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TWO COMPLEMENTARY TESTS TO DETERMINE THE QUALITY OF PE 100-RC

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ABSTRACT
The Strain Hardening (SH) test and the Point Load Test (PLT) are two complementary tests designed to determine the quality of PE 100-RC. The SH test is a resin test, whereas the PLT is a pipe test.
The SH test is a tensile test performed at 80 °C. Extensive research on the SH test has been conducted and published in the proceedings of previous International Plastic Pipe Conferences. The latest Round Robin, performed by seven different laboratories, resulted in similar outcomes with a very low variation coefficient. Experiments have shown that, due to the preparation by compression moulding of the test specimens, the SH test is a resin test and gives no information about pipe quality. Experiments in which specimens are prepared directly from pipes should give insight into whether the SH test can also be used to test the remaining pipe material quality. Future research will focus on checking the robustness of the SH test method. The minimum SH values for PE 100-RC resins must be agreed upon for the purposes of standardisation.
In the PLT, a pipe is placed under static internal pressure in a water bath at 80°C. A detergent is circulated inside the pipe while a point load is applied to the outer pipe surface. By changing the detergent from Dehyton to Arkopal results in reduced testing times. Multiple cracking is observed in the PLT. This is due to the high stresses necessary in order to speed up the failure process. Lowering the internal pressure of pipes in practice makes them less prone to failure due to point loading. This has practical implications for old pipelines. Further research will focus on standardising the test parameters and obtaining the same results between three international labs. A threshold failure time for PE 100-RC pipes should eventually be found.

INTRODUCTION

The mechanical quality of polyethylene (PE) resins and pipes has improved tremendously over the last 50 years. While the first generation of PE pipes were PE 50, the MRS value of PE types quickly increased to PE 63, PE 80 and now PE 100 (third generation), one of the most common PE types deployed in distribution systems.

Not only has the MRS value increased, but the resistance to slow crack growth (SCG) has also increased substantially. SCG is one of the most important failure mechanisms for installed PE gas and water pipelines. Hydrostatic tests show that brittle failures can no longer be expected within 50 years for third generation PE materials, provided that installation is carried out properly. In 2002, PE 100-RC reached the market. This material does not have a higher MRS value but does have a better SCG resistance. This makes it possible, for example, to lay the pipes without a sand bed. This reduces installation costs.

The improved SCG resistance of PE 100-RC creates problems for the current short-term quality tests. Testing times of more than a year for PE 100-RC materials are not exceptional. These long-term testing times are not only in themselves unacceptable, but also give rise to numerous other problems. For example, in the Pennsylvanian Notch Test (PENT [1]), materials no longer develop brittle fractures [2] and the detergent Arkopal used for the Cone test [3] and Full Notched Creep Test (FNCT [4])degrades [5].
In addition, there is criticism of the often-used FNCT due to the suboptimal sample shape (square instead of round), giving rise to stress singularities [6]. Furthermore, the multiple Round Robins show large scatter between the different laboratories [7], especially for long testing times.
There is therefore a need for new, quick and relevant tests. These have been found in the Strain Hardening (SH) test [8],used to determine the resin quality, and the Point Load Test (PLT), used to determine the pipe quality when under point loading.

RESIN TEST – STRAIN HARDENING TEST

SCG failure may occur when the applied stress is much lower than the yield stress. On a macro scale the fracture appears brittle but microscopically many fibrils are visible on the fracture plane [9]. Disentanglement of tie molecules in these fibrils will determine the resistance of the PE material to SCG [10,11,12].

The Strain Hardening (SH) test developed by SABIC (the Netherlands), published by Kurelec et al [13], McCarthy et al [14], Havermans et al [15,16] and Deblieck et al [17], probes the disentanglement capability of the tie molecules. This is an intrinsic material property. The strain hardening modulus value allows discrimination between materials.

Figure 1. Six SH samples of 0.3 mm thick.

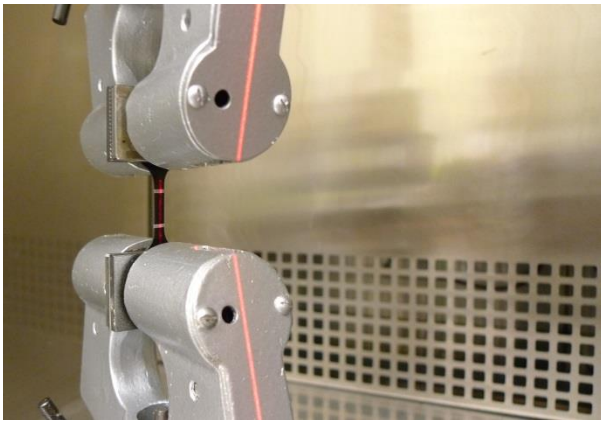


Figure 2. A SH sample ready for testing using a laser extensometer.

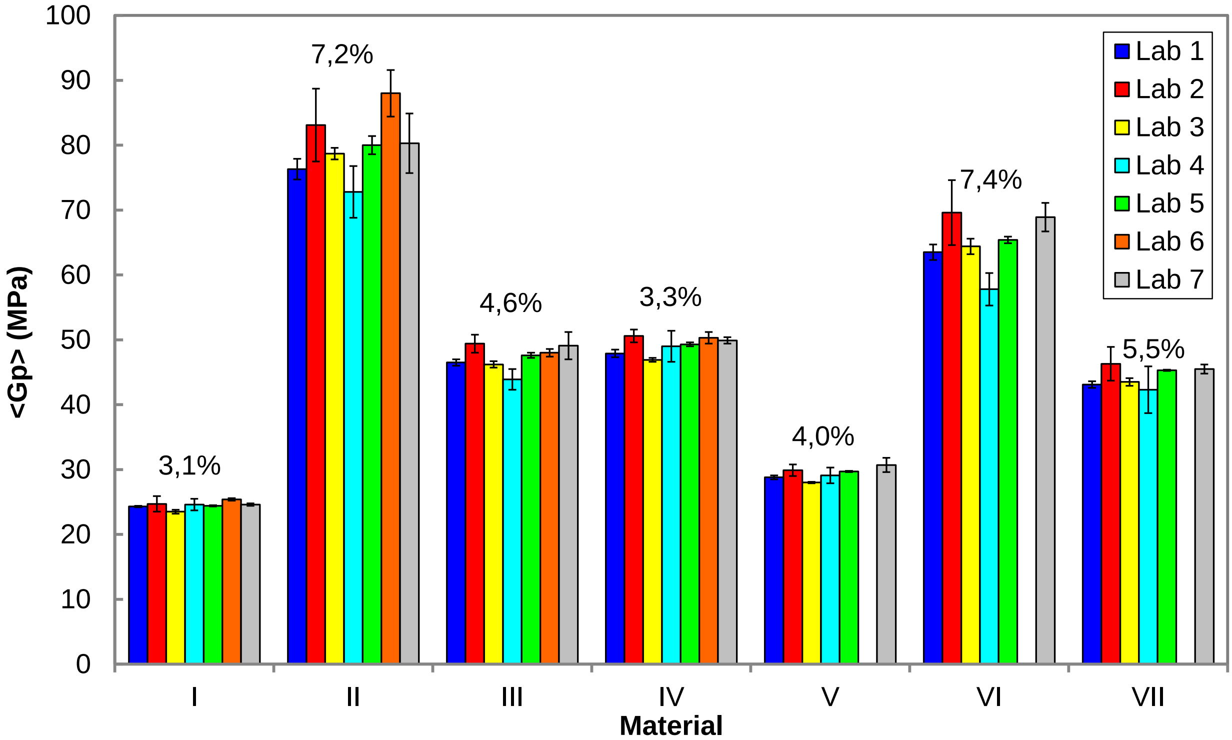


Figure 3. Results of a Round Robin test performed by seven laboratories on seven PE materials in which each laboratory prepared its own 1 mm thick samples. The coefficient of variation (the relative standard deviation) based upon all individual samples is given above the histogram of each material.

The SH test is a modified tensile test performed at 80 °C (176 °F) on specially prepared thin samples (Figure 1). The 0.3 to 1.0 mm (0.0118 to 0.0394 inch) thick samples are punched from a compression moulded sheet made from PE granules pressed in accordance with ISO 1872-2 [18]. The samples are clamped and pulled at 20 mm/min while the elongation is carefully measured. A non-contact (optical or laser) extensometer (Figure 2) must be used because the test is performed at 80 °C []. The displacement of the cross head cannot be used.
A subsequent data treatment using the Neo-Hookean Strain Measure (NHSM) model gives the slope after the natural draw ratio. This slope is correlated to the strain hardening modulus (<Gp>) [,].

The good performance of the SH test in discriminating between different PE types and the low interlaboratory scatter has been published previously []. A new Round Robin was set up in which each laboratory prepared its own samples of 1 mm thickness (instead of 0.3 mm) of each material. The results are given in Figure 3.
Once again, very little scatter is found between the laboratories. The coefficient of variation is calculated based on all individual samples rather than the average of each laboratory. While this gives a higher coefficient of variation, this never exceeds 10 %.

Since the temperature used for compression moulding is 180 °C, the granules in fact become molten. The hypothesis is that this completely removes the thermal (and thus mechanical) history of the sample. This would mean that the SH test, as currently described, cannot be used in order to determine the quality of the pipe material, but only the original resin quality. The following test is executed in order to test this hypothesis.

Material and Method

To simulate mechanical degradation in practice, three segments from the same first generation PE pipe (coded “13-149”) are hydrostatically pressurised to 3 MPa at 80 °C. The segments are stressed for 125, 250 or 500 hours. For the purposes of comparison, one segment is not subjected to the hydrostatic pressure test (0 hours).

Samples for the PENT and SH tests are taken from the middle of the pipe segment, as far as possible from the ends of the pipe. The PENT samples are notched as prescribed in ISO 16241 [] and tested at 80 °C at a stress of 1.5 MPa (instead of 2.4 MPa []). The SH samples are prepared in accordance with ISO/DIS 18488 [] as described above.

Results and Discussion

Figure 4 shows that the PENT failure times (left axis on a logarithmic scale, light blue) decrease when the hydrostatic loading time increases. This indicates that the PENT is sensitive to the previously imposed mechanical degradation. The PENT is however not usable for PE with a high resistance to SCG due to the increased chance of ductile failure as described in the introduction. The SH test on the other hand can also be used on PE 100-RC.

The strain hardening modulus (<Gp>, right axis, red) is independent of the imposed mechanical degradation; the value remains more or less equal. The hypothesis made earlier therefore appears to be correct: The SH test does not measure the pipe quality when the samples are prepared by compression moulding.

The hypothesis is examined further by comparing the results of the PENT and the SH tests for three additional first generation pipes (PE 50) from the field. The pipes are from different batches and installation years. No data is available regarding their mechanical quality before they were installed. The three pipes are:

- A pipe that was (wrongfully) used at a pressure of 8 bar(g) for about 40 years and that has thus endured considerable mechanical loading (material “12-191”)
- A pipe with a very short PENT failure time: 0.5 hours using a stress of 1.5 MPa (material “09-098”)
- A pipe with a (for first generation PE) long PENT failure time: 8.4 hours using a stress of 1.5 MPa (material “12-137”)

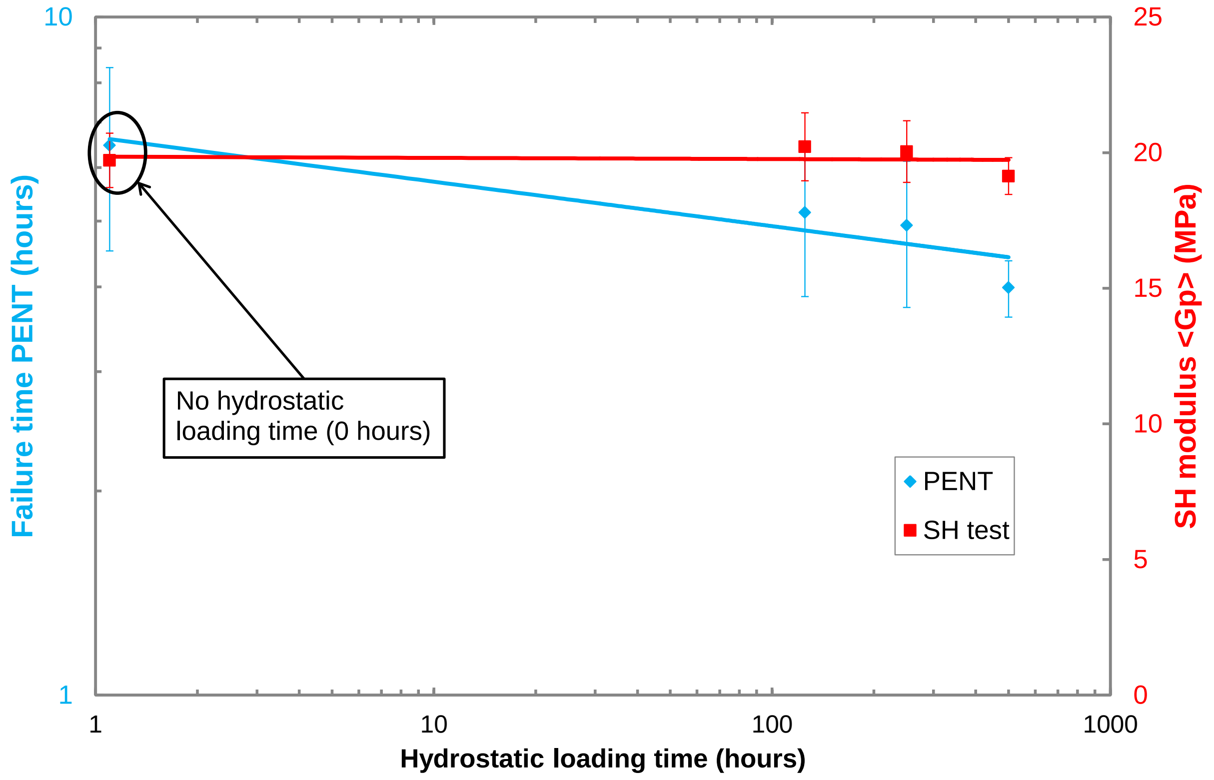


Figure 4. The failure time for the PENT (left axis on logarithmic scale, light blue) and the strain hardening modulus of the SH test (<Gp>, right axis, red). The hydrostatic loading time is shown on the horizontal axis (logarithmic). There is no "0" on a logarithmic scale; the value without hydrostatic loading is thus placed at 1.1 hours for clarity.

The four measured data points of Figure 4 are shown again in Figure 5 on the left side. This is material "13-149". The data points of the three materials as mentioned above are shown on the right side. The graph shows that pipe 09-098, with a short PENT failure time, also has a low strain hardening modulus (<Gp>). Pipe 12-137 has a longer PENT failure time and also a higher <Gp>. The pipe with a large mechanical loading in practice (12-191) has a somewhat shorter PENT failure time, although the difference is not substantial. The strain hardening modulus is also lower, but again only slightly. It is therefore unclear to what extent the PENT results are due to the resin quality and to what extent they are due to mechanical degradation during the lifetime of the pipe.

The results show that there is currently a certain correlation between the PENT and the SH test (Figure 6). Based on other correlations [], this is expected to be linear. It should however be noted that phenomena that are potentially different (resin quality and residual pipe quality) are being compared.

Future research will focus on the preparation of SH samples directly from pipes (without compression moulding). This may increase the suitability of the SH test for testing pipe quality. This would mean that test results on pipes would become available within a matter of days. A distinction between PE pipes, and not only resins, could also be made. On the other hand, only a small sample from the pipe would be considered. To test the quality of the entire pipe, including its residual stresses and pipe geometry, a pipe test is needed. The Point Load Test (PLT) was therefore developed.

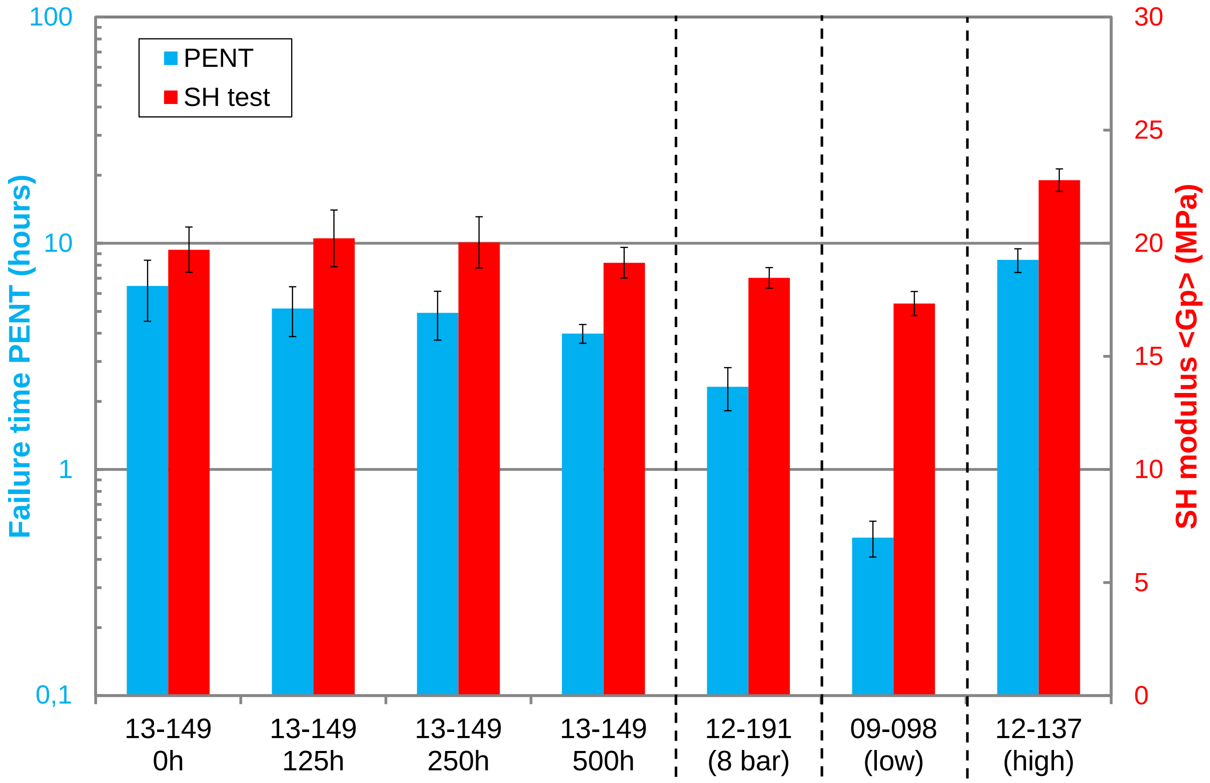


Figure 5. The failure time of the PENT (left axis on logarithmic scale, light blue) and the strain hardening modulus of the SH test (<Gp>, right axis, red).

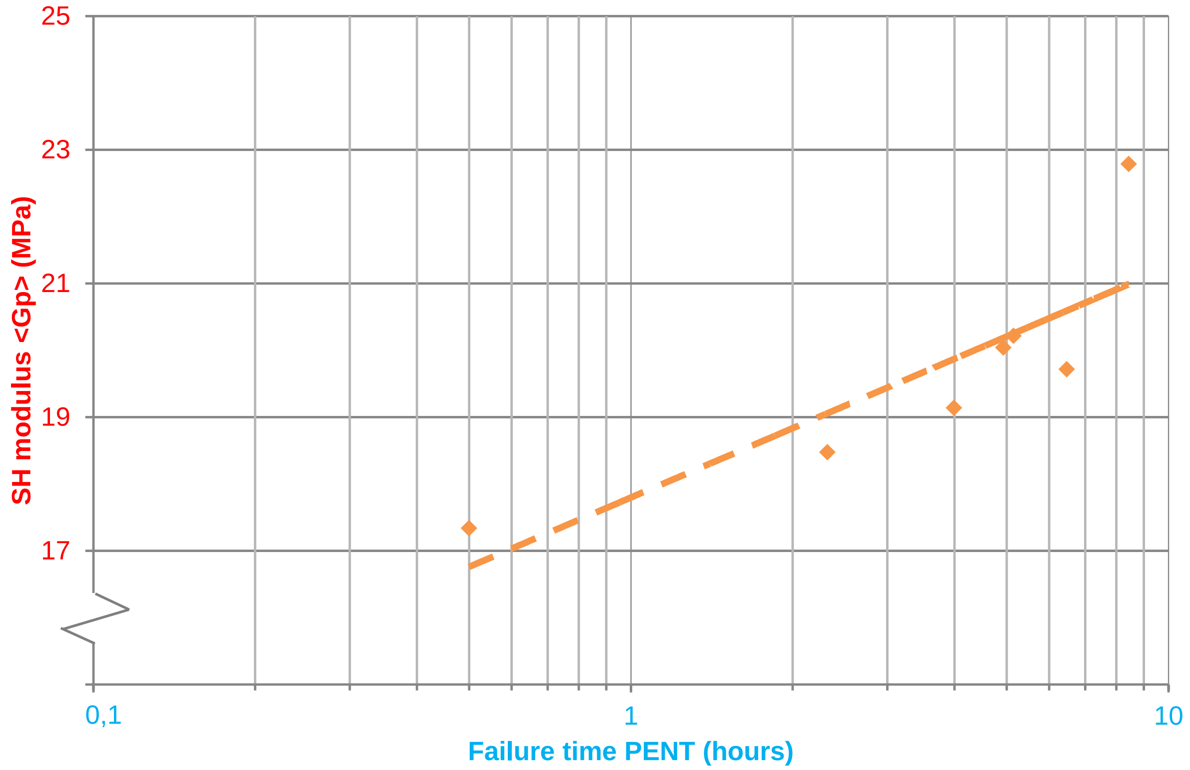


Figure 6. Correlation between the SH test (<Gp>, vertical axis, red) and the PENT (horizontal axis logarithmic, light blue).

PIPE TEST – POINT LOAD TEST

PE 100-RC has been specially developed for (trenchless) pipe laying without sand beds. If a stone presses into a PE pipe, then a dent will be formed on the outside of the pipe. A bulge will in turn emerge on the inside of the pipe. This gives rise to tensile stresses on the inner side of the pipe wall. These tensile stresses are additional to the existing tensile stresses caused by the internal pressure of the medium. A premature brittle fracture at the bulge that would not occur in the absence of the point load will eventually emerge in axial direction.

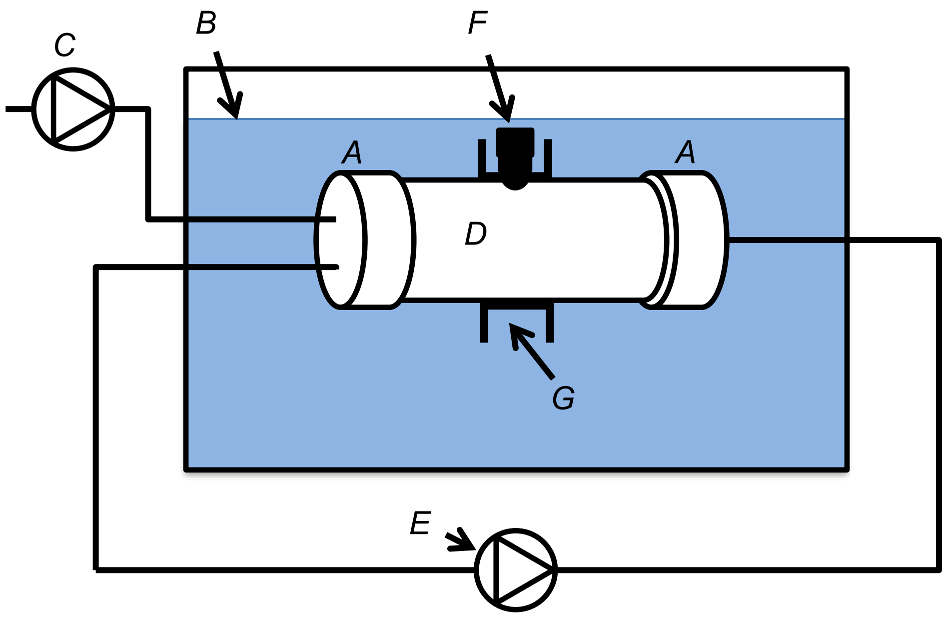
The Point Load Test (PLT) has been developed in order to simulate the resistance to external point loadings. The test is described incomplete and imprecise in the PAS 1075 [19], which is the subject of many discussions. Three international laboratories are therefore redeveloping the test in order to create an open test method.

Current Test Method

Figure 7 shows the test setup for the application of an external point load and hydrostatic internal pressure schematically, including circulation of the detergent solution inside the test specimen (pipe section). The water in the test container (outside the test specimen) is also circulated.
Photographs of the test setup are given in Figure 8 and Figure 9.

The point loading tool (Figure 9 and *F* in Figure 7) introduces a displacement in the radial direction from the external pipe surface towards the pipe centre. The displacement is such that the outer fibres of the interior pipe surface exceed the strain at yield point of the material. During the entire test the loading tool is kept in a constant position, giving a constant deformation. The point loading tool has a hemispherical tip combined with a cylindrical stem. The radius of the tip is 5 mm.
A bending force would be introduced when the point load is applied to the pipe. To prevent bending of the pipe, a support is used (Figure 9 and *G* in Figure 7). The horizontal part of the support is 20 mm wide. The angle of each side is 30°. A Ø110 mm pipe is supported only on the two tilted surfaces and not at the horizontal part.

The pipe is subjected to a constant hydrostatic internal pressure of 4 MPa (*C* in Figure 7) simultaneously to the application of the point loading, at a defined temperature.
A detergent solution is introduced inside the pipe as hydraulic fluid. This detergent is circulated during conditioning of the test pieces and during the test itself when the test piece is under pressure (*E* in Figure 7). The flow rate of the detergent is 1.5 l/min to 1.7 l/min. Stir equipment is used inside the test piece in other test setups.
During the test the pipe remains immersed in water at the stipulated constant temperature (*B* in Figure 7).



Key:

A End caps (Type A)
B Test container filled with hot water
C Pressure generators for pressurising the test piece
D Test piece
E Pump for circulating the detergent inside the test piece
F Point loading tool
G Support

Figure 7. Test setup for the application of the external point load and the hydrostatic internal pressure.

Figure 8. *Execution of the PLT. The PE pipe sample is hydrostatically tested with detergent inside and a point load on the external surface.*

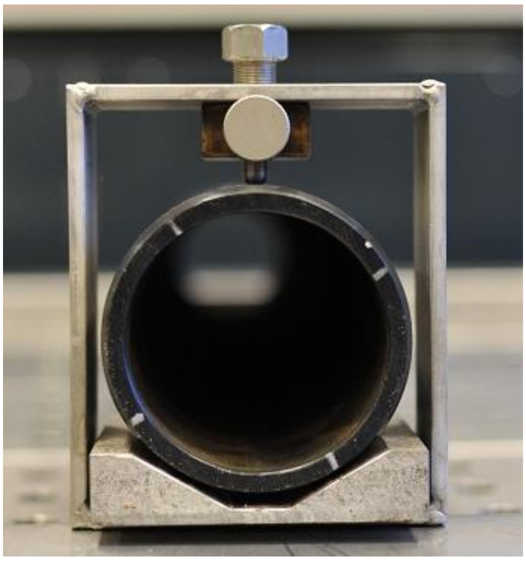


Figure 9. The point loading tool at the top and the support at the bottom. A Ø110 mm PE pipe is placed in between.

Detergent residues from preceding tests in the surrounding water bath are considered to be insignificant, since the tensile stresses (and thus the starting point for cracks and subsequent failure) are highest on the inner wall of the pipe.

Requirements for PE 100-RC

A minimum testing time in the PLT for PE 100-RC is sought as a starting point in order to guarantee a level of quality for polyethylene piping where the useful service life is to be at least 100 years when a point load is present. Using Arrhenius, an activation energy of at least 66.1 kJ/mole is needed to obtain an acceleration factor of 100 when increasing the temperature from 20 °C to 80 °C [20, 21]. However, by taking a conservative approach, a 1 year test is needed where the detergent Arkopal covers the safety factor (or design coefficient).

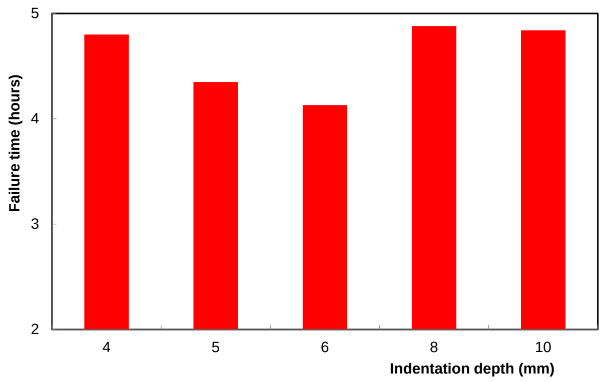
Arkopal is however not stable []. Moreover, there is a need to decrease the testing time to much less than 1 year, if possible to around 1000 hours. Temperature, type of detergent and internal pressure determine the time to failure. This means that a more aggressive (and stable) detergent is needed. The test temperature may also need to be increased. The internal pressure cannot be increased as ductile failure may then occur.

The results hereafter are those of the tests intended to determine the correct testing parameters for an open test method for the PLT.

Results and Discussion

Initial tests were carried out using the detergent “Disponil® LDBS 25” (also known as “Maranil A55”) at 80 °C. For first generation PE pipes (PE 50) the average time to failure is 4.7 hours. The indentation depth of the point load, which varies between 4 and 10 mm, appears to have no significant influence on the result (Figure 10). This is because at 10 mm pipe wall thickness, even 4 mm indentation exceeds the strain at yield of the material.

A comparison between a first, second and third generation PE pipe was made by using the same detergent “Disponil LDBS 25” at 80 °C and an indentation depth of 8 mm (Figure 11). This comparison shows that it is possible to differentiate between pipes of various PE grades. The failure time for PE 100 is nevertheless still much too long. Further optimisation of the test conditions, like increasing the test temperature and using an even more stable detergent, should make it possible to test pipes made from PE 100-RC with far shorter testing times than has been possible so far.



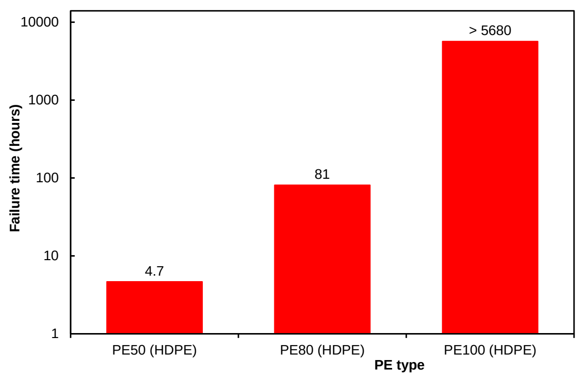


Figure 10. Time to failure for various indentation depths of the point load for first generation PE grades (PE 50) at 80 °C in Disponil. The average failure time is 4.7 hours.

Figure 11. Times to failure in point load test for various PE grades. The test was performed at 80 °C in Disponil.

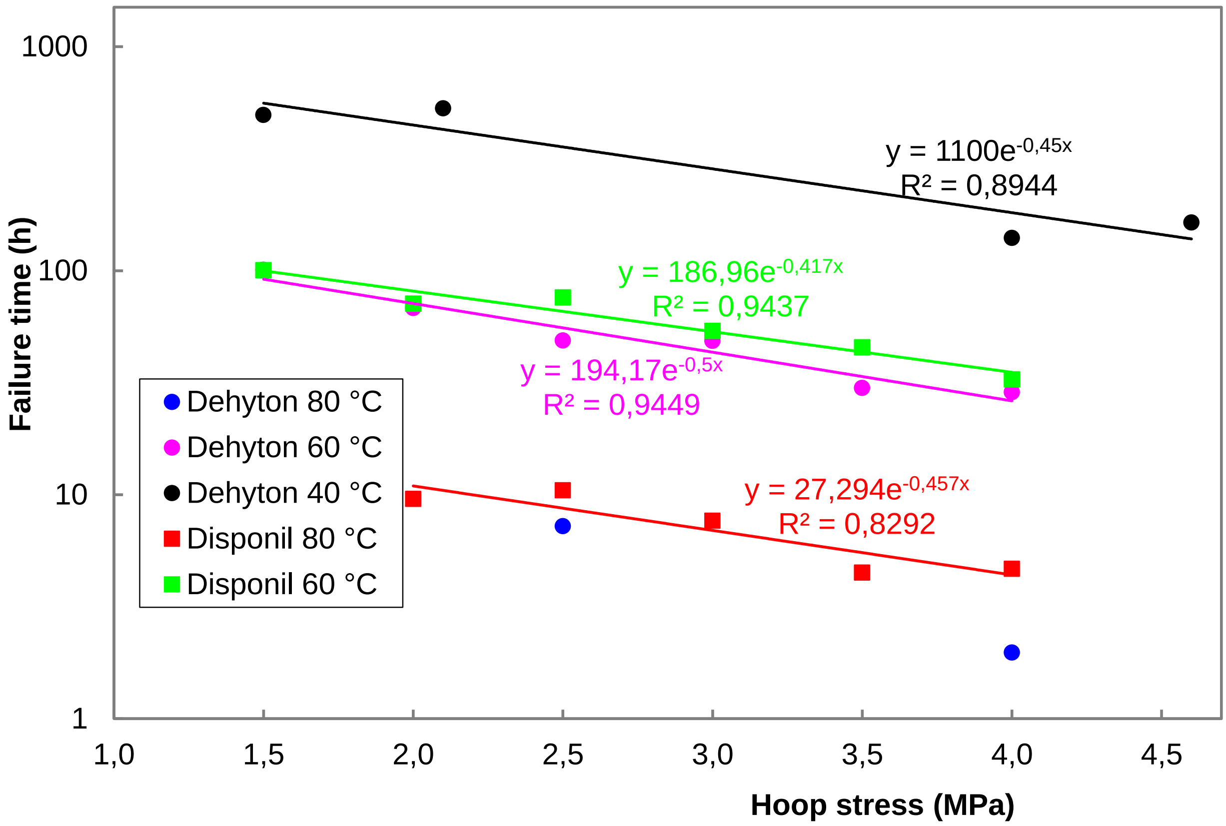


Figure 12. Comparison between the detergents Dehyton and Disponil at different hoop stresses and temperatures, using PE 50 pipes and an indentation depth of 8 mm.

One of the test parameters is, as mentioned before, the detergent type. “Dehyton® PL” is known to be more aggressive than Arkopal and is known to be similar to Disponil. Dehyton is however even more stable than Disponil. This is in turn more stable than Arkopal, which is known to degrade during tests []. Figure 12 shows that the failure time of Dehyton for PE 50 (penetration depth 8 mm) is indeed similar to Disponil for short-term tests.

In this test the hoop stress was varied via the water pressure between 1.5 MPa and 4.6 MPa. It is clear from Figure 12 that a decrease in the hoop stress increases the failure time. For practice this means that decreasing the operating pressure of the medium in an old PE50 pipeline at locations where point loadings are known or expected has a positive effect on the life expectancy of the pipeline. Lowering the pressure by a factor 2 (e.g. from 4 MPa to 2 MPa), increases the life of the pipe by about 2.5 to 3 times.

The activation energy of the PLT using Dehyton and an 8 mm indentation depth can be determined, since the failure times are known for different temperatures. Depending on the hoop stress, the activation energy is between 80 and 110 kJ/mole. This is in accordance with data from Brown [22] who found 85 – 95 kJ/mole for the PENT. This means that the PLT has a range of activation energies that would be expected for a slow crack growth process.
Consequently, a further increase from 80 °C to 95 °C will decrease the failure time by a factor 3 to 4, which would mean 3 to 4 months of testing instead of 1 year.

To gain more insight into the failure process, the failures were investigated visually. Figure 13 shows a representative sample after the PLT using Dehyton at 80 °C and 8 mm indentation. The photograph shows the inner wall with the bulge due to the indentation in the centre. Yielding occurs at the centre, making the PE stronger due to molecular orientation. Cracks will therefore start just next to the centre of the indentation where no yielding occurs. This means in a circle around the centre. This is exactly where the cracks (multiple cracking) start in the PLT (see Figure 13). The cracks are brittle at the macro scale. Figure 14 shows an optical micrograph of the fracture surface with the typical phenomena associated with slow crack growth.
Scanning Electron Microscopy (SEM) will be performed at a later stage.

In practice only a single axial crack is observed. The multiple cracking therefore represents a clear difference between testing and practice. Multiple cracking occurs when the stress is very high. The stress in the PLT is indeed very high, but this is necessary in order to accelerate the failure process. This means that the test could either be improved in order to better simulate practice, or could be improved in order to speed up the test, but probably not both.

Pipe axis

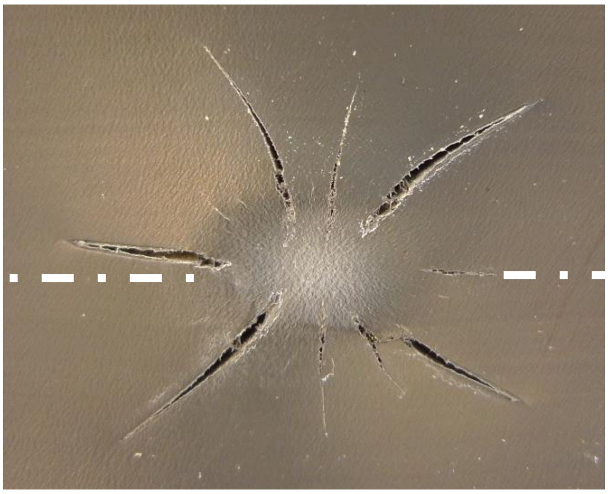
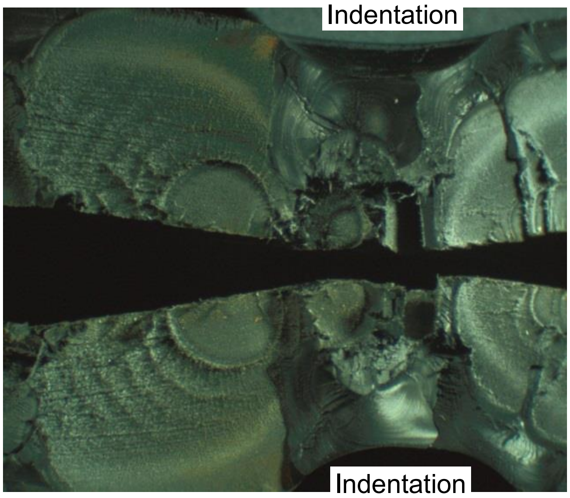


Figure 13. The inner wall of a second generation PE pipe after the PLT at 80 °C and 8 mm indentation in Dehyton shows multiple cracking. The cracks start just next to the centre of the indentation where no yielding occurs.



Outside
Inside
Outside

Figure 14. Brittle fracture surfaces after the PLT at 4.0 MPa and 80 °C in Dehyton. On the left the concentric ovals typical for slow crack growth are present.

CONCLUSIONS

1. The latest Round Robin of the SH test, in which each laboratory prepared its own samples, shows very low scatter between the seven different laboratories.
2. Due to the preparation by compression moulding of the test specimens, the SH test is a resin test and gives no information about the residual quality of the pipe. Experiments in which specimens are prepared directly from pipes should give insight into whether the SH test can also be used for the remaining quality of the pipe.

3. The PLT has been developed as a pipe test for PE 100-RC. Using the detergent Dehyton inside the pipe leads to less scatter than in Arkopal and also speeds up the failure process. Increasing the temperature would accelerate it even further.
4. Lowering the internal pressure of old first generation pipelines in practice makes them less prone to failure due to point loading.
5. Multiple cracking occurs in a circular region around the bulge in the internal pipe surface as a result of the point loading. This is due to the high stresses, which are however necessary in order to speed up the failure process.

FUTURE WORK

Future work will focus on ensuring the reproducibility and comparability of the tests. For the SH test, much work has already been performed in this field []. This means that more investigations should be performed into the robustness of the SH test (the influence of variations in test parameters). For the PLT, it means that the correct test parameters must be determined and test results between labs must be compared.

The minimum requirements for PE 100-RC for both the SH test and the PLT must eventually be agreed upon. For the SH test, many experiments with PE 100 and PE 100-RC have already been performed. This has yet to be done for the PLT.

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