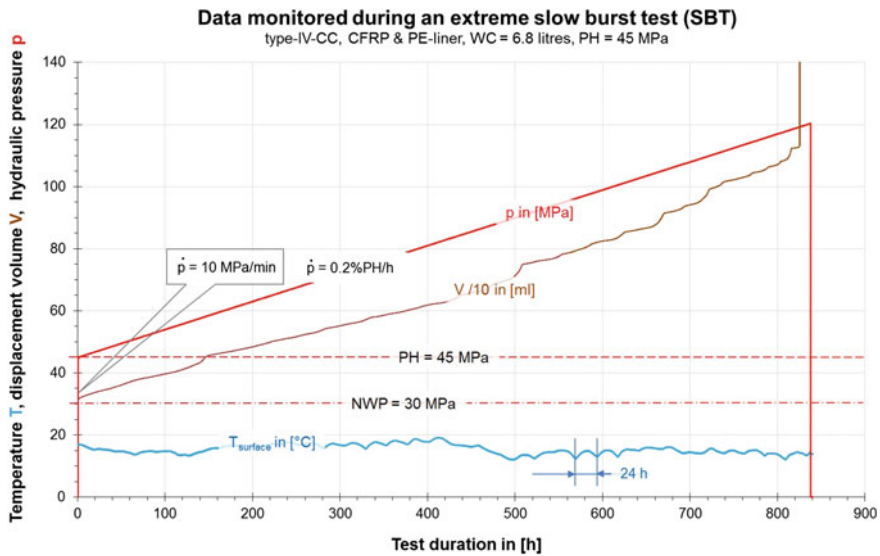


Fig. 2.26 Pressure–time curve that is generated by a spindle piston pump with further data measured during the test

noteworthy effects. On the one hand, there is a general tendency of the volume curve to get steeper at higher pressures. In this case of a type of CC-test specimen with approximately linear strain behaviour up to rupture this can primarily be retraced to the compressibility of the test medium (water with additives). The undulation of this curve gives hints on settlement procedures and possibly procedures of local ruptures. The influence of the temperature expansion particularly above $t = 500$ h is not excluded, even if a direct correlation with the temperature curve is not clearly possible.

The disadvantage of such a single-piston pump is the limited delivery volume. If the tests require more volume than the displacement volume of the piston, the piston must be moved back, which is referred to in the Sect. 2.4.4.

Additionally to the options shown in Figs. 2.14, 2.23 and 2.24, there are a number of other conceivable variants of the time–pressure curves, which are shown and explained in [22–24]. The most important ones of the presented findings can be described as follows:

For example, Fig. 2.27 shows a measurement of residual strength of three different design types with aluminium liner and glass fibre winding (type A to C). Here, determined burst pressures are related to the test pressure PH. Despite the advanced operation process with partially significant traces of use, a relatively high burst ratio of the test specimens can be found. In all cases, the results from the conventional burst test and from the slow burst test are presented in pairs.

does not show any noticeable difference to the new CCs (blue). On the other hand, for an lower pressure rate of 0.1 MPa/min all samples with the same or a lower degree of ageing feature a significant drop (blue arrow) compared to the new sample (slow burst test with 0.1 MPa/min = 20% PH/h; PH = 30 MPa). This behaviour cannot be generalized, but animates to further analyses and tests on the observed phenomenon.

In this way, the described observations are followed as a working hypothesis: there are minimum test durations which must be followed in order to (especially) reveal degradation effects.

In this context, the simulations introduced in [26] were carried out. These analyses are also based on the micro-mechanical approach (see [26–29, 40]) shown in Figs. 2.16 and 2.17. According to this approach and in accordance with [30], strength and cracks are statistically distributed as imperfections of each filament (single fibre). Via matrix material, which is modelled through viscous-elastic-plastic properties, strain peaks are transported into the neighbouring filaments as a time-dependent property. Thus, it comes to a time-dependent rise of fibre breakage even without considering micro-cracks inside the matrix and fibre-matrix-debonding. Additionally to this, a higher-level failure criterion becomes more relevant. It says that a global failure of a single layer occurs with a defined local accumulation of broken filaments (cluster). With a side view to the discussion on the failure of monolithic pressure vessels (type I), the local failure of an individual layer inside the multi-layer network could be seen to be equal to a technical crack in its importance and detectability. In both cases, the micro-level is transferred to the component level. Furthermore, in both cases, strain distribution inside the wall changes in a basically measurable amount.

The mentioned micro-mechanical approach was used in [31] in order to reproduce the design Y. The resulting simulation outcome is shown in Fig. 2.29 in analogy to Fig. 2.28. The differences in the individual results of test with the same pressure rate result from statistically different randomly distributed imperfections in the initial state of the simulation. Hence, it is shown that even in accordance with substantiated analytical models the duration of tests has a discernible influence. This is valid even without having modelled the fibres themselves with a time-dependent strength; which is not generally agreed for carbon fibre.

For determining effects of the test duration, respectively, of the pressure increase rate a comparison of conventional tests (exactly constant pressure increase rates) with slow burst tests was made. The initial 26 burst tests were based on samples of four production batches of one design type, which was made out of HDPE-liner and CFRP (to be called “design D” in the following). The results are shown in Fig. 2.30, based on [33], and supplemented by 27 further test results out of the batch testing.

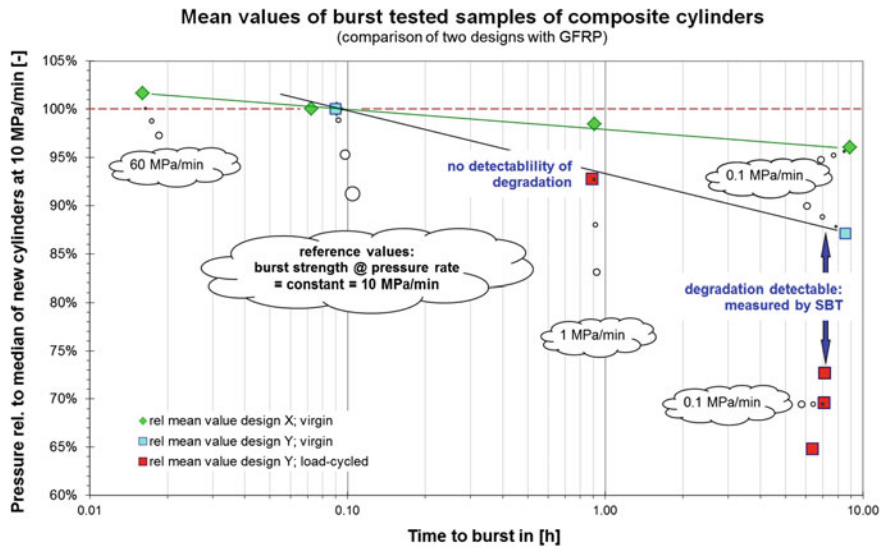


Fig. 2.28 Influence of pressure rate on the burst strength of design type Y and X (cf. [24, 33])

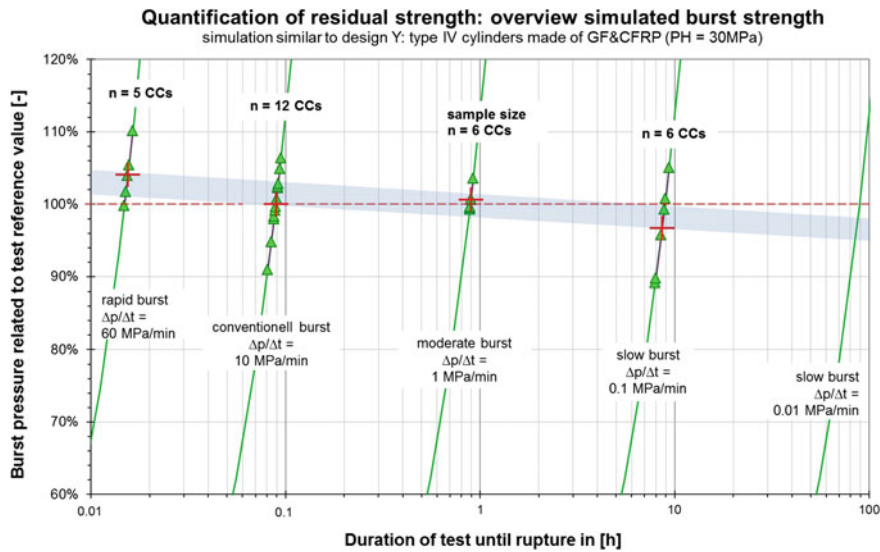


Fig. 2.29 Results of the arithmetical simulation of the influence of the time on the burst strength

It was noticed that the scatter of the results out of the manufacturing batch testing (left in the figure) is relatively large. Also, the latest test specimens out of two batches (blue filled symbols in the right half of the figure) vary considerably. An unusual fact of these results for carbon fibre-reinforced pressure receptacles present

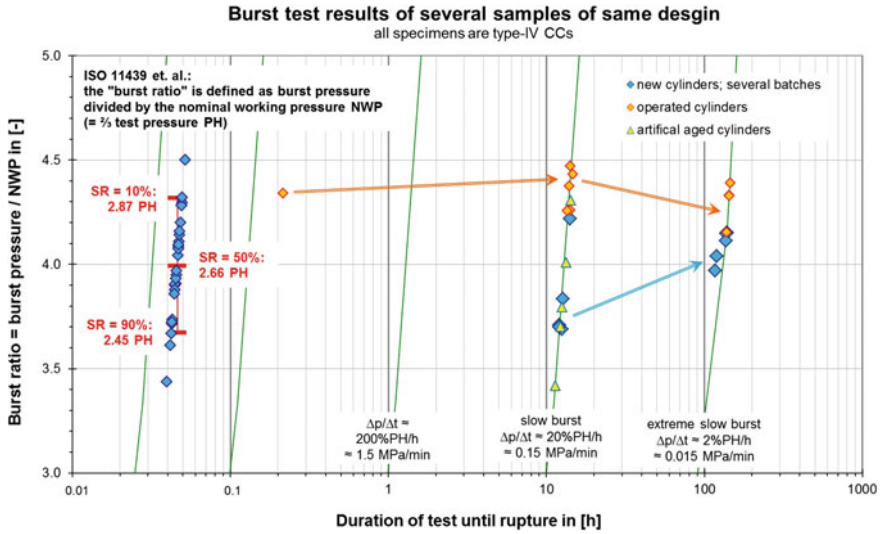


Fig. 2.30 Results from 53 burst tests for design type D: new, artificially aged and taken out of service (cf. [22, 23])

the test results of CCs, which were previously in use (diamonds filled with orange). These results are situated on the upper edge of the strength range. The virgin test specimens out of the manufacturing batch, whose test results are situated below the long-term mean strength of the manufacturer batch without featuring any previous damage (some of the blue diamonds), show an increase of strength when slowing down the test procedure (blue arrow). Due to the large basic scatter, no rapid loss of strength was noticed at a pressure increase rate of 20% PH/h or 2% PH/h like it was the case for GFRP, displayed in Figs. 2.28 and 2.29. In the course of the test campaign, first signs indicated that a sustained load tends to increase the (slow) burst pressure while hydraulic load cycles reduce (slow) burst pressure.

To get a deeper understanding of the effects shown in Fig. 2.30, further tests within this context were necessary. Hence, another 75 CCs of this design type out of two well-mixed and adjacent production batches were destructively tested. The concept for testing these 75 CCs, featuring variations of the kind and intensity of pre-conditioning (pre-damaging in the meaning of artificial ageing) as well as the parameters for the associated burst tests, is comprehensively explained in [22, 23, 31, 41, 42]. For artificial ageing the test specimens have experienced in total more than 2.5 million load cycles and about 20,000 h of sustained loading.

First of all, via systematic variation an evaluation of different artificial ageing procedures could be created as described in the literature mentioned above. The ranking displayed in Fig. 2.31 results from an analysis based on mean values and standard deviations. Here, the basic equations for the calculation of the probability of survival (survival rate SR) according to the GAUSSian normal distribution are used. However, for the ranking of the different levels of degradation caused by

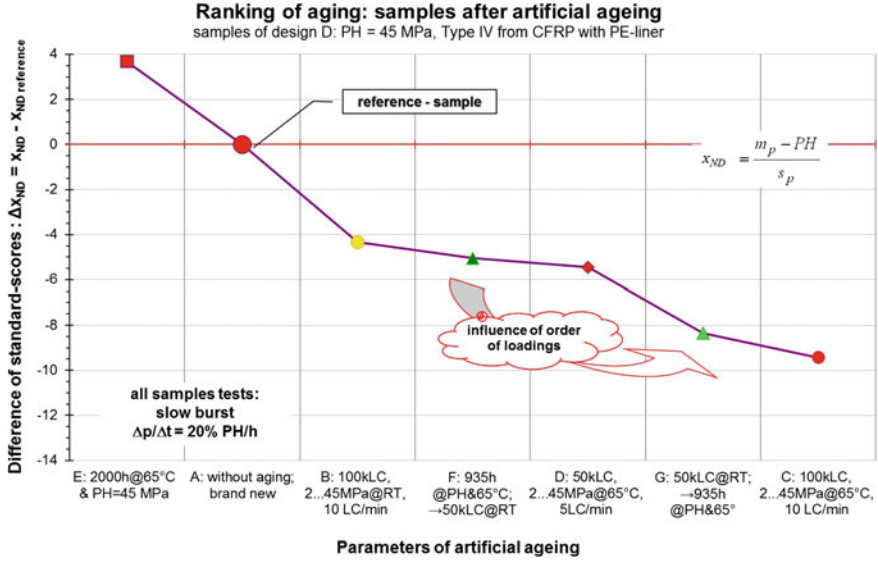


Fig. 2.31 Ranking of pre-damaging processes according to the standard-score x (cf. [23, 24])

artificial ageing, the quantification of survival rate SR can be dispensed with. As a criterion, the standard-score (standard-deviation) in accordance with Eq. 2.1 is sufficient.

The scatter or the standard-score x , as considered in Fig. 2.31, is defined to be the difference between mean value of burst pressure m_p (strength) and test pressure PH (maximum load) divided by the corresponding standard deviation of burst pressure s_p of a random sample. In the case of a GAUSSian normal distribution (ND), the probability of survival SR for a supposed example can be written as follows:

$$\text{Risk} \sim SR \approx x_{ND} = \frac{m_p - PH}{s_p} \quad (2.1)$$

Equation 2.1 is the general valid formula for the calculation of the standard-score x , as necessary for the normalization of a GAUSSian normal distribution (ND). The difference between load and loading capacity is related to the standard scatter. For each of the samples, a value of standard-score x_{ND} is calculated. In standard manuals of mathematics (e.g. [43]) and each calculation programme, it is commonly used for the calculation of the failure rates or survival rates, based on ND. Within the scope of this chapter, values will be compared only without discussing the absolute reliability. For this reason, it is not necessary to get to know the distribution function in detail. For a comparative evaluation, it is sufficient to use the standard-score x_{ND} as qualitative placeholder for the reliability as it is defined in Eq. 2.1.

Based on this, Fig. 2.31 shows the ranking of the effect of the pre-conditioning procedures on the degradation of the samples. The value of deviation x_{ND} is

calculated for each sample. The type of damage of these samples is represented by a letter (column in the diagram) in Fig. 2.31.

In Fig. 2.31, Δx_{ND} indicates the difference between the standard-score of the relevant differently pre-conditioned samples (artificial ageing) and the value of standard-score of the reference sample of new CCs with $x_{ND} = 12.5$. The more this difference gets negative, the greater is the loss of survival rate SR, i.e. loss of the residual service strength. All samples were tested with a pressure rate of 20% PH/h until rupture occurred (cf. burst procedure “C” in Fig. 2.24). The display in comparison to the reference sample is used in order to compare the intensity of degradation. The following effects can be found as supplement to the list of Sect. 2.3.1:

- (f) A pure cycle fatigue pre-conditioning of 100,000 LCs at RT (column B) leads to a significant loss of survival probability SR.
- (g) A comparison of two samples which have performed for the same duration under a cyclic load at 65 °C shows: the loss of residual burst strength of the sample (column C), which was pre-conditioned at 65 °C via 100,000 LS, is larger than the strength loss of sample (D), which was loaded 50,000 times at 65 °C with a doubled cycle duration (half load cycle speed). Thus, both tests took the same time.
- (h) The probability of survival SR decreases with increasing temperature during a cyclic inner pressure load (LC), starting with $A \rightarrow B \rightarrow D \rightarrow C$.
- (i) The sample (E), consisting of CCs, which were preloaded for 2000 h under sustained load at the test pressure (PH) and 65 °C, show a higher SR than the reference sample without pre-conditioning.
- (j) The sample (F), being pre-conditioned at PH and 65 °C for 935 h, followed by 50,000 LCs at RT shows a higher residual probability of survival as the sample (G), which CCS were pre-conditioned equally but in reverse order.
- (k) Despite the moderate level of sustained load pre-conditioning, sample (F) does not show any improvements compared to sample (D); despite, improvements between sample (E) and (D) can be observed. However, it seems that the cyclic loading subsequent to a static pre-conditioning (sample F) is less critical than in reverse order (sample G).

The intention behind Fig. 2.32 is to work out the one pressure increase rate of the quasi-static burst test, which is most suitable to differentiate the degree of damage and of degradation. Based on the phenomenon displayed in Fig. 2.28 it was expected to find minimum test duration or maximum pressure rate, which can show a clearer decrease of strength to the degraded samples as a burst test with its usually short test durations can do.

For this purpose, mean values of new (not pre-conditioned) samples from different burst procedures are compared in Fig. 2.32 with results out of the load cycle pre-conditionings of 50,000 and 100,000 LC at RT and 65 °C. The mean values of the samples (normally 5 test samples) are displayed versus the duration of the test

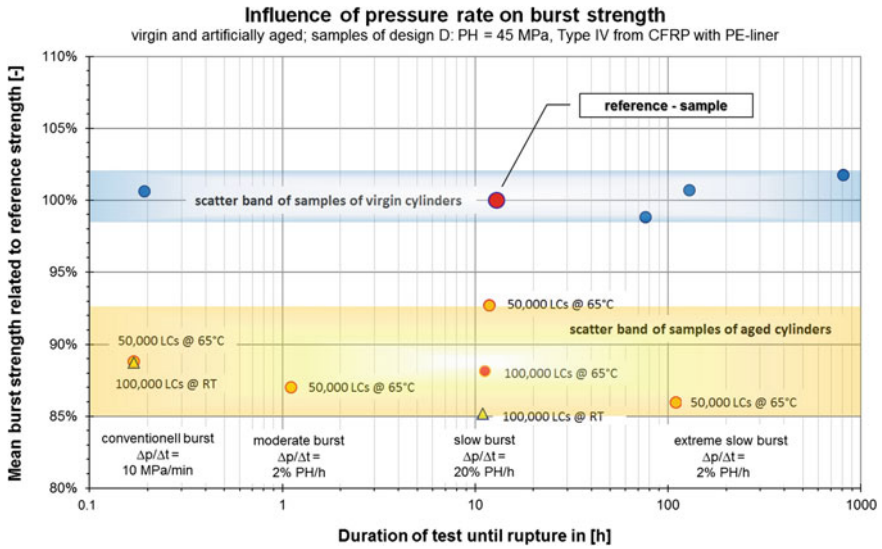


Fig. 2.32 Mean values of burst strength of samples versus burst test duration

period. The mean value of each sample is normalized to the mean value of reference sample which is already used in Fig. 2.31 (7 CCs; new; pressure rate = 20% PH/h).

In the burst test, all samples that were not pre-conditioned (blue band = new) showed a just marginal deviation from the reference sample. This variation could be attributed to the scattering which occurs randomly when several small samples are taken out of a large basic population (see Sect. 3.4.1). Therefore the width of the upper (blue) band could be limited by increasing the samples size. As this was not possible in course of the test campaign it remains a hypothesis.

The test results of artificially aged samples out of CCs scatter in an area marked through the lower band (orange) in Fig. 2.32. It covers the residual strength after 50,000 LC at 65 °C and those ones after 100,000 LCs at RT and 65 °C. There are no clearly visible trends of a dependency on test duration—with one exception: as already shown in Fig. 2.28. There, the samples with a pre-conditioning at 65 °C feature in the slow burst test (SBT; 20%PH/h) a higher average strength than those ones which have been loaded at room temperature. Furthermore, a tendency in comparison of both samples loaded with 50,000 LCs at 65 °C could be noticed. With increasing test duration, the average strength values tend to decrease. Overall, primarily the above-mentioned fact is valid: due to the small sample sizes these supposedly discernible trends of the mean values could be explained by statistics; i.e. they are not considered to be significant.

In the end, these observations argue against an exclusive consideration of the mean values for the measurement of degradation.

However, as a working hypothesis it will still be assumed that the differences in burst strength between new and artificially aged samples are principally an indicator for the degree of degradation.

Figure 2.33 illustrates the influence of the test duration, respectively, the pressure rate, on the standard-score x_{ND} of burst strength according to Eq. 2.1. Thus, the difference in x -score Δx_{ND} is used as a scale for degradation. It shows the trend of the standard-score Δx_{ND} in the shape of two lines. The difference between the two x -scores Δx_{ND} is introduced in Fig. 2.31. It cannot be obtained directly but read as a difference between the respective pair of samples or the relevant lines. Each pair was tested with the same pressure increase rate. The new samples consist of 5–7 CCs. The pre-conditioned samples consist of a number of 5 CCs; as well as of 3 CCs in one case of artificial ageing.

The results of the artificially aged samples are interpolated by the dashed line (blue) while the solid line (red) interpolates the properties of the new samples. Due to the low scatter of the artificially aged samples out of the (common) rapid burst test (left in Fig. 2.33), it comes to a remarkable effect: the line fitting the highly damaged samples crosses the line of the new samples. Thus, it seems that pre-conditioned samples in case of rapid burst testing perform better than the new samples. As samples subsequent to sustained load pre-conditioning (Fig. 2.31; sample E) show increased strength, at least for special cases this might be plausible. This applies in particular in cases of burst values according to moderate pre-conditioning are higher than those ones of new CCs. In the case of Fig. 2.33, the degree of pre-damage is not moderate any more. Also, other test durations,

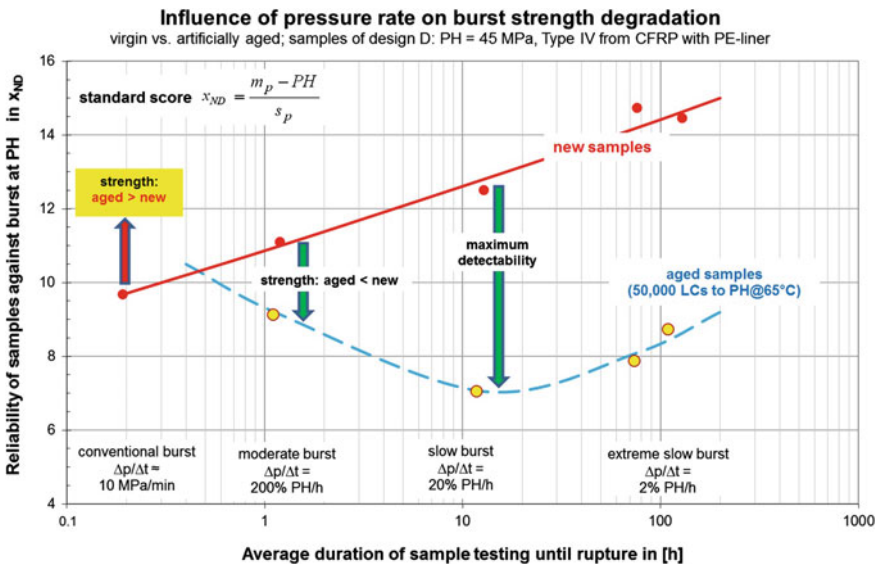


Fig. 2.33 Influence of increasing test duration on the value of standard-score x_{ND}

which stand closer to the real operating loads show a significant decrease of strength. Hence, this observation is likely to be interpreted as a special effect and, at least, doubts the results of the usual burst test.

The distance between the lines of the new test samples (continuous line; red) and the aged test samples (dashed, blue) increase from left to right. It increases until the difference for test duration of 10 h reaches its maximum and seems to continue at an equal distance with increasing test duration. A statement concerning test durations longer than 100 h is not made here. Test duration of approximately 10 h is reached for most test specimens by a burst pressure of the doubled test pressure featuring an increasing pressure rate of approximately 20% PH/h.

As conclusion according to this observation, a burst test with constantly increasing pressure rate of 20% PH/h is recommended instead of using conventional test procedures.

In case of significantly low burst pressure, a further reduction of pressure rate should be considered, e.g. 20% MSP/h or less.

With this intensity, up to now a systematic analysis for an optimal test velocity could be investigated on only one design type. Hence, the result cannot be considered to be generally valid. However, the possibility of false assessment displayed in Fig. 2.33 prohibits the use of a conventional burst test in order to evaluate degradation. This is supported by the evidence of Fig. 2.28 (both design type IV).

A pressure rate of 20% PH/h or less allows an optimal gain of knowledge and offers a maximum differentiation of strength properties. Such a long time under pressure also corresponds better to the operational loads than a rapid burst test. A gas filled CC has to hold the operating pressure for a very long time. One filling cycle until refill (filling, holding pressure and releasing the gas again) usually counts between 10 h (vehicles) and 3 months (e.g. medical oxygene).

The following three working hypotheses are used in conjunction with [30, 44] as feasible explanations for the partially surprising phenomena. However, at this point it is neither possible nor intended to prove them according to general validity:

- Temperature might have a positive homogenizing effect. This works through an accelerating effect to the creep mechanism (viscos-elastic relaxation). It could finally result in minimal dislocations of highly strained filaments (single fibres) faster than at RT and hence other fibres and fibre layers would be strained.
- Interlacing might occur which would increase the strength of the matrix material.
- The micro-mechanical failure process under a rapid load increase might be different from the one which dominates the degradation in operation; i.e. under sustained loads.

This means that in case of competing failure mechanisms, which potentially result in failure at slow test velocity, a higher scatter is visible than when applying a

high-pressure rate. It is assumed that rapid testing provokes less or other failure mechanisms.

Hence, it is principally necessary to check which phenomena are to be expected and which ones have to be evaluated for certain design types (e.g. type IV; i.e. fully-wrapped CC without a load-sharing liner). Because of these uncertainties and the many possible influences, it is generally recommended to apply a pressure increase rate of not more than 20%PH/h. This is especially valid in all cases of lack of the appropriate design type-specific analysis, which has been mentioned above.

After the recommendation had been made for the design type of the breathing air CCs (type D; introduced above; for “self-contained breathing apparatus” SCBA) to test with a maximum of 20% PH per hour, this specific pressure rate was applied for the analysis of further questions, too. In this way, three samples out of 5 test specimens from one design type E were tested with the same constant pressure rates subsequent to artificial ageing. In order to evaluate the influence of ageing through gas cycles (hydrogen cycles) compared to hydraulic cycles, three samples were differently pre-conditioned as follows: artificial ageing through 1000 hydraulic load cycles at RT, artificial ageing through 1000 hydraulic load cycles at 85 °C and artificial ageing through 1000 gas cycles at (nominal) ambient temperature. The comparison of these results is shown in Fig. 2.34.

To enhance comparability, strengths values in Fig. 2.34 are related to the average strength value of the sample hydraulically cycled at RT. Unfortunately, comparable values without pre-conditioning are not available.

Thus, it gets obvious that the scatterings, which are shown here for the first time, could give informative basis for the intensity of an ageing process and for reliability after ageing. This is underlined by the differences in interpretation between

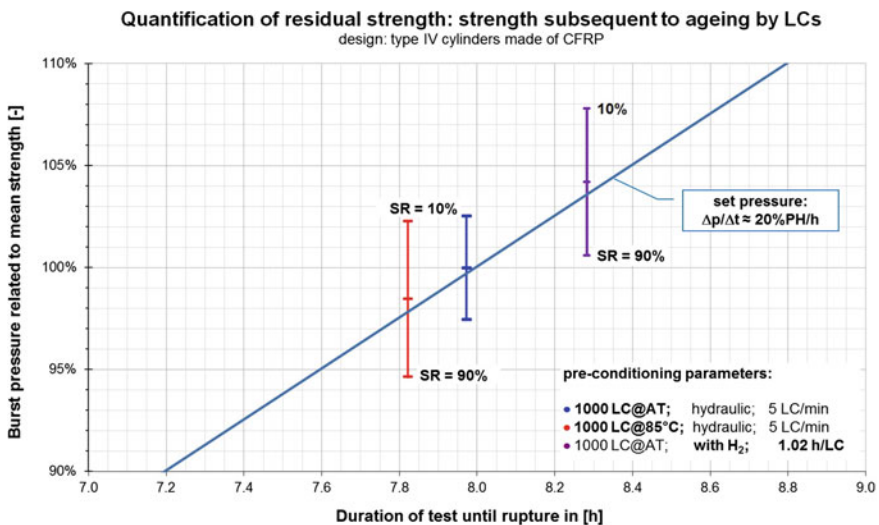


Fig. 2.34 Strength values with SR = 10, 50 and 90% of three differently aged samples of CCs

Fig. 2.32 (pure mean value analysis) and Fig. 2.33 (observation of mean and scatter according to Eq. 2.1) and leads to the sample performance charts (SPC), which are developed in the following Chap. 3.

2.4 Detailed Recommendations for a SBT-Procedure

Since the slow burst test, (SBT: pressure rate $\dot{p} \leq 20\%$ PH/h respectively 20% MSP/h) is especially designed in order to determine residual strength for the purpose of subsequent statistical analysis, the question of reproducibility is of extraordinary high importance. As explained in the Sect. 2.1, the statistical evaluation requires test procedures which feature a small scatter caused through fluctuating test parameters. This creates the need for a detailed and restrictive description of test procedures and to make a lot of effort for recording of relevant data. For this reason, in the following some practically well-experienced requirements are shown, which provide a significantly higher degree of detail than usual.

2.4.1 Sampling of Test Specimens (Composite Cylinders)

All test procedures must be applied to a number of test specimens (i.e. CCs), which provides a sufficient level of confidence in the respective test result (see Sect. 3.4.2). All CCs of a sample must belong to dedicated design type and have represented a narrowly determined range of age and load history.

For the investigation of various degradation procedures or for scientific investigation of fundamental influences on the behaviour of the CCs, all test specimens of all the relevant samples should be taken out of one identical production batch, as far as possible.

Test specimens, which have been exposed to a load of more than 20% of the test pressure, should not be defined as “new” or “unloaded” anymore. This is valid subsequent to the mandatory initial proof testing.

2.4.2 Test Parameters

The following listed details should be collected and recorded during testing for the purpose of statistical analysis in order to identify and describe the test procedure and the test specimens:

Date of test and test engineer (inspector); manufacturer; construction/cylinder type and designation of design type/variant; (nominal) working pressure (NWP or PW), maximum allowable working pressure (MAWP) or maximum service pressure (MSP); test pressure (PH or TP); material of fibre and liner; serial or

identification number of each test specimen; date of manufacture and/or the batch number of the test specimen; if appropriate, detailed description of the applied ageing procedures and the intention of the artificial ageing of each test specimen; if applicable, details of the previous usage (type of gas, intensity of usage, number of fillings etc.; as far as available) and other service conditions (e.g. country of use/climate zones) of each test specimen.

Parameters which must be monitored or recorded during the test are the internal hydraulic pressure of the test specimen. Alternatively, the fluid pressure can be measured at the pressure connector/fitting port or inside the hydraulic pipe as close as possible to the test specimens' valve thread. The measuring and recording frequency should not be less than one measurement per second.

For retrospective evaluation of unexpected effects the following data should be recorded during each test/handling: temperature of the test environment and the CC surface at the beginning and at the end of each test; relative humidity during CC storing and inside the test chamber (at least before launching a test); used pressure fluid/test medium, used pressure sensors including the class of accuracy and all relevant further devices of the measuring chain; information on the pressure generator (functional principle of the generator, settings at the supplying device such as set-pressure rate; if necessary settings of holding phases and pressure levels etc.); each relevant special feature before, during and after the test according to the test specimen, the testing and measurement technique; maximum pressure reached in connection with the type of failure, in particular if leakage or bursting occurs (leak before burst; LBB)

2.4.3 Test Procedures

The test procedures must be executed as identically as possible for each test specimen of the dedicated sample and for all other samples bound for direct comparison of test results. For the analysis of the absolute survival rate/value of reliability, further efforts addressing quantitative uniformity of the test parameters should be made. This also includes inter-laboratory tests on the neutral comparison of resulting test performance

During each test, the following boundary conditions have to be complied with: temperatures in the test medium, of the test specimen and in the test chamber should be at 23 ± 5 °C. If only a wider range of temperature was realized this would have been noted. At the beginning of each test, the humidity inside the test chamber should be between 30 and 70% relative humidity. Each test specimen shall be completely filled and be free of air. The compatibility of the test medium with the liner material and other pressurized equipment shall be confirmed. The temperature of the pressure medium (i.e. in the media) should be similar to that of the test sample and the ambience. On the one hand, this helps to avoid unintended temperature fluctuation and, on the other hand, it helps to avoid control problems during a slow pressure increase, e.g., due to medium expansion caused by

temperature fluctuation. The pressure during an initial tightness test should not exceed 20% of the test pressure (approximately 10% of burst pressure).

All test samples have to be processed with a constant increase rate of the internal pressure until rupture featuring a rate of $\Delta\Omega = p/\Delta t = 20\% \text{ PH}$ (or MSP) per hour (cf. [37]). The initial pressure for a uniform increase of pressure with $\dot{p}$ should be set to nominal zero MPa. Practically, an initial pressure of 5% PH should be accepted.

If, in the case of a particularly slow pressure generation, the increasing pressure rate should occur with $\Delta\Omega = p/\Delta t \leq 2\% \text{ PH/h}$. In this case, the starting point of the slow increase phase can be set up to the test pressure PH without expected influence on the test result (cf. Fig. 2.24). For a comparable evaluation of test duration to rupture it must be ensured that the duration of the first rapid stage of increasing pressure will not exceed 1% of the total test time until rupture/ leakage.

The start-up and alignment phase of many pressure generators, which are associated with extraordinary pressure fluctuations, should be brought to a termination below a pressure of 20% of the test pressure PH. After the end of the stabilization phase, the deviation of the actual pressure from the set pressure increase should not be bigger than:

$$\Delta p \leq 1.0\% \text{ PH} \quad (2.2)$$

This so described permissible scatter band is highlighted in Fig. 2.35. Compliance with this requirement guarantees reproducibility and comparability of test results; i.e. the band of tolerance is less than 0.5% of the burst pressure in most cases. For an example of a CC with a NWP of 70 MPa and a test pressure PH of 105 MPa this means an acceptable value of deviation of 1.05 MPa.

A leakage due to an insufficient sealing of the connection thread must not be defined as leakage in the framework of the burst test. It is recommended to continue the test after re- sealing the thread. In the case of an interruption of the test procedure, the following should be noted: if an interruption occurs during the testing of new test specimens above 20% of the test pressure PH, a continuation of the examination or a new test with the relevant test specimen is not recommended. For test specimens taken out of service the recommended limit is two-thirds of the test pressure PH, which is significantly higher than for new ones. In cases of higher pressures at time of interrupting a test, the test specimen has to be discarded and replaced by an additional one which meets the defined properties of the sample.

If a continuous increase of pressure in accordance with Fig. 2.35 is not possible, the pressure can also be increased in steps. This can be an effective equivalent test as long as the following guideline is regarded.

2.4.4 SBT-Procedure with Stepwise Pressure Increase

Very precisely controlled test equipment often operates with one or two alternating pistons as pressure amplifiers. In these cases the high-pressure pump does not

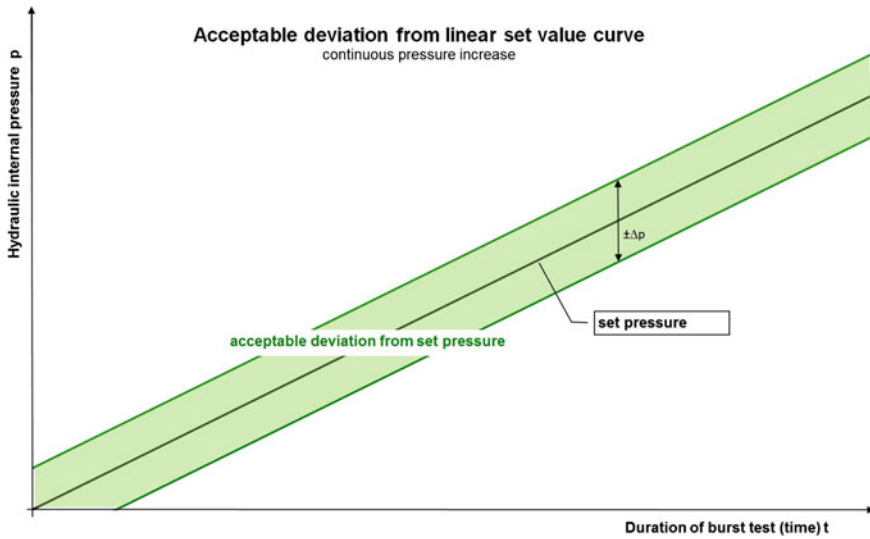


Fig. 2.35 Acceptable band of tolerance for the deviation from the ideal target pressure curve

directly affect the test specimen. In particular for such equipment, it is usually not possible to perform the test as shown above. In these cases the extracted volume per piston stroke is limited and thus the attainable pressure increase is limited, too. Usually, the volume is not big enough to realize the required volume which is necessary to build up the pressure for a rupture with one stroke. In these cases, it is usually necessary to shut off the piston at its idle stroke end against the sample and to drive it back without applying any pressure. In this way, further strokes can be repeated until the burst pressure is reached. For this reason, a procedure is of interest that realizes a gradual pressurizing, too. When creating steps in this procedure pressure holding phases in each step must be ensured, which provides the piston retraction to the initial position and consequently the adaption to the pressure inside the test specimen, again. For this case, a valve technology has to be used which prevents a pressure reduction in the test specimen and piping when opening and closing this valve placed between piston and test specimen.

In case of a stepwise pressure increase, each step consists of two phases: the phase of a more or less rapid pressure increase (ramp) around pressure difference Δp_s and the holding phase of constant pressure with its duration Δt_h . At the end of each step, the ideal curve according to Eq. 2.2 and Fig. 2.35 has to be achieved. Therefore, the pressure increase during the ramp of a step has to be driven faster by a certain amount compared to the Sect. 2.4.3. The relevant amount of velocity is determined by the time, which is needed to drive back the piston during the pressure holding phase and to reach in the piston again the current pressure level in the test specimen. As far as technically possible all pressure steps should be identical. In the case of the stepwise pressure increase the acceptable deviation of

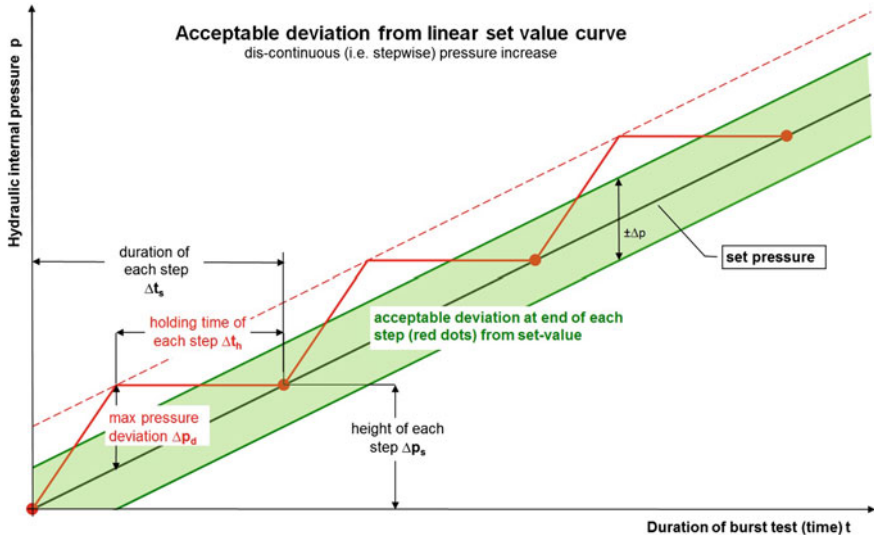


Fig. 2.36 Range of the pressure tolerance towards the ideal pressure set curve; including the details for the stepwise pressure increase

the pressure at the end of each step has to be controlled (points $(\Delta t_s; \Delta p_s)$ in Fig. 2.36).

The height of each pressure level Δp_s depends on the number of steps, which is not limited. Regarding the influence on the test result, a division in less than 100 steps is not recommended. In practice, the number of steps is based on the measured pressure increase per step as well as on the cyclically resulting deviation from the ideal time pressure set curve. Assuming at least 100 steps and the maximum cyclic deviation Δp_d , at the end of each pressure ramp, the following applies:

$$\Delta p_d \leq 2.0\% \text{ PH} \quad (2.3)$$

According to Δp_d and the ideal set-pressure rate $\dot{p}$, the holding pressure phase Δt_h of each step can be derived:

$$t_h = \frac{\Delta p_d}{\dot{p}} t_{h\max} (\dot{p} = 20\% \text{ PH/h}) = \frac{2\% \text{ PH}}{20\% \text{ PH/h}} = 6 \text{ min} \quad (2.4)$$

This can be explained by the following example: $\dot{p} = p/\Delta t = 20\% \text{ PH/h}$, $\text{PH} = 105 \text{ MPa}$; $\dot{p} = p/\Delta t = 0.35 \text{ MPa/min}$; $\Delta t_s = 135 \text{ s}$; $\Delta p_s = 0.79 \text{ MPa}$; $t_h = 90 \text{ s}$; $\Delta p_d = 0.5\% \text{ PH} = 0.53 \text{ MPa}$. This example means a test duration of 5 h until the test pressure PH is reached and allows a maximum holding time of 360 s per pressure step.

When investigating the measured pressure curve, the measuring faults and calibration fluctuations have to be taken into account. This means that pressure

transmitters of class 0.2 will be sufficient if the measuring range of the pressure transmitter is less than twice the test pressure and this low value is sufficient. For other cases, an accuracy class¹ of 0.1 is needed and recommended in general.

Conclusion from the discussion of hydraulic test procedures

For consideration of statistical properties, the descriptions of the used destructive test methods have to become more detailed within the relevant standards. This is necessary to ensure reproducibility of test procedures according to the required level. The accuracy in details of the test procedures has to be improved in order to limit the measured scatter to the properties of the tested pieces in one sample and of different tested samples. Otherwise, the scatter of test results is probably dominated by an insufficient reproducibility. In every case, the test procedure for load cycle tests has to be run until first failure occurs in order to allow statistical analyses at all.

It is not manageable to check the service strength by performing load cycle tests for a high number of design types. Nor it is practicable to cover the time under load as an aspect of service strength by performing the creep rupture test until failure. For this reason, the slow burst test (SBT) has been developed as a practicable substitute for the creep rupture test. Finally, this test seems to be a good alternative for the load cycle test whenever high cycle fatigue strength of the design type, which needs to be assessed, is too high to perform the load cycle test until failure.

The validity of the conventional burst test might be doubted. It is neither appropriate for the assessment of service strength nor for the degradation caused by service. Therefore, for CC-test specimen which has been in service already it is recommended to perform the slow burst test whenever a safety inspection against rupture is essential.

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¹Specifies the limit of the deviation (accuracy) in % of the measuring range.

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