STRAIN HARDENING TESTS ON PE PIPE MATERIALS

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Plastic Pipes XVI Conference – Barcelona, Spain, September 24-26, 2012

The Strain Hardening (SH) test developed by Kurelec et al. has been applied to a variety of PE pipe and resin materials. The clear trend in the strain hardening modulus, of first, second and third generation PE pipe resins and PE100 RC pipe resins, illustrates the large progress in resin quality during the past four decades.
Next, a Round Robin test on a PE100 RC, PE100 and PE80 material was set up. The results obtained by eight labs are reproducible. This illustrates that the SH test is very promising to assess the resistance to slow crack growth of a wide range of PE pipe materials in a short-term experiment and in a reproducible manner.
SH tests were also performed on five pipe materials selected from the GERG (European Gas Research Group) Materials Bank and compared to their previously published FNC, Cone and Notch test results.
Tentative results of SH tests on another semi-crystalline polymer (Polyoxymethylene - POM) were obtained. Since the results of the SH test are determined by the intrinsic material properties, the test parameters used for PE cannot simply be copied.

INTRODUCTION

Slow crack growth (SCG) is one of the most important failure mechanisms for installed PE gas and water pipelines. SCG failure may occur if 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 (see Figure 1).

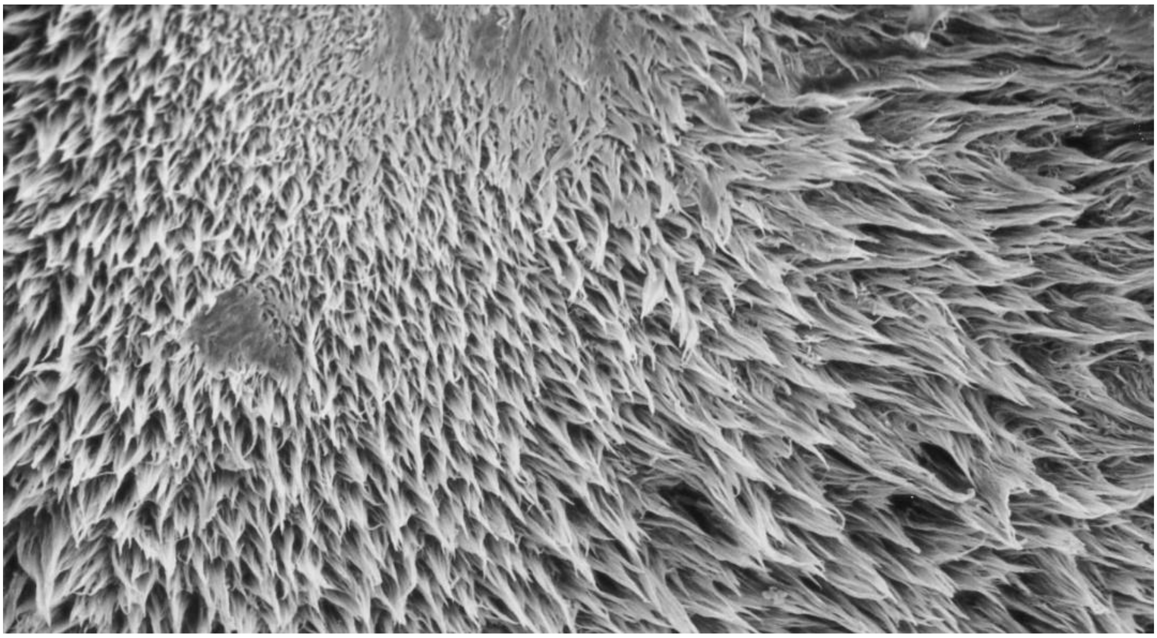

Figure 1. High-resolution SEM photograph of a SCG fracture.

Many different test methods exist for measuring and ranking the resistance of the material against this phenomenon, like the Internal Water Pressure (Stress Rupture) test [1], the Notched Pipe Test (NPT) [2], the Cone test [3], the Pennsylvania Notch Test (PENT) [4] and the Full Notch Creep Test (FNCT) [5].
However, because the properties of PE resins for pipes have been improved continuously during the last decades, testing times and testing costs have increased considerably. Moreover, some tests, like the FNCT show considerable inter-laboratory scatter, most likely due to degradation of the detergent. Some detergents are after all very vulnerable to oxidation [6].

The most recent addition of improved materials is PE100 “RC” (Resistant to Cracking). Slow Crack Growth resistance has now been improved far beyond that of a traditional PE100 material.

The new Strain Hardening (SH) test of SABIC (the Netherlands), published by Kurelec et al [7], McCarthy et al [8, Havermans et al [9,10] and Deblieck et al [11], probes the intrinsic material property that correlates with the resistance to SCG, thus making a ranking possible. A good correlation with the FNCT, which ranks the material according to the resistance to SCG as well, has been found [9,11].

TEST METHOD

The SH test is a modified tensile test performed at 80 °C (176 °F) on a specially prepared thin sample (Figure 2). The $(0.30^{+0.05}_{-0.03})$ mm (0.0118 inch) thick samples were punched from a compression moulded sheet made from PE granules that were pressed in accordance with the press parameters in ISO 1872-2 [12]. The thin samples were clamped and pulled at 20 mm/min while carefully measuring the elongation. An optical or laser extensometer must be used (Figure 3), because the sample is tested above its natural draw ratio, where the strain hardening occurs, and the displacement of the cross head cannot be used.

Figure 2. Six SH samples.

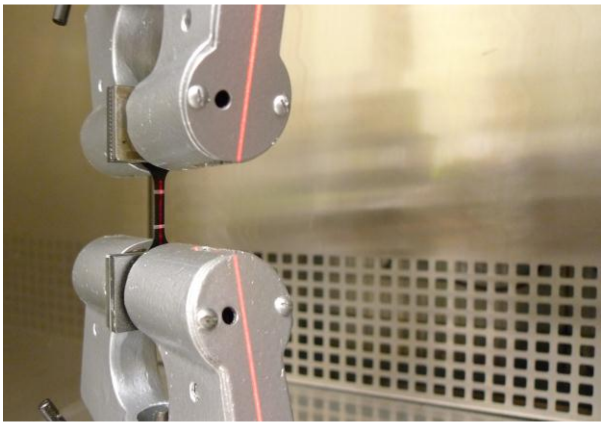

Figure 3. A SH sample ready for testing using a laser extensometer.

The true strain, λ (-), is calculated from the length, l (mm), and the gauge length, l_0 (mm), as shown by formula 1.

$$\lambda = \frac{l}{l_0} = 1 + \frac{\Delta l}{l_0} \tag{1}$$

where

Δl is the increase in the specimen length between the gauge marks (mm)

The true stress, σ_{true} (MPa), is calculated according to formula 2, assuming conservation of volume between the gauge marks:

$$\sigma_{\mathrm{true}} = \lambda \cdot \frac{F}{A} \tag{2}$$

where

F is the measured force (N)

A is the product of initial width and thickness (mm^2)

Two examples (materials X and Y) of the 'true stress - true strain' curve are given in Figure 4 (red and blue curve respectively). The curve between λ=8 and λ=12 can be modelled with the Neo-Hookean Strain Measure (NHSM) according to:

$$NHSM = \left(\lambda^2 - \frac{1}{\lambda}\right) \tag{3}$$

This is shown with a dashed green line in Figure 3.

It is also possible to plot the true stress against NHSM, resulting in a linear correlation between NHSM=63.875 (λ=8) and NHSM=143.917 (λ=12) (Figure 5). The slope between these two NHSM-values is called the G_p. The **strain hardening modulus** is defined [7] as

$$<G_\mathrm{p}> = 20 \times G_\mathrm{p} \tag{4}$$

The calculated strain hardening modulus for material X is:

$<G_\mathrm{p}> = 20 \times 2.2596 = 45.2$ MPa

and for material Y:

$<G_\mathrm{p}> = 20 \times 1.2840 = 25.7$ MPa

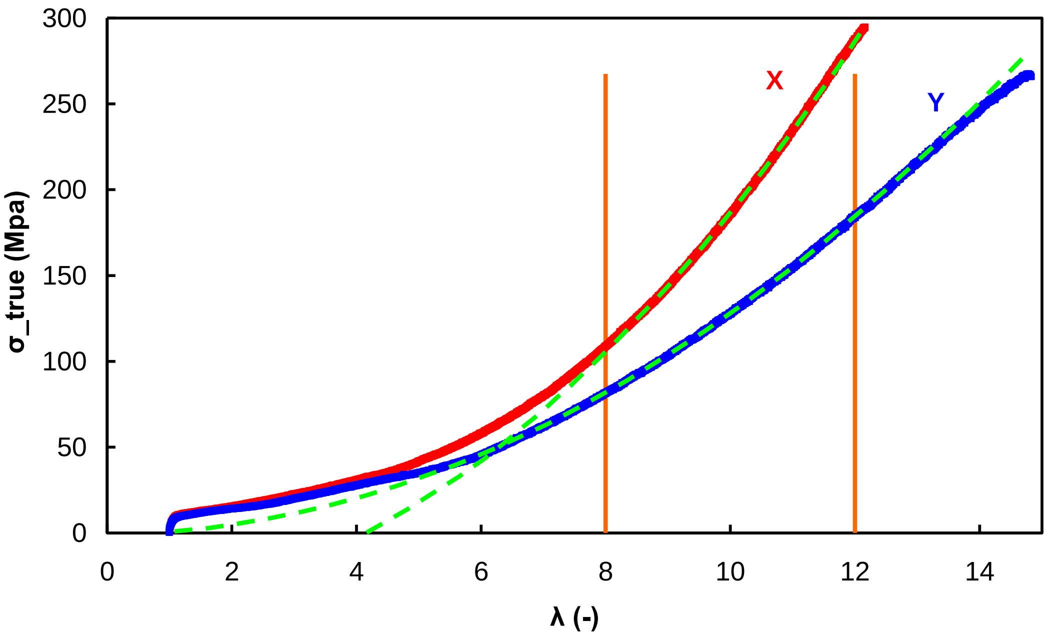

Figure 4. Results of the SH test (true stress against true strain of PE materials X and Y). The dashed green line is the NHSM fit between λ=8 and λ=12

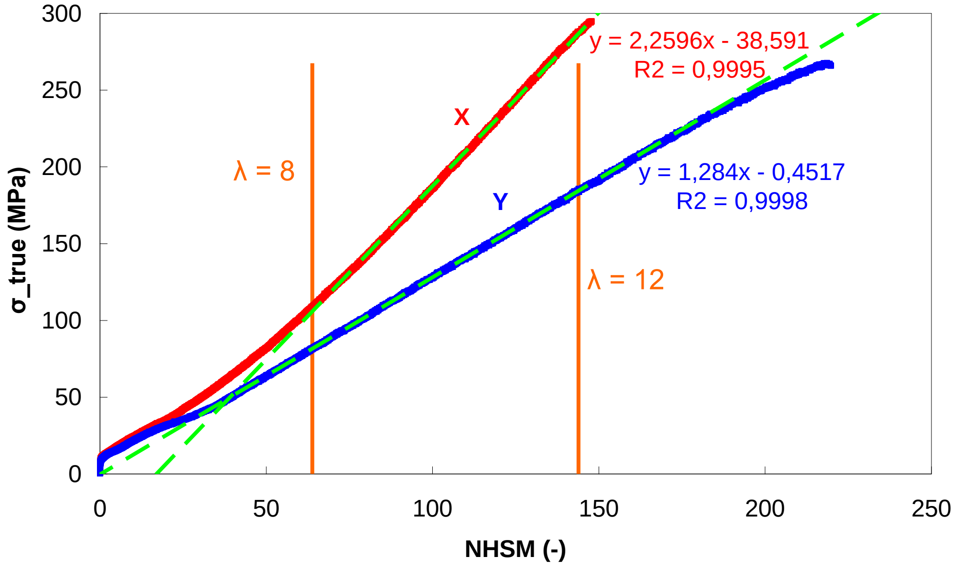

Figure 5. Results of the SH test (true stress against Neo-Hookean Strain Measure) on the same materials X and Y as in Figure 4. The dashed green line is the fit between NHSM≈64 (λ=8) and NHSM≈144 (λ=12).

RESULTS WITH DIFFERENT TYPES OF PE

The potential of the SH test has been investigated by several major European resin and pipe manufacturers together with several research institutes. The goal of this group is to draft a guideline for PE100 RC.

First, the potential of the SH test is illustrated by investigating different generations of PE. Two first generation PE (HDPE) types were tested as well as two second generation PE types (HDPE and MDPE). Furthermore, five European resin manufacturers sent one PE100 and one PE100 RC (one manufacturer sent two PE100 RC materials), without revealing which material was which. For each material, six samples were measured.

Figure 6 shows the results of the comparison. The distinction between the different types is clear, with the strain hardening modulus ranging from just below 20 MPa up to almost 80 MPa. The different PE types can easily be distinguished.

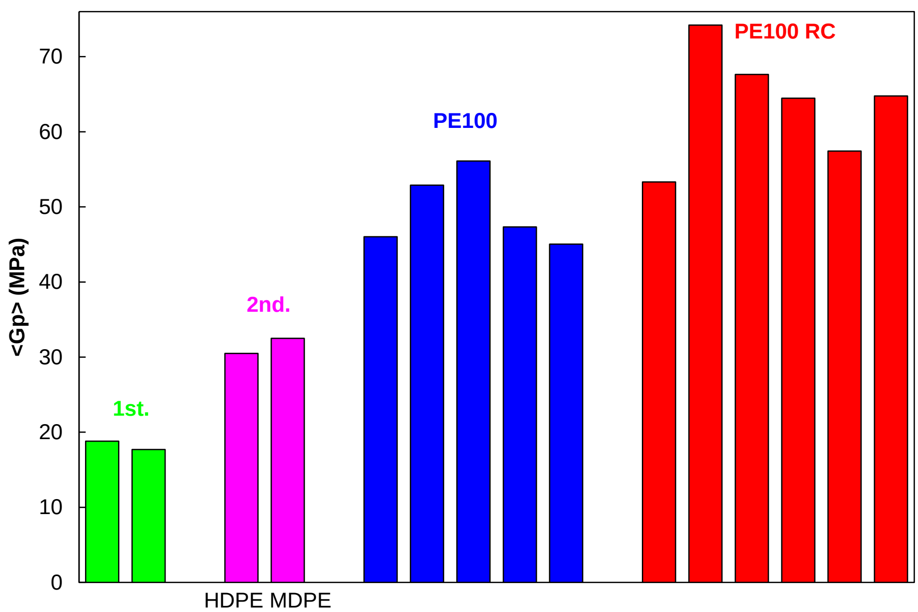

Figure 6 Strain Hardening Modulus <Gp> of four classes of PE pipe materials.

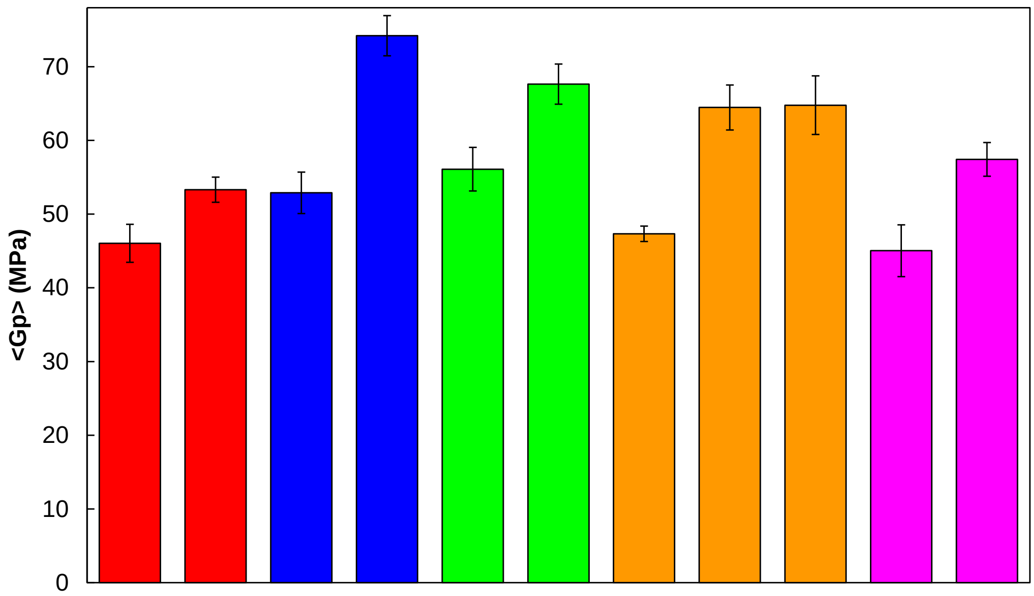

Figure 7. Strain Hardening Modulus <Gp> of PE100 and PE100 RC by five resin manufacturers.

Please note that these results were obtained within a matter of days, in contrast to tests with FNCT, which would have taken months or probably longer.

When looking only at the PE100 and the PE100 RC materials from each manufacturer who sent their materials without any indication of PE type, the results speak for themselves (Figure 7). For each manufacturer the PE100 RC material was easy to point out, having the highest value. The Y error bars indicate one standard deviation up and one down. Each colour represents one manufacturer. Please note that some of the best PE100 materials could compete with some PE100 RC materials.

ROUND ROBIN TEST

Next a Round Robin test between different laboratories was performed with three materials:

A. PE100 RC
B. PE100
C. PE80 (2^{nd} generation HDPE, produced in 1986)

All materials are black and cannot be distinguished except for the designated type letter.

Eight laboratories performed the SH test. The results are given in Figure 8 and Table 1.

The average results are based on all individual samples.

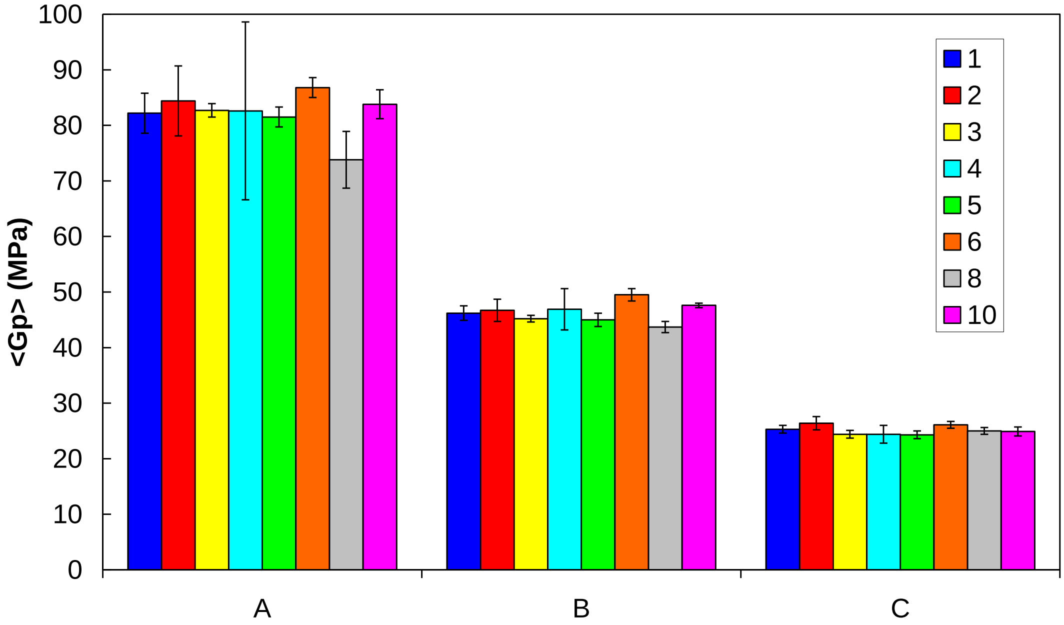

Figure 8. Results of the Round Robin test performed by eight laboratories on three PE materials.

Table 1. Round Robin test between eight laboratories on three materials.

	A				B				C			
Lab Nr.	**<G_p> MPa**	**SD MPa**	**C_v %**	**n**	**<G_p> MPa**	**SD MPa**	**C_v %**	**n**	**<G_p> MPa**	**SD MPa**	**C_v %**	**n**
1	82,2	3,6	4,4	6	46,2	1,3	2,8	6	25,3	0,7	2,8	6
2	84,4	6,3	7,5	6	46,7	2,0	4,3	6	26,4	1,2	4,5	6
3	82,7	1,2	1,5	3	45,2	0,6	1,3	3	24,4	0,7	2,9	3
4	82,6	16,0	19,4	9	46,9	3,7	7,9	6	24,4	1,6	6,6	6
5	81,5	1,8	2,2	6	45,0	1,2	2,7	6	24,3	0,7	2,9	6
6	86,8	1,8	2,1	5	49,5	1,1	2,2	5	26,1	0,6	2,3	5
8	73,8	5,1	6,9	3	43,7	1,0	2,3	3	25,0	0,6	2,4	3
10	83,8	2,6	3,1	6	47,6	0,4	0,8	6	24,9	0,8	3,2	6
All samples	**82,7**	**8,0**	**9,7**	**44**	**46,6**	**2,3**	**4,9**	**41**	**25,1**	**1,1**	**4,4**	**41**
Without 4 & 8	**83,5**	**3,7**	**4,4**	**32**								

For materials B and C, the results of all eight laboratories are very similar, with a variation coefficient (C_v) or relative standard deviation under 5 %. The variation coefficient for material A is somewhat higher, which is caused mainly by laboratory 4 (large standard deviation (SD), but a good average result) and laboratory 8 (average value too low). The large scatter for material A measured by laboratory 4 is caused by the fact that the distance between the gauge marks have been decreased from 12 mm to 4 mm. This was needed to measure the elongation over the entire range. The deviating value for material A measured by laboratory 8 is caused by the fact that no camera or laser has been used by for the extensometer, but a contact

extensometer has been used. This combined with the measurement of only three samples, caused the deviation in the average results when only one sample had a too low value.
If these two laboratories are omitted from the results, the variation coefficient also drops below 5 %.

RESULTS OF GERG MATERIALS

The GERG Materials Bank was founded in 1987 by various European gas organisations (Advantica Technologies, Gaz de France, Italgas, Ruhrgas, ARGB, gasNatural and Gastec). Various PE pipe grades were mechanically tested and structural analyses were performed. Results have been reported at several Conferences [13,14,15,16].

The SH test was performed on five pipes produced in 1987: O1, O2, O3, O5 and O6 [16]. In addition one PE100 pipe produced in 1995 (M5) and one PE100 pipe produced in 1999 were tested (M9) [17]. The results of the SH test are compared to SCG tests, such as Cone, internal water pressure test, PENT, Notch test and FNCT. These results are given in Table 2 and sorted by the SH test results for easy comparison.

Table 2. Results of various mechanical tests compared. The materials are sorted on the results of the SH test.

	SH	**Cone [16]**	**Internal water pressure at 4.6 MPa [16,17]**	**PENT at 2.4 MPa [16,17]**	**Notch at 4.6 MPa [16,17]**	**FNCT at 3.5 MPa [16,17]**	**FNCT at 4.0 MPa [16,17]**
Mat. code	**<Gp> (MPa)**	**Crack growth rate (mm/day)**	**Failure time (hours)***				
O2	23.0	4.24	2791	173.5	159.1	24.2	19.4
O5	26.1	6.82	401	8.0	60.6	22.0	17.8
O1	30.5	3.83	480	28.2	53.8	36.0	22.5
O3	32.5	0.00	3805[§]	>1151.0	2228.0	139.0	79.6
O6	32.7	0.00	>7402[§]	>1125.4	2023.5	177.5	106.7
M5	41.8	0.99 / 0.55[#]	>15763[†] / >2875[‡§]	>1076.0	2478.7	395.1	273.9
M9	45.0	0.58[#]	>7746[†] / 544[‡§]	370.7	>1210.5	311.0	174.5

* *Geometric mean instead of arithmetic mean is used*
§ *One or more ductile failures*
\# *Surrounding atmosphere nitrogen instead of air*
† *Internal water pressure at 5.3 MPa*
‡ *Internal water pressure at 5.7 MPa*

The crack growth rate order in the Cone test is comparable to that of the $<G_p>$ in the SH test. Material O5 has a high crack growth rate, indicating a low resistance to SCG. The best material in the SH test (M9) also has almost the lowest crack growth rate, except for materials O3 and O6 of which no crack growth was measured. These two materials are MDPE's, which may show a rather long incubation time of zero crack growth in the Cone test before stable crack growth sets in [6].

All five pipes produced in 1987 were tested with internal water pressure at 4.6 MPa. The results of the five materials in this test are ordered in the same way as the results of the SH test except for O2.

The PENT, performed at 2.4 MPa, only resulted in very short failure times for materials O5 and O1, which also have a low <G_p>. No distinction between materials O3, O6 and M5 could be made, but all three are better than the other pipe samples. Materials O2 and M9 have an average result in the PENT, but a low and high value in the SH test.

In the Notch test the best distinction is that O2, O5 and O1 have short failure times and O3, O6, M5 and M9 have longer failure times.

The FNC test shows almost the same order as the SH test, regardless of whether the test was performed at 3.5 MPa or 4 MPa. This correlation is shown in Figure 9. The correlation is only a small part of the correlation published by Havermans et al [9].

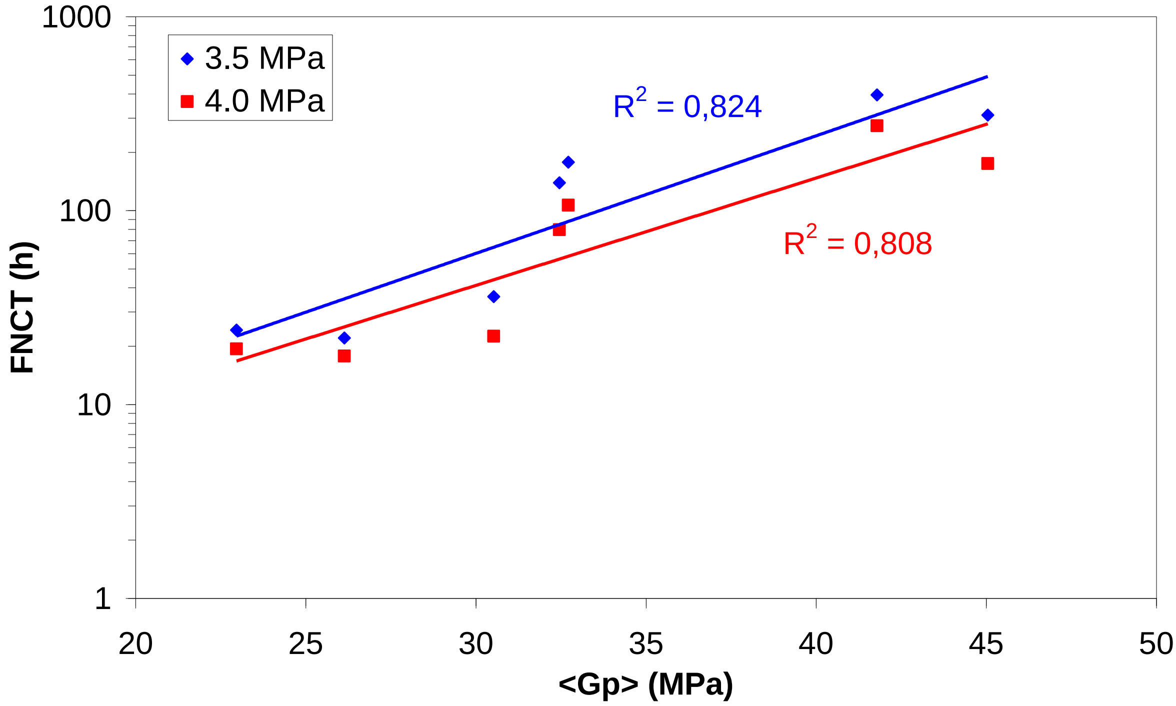

Figure 9. Correlation between the FNCT in 2 % Arkopal N110 [16,17] and SH test for seven GERG pipe materials.

STANDARDISATION

The results of these investigations are shared with ISO (ISO/TC138/SC5/WG20 "Slow crack growth (SCG)"). This has resulted in sending the SH test method as a New Work Item Proposal for making it an international test standard.

RESULTS ON POM

POM, or Polyoxymethylene or Polyacetal, is a semi-crystalline polymer like PE. POM is widely used in many applications. The materials exist as homopolymer and copolymer. Since the results of the SH test are determined by the intrinsic material properties, the test parameters used for PE cannot simply be copied.

This can quickly be seen from the results for when the same test parameters as for PE were used (Figure 10). Since POM is far more brittle, the elongations were very limited. Nevertheless, clear differences between the maximum elongations of materials P, Q and P can be seen.

Tests at higher temperatures were performed in an attempt to improve testing parameters. Figure 11 and Figure 12 show the results of the same POM materials using 100 °C and 120 °C respectively.
At 100 °C material N has the longest elongation, while material P has the shortest. The materials demonstrate a slope that can be modelled with the NHSM (dashed green line in Figure 11). The $<G_p>$ of materials N and Q have been calculated between λ=3 and λ=5 (see Table 3).
At 120 °C material Q suddenly has the longest elongation. P again has the shortest elongation. Again, the slope can be modelled with the NHSM. The $<G_p>$ between λ=8 and λ=12 is shown in Table 3.

It can be concluded that more research is needed to find the correct test parameters for semi-crystalline materials other than PE.

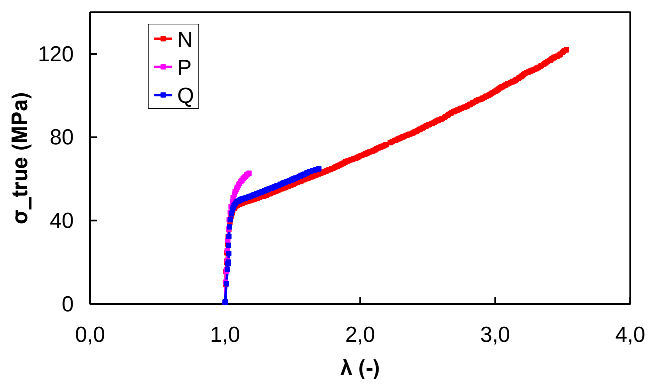

Figure 10. SH results on POM using the same test parameters as for PE.

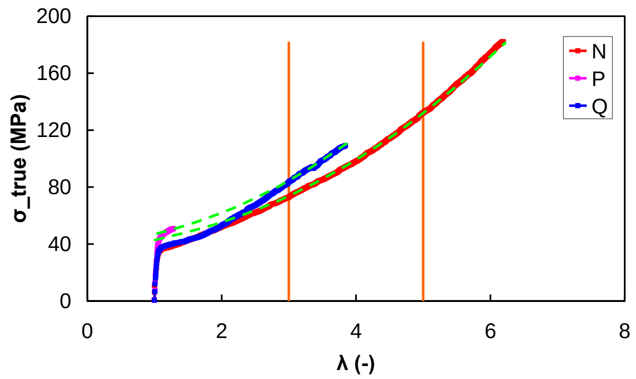

Figure 11. SH test on POM performed at 100 °C. The dashed green line is the NHSM fit between λ=3 and λ=5.

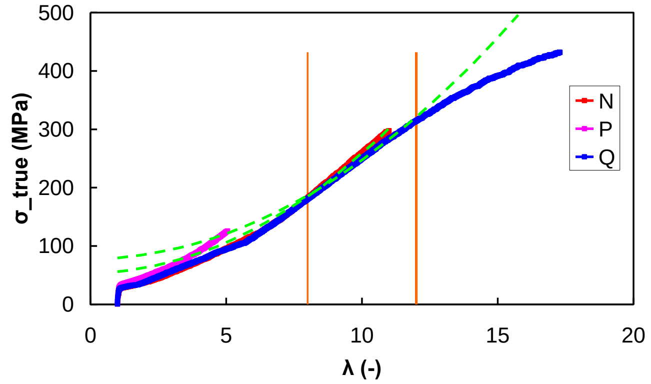

Figure 12. SH test on POM performed at 120 °C. The dashed green line is the NHSM fit between λ=8 and λ=12.

Table 3. Strain hardening modulus <Gp> (MPa) for two POM materials at two temperatures.

Temperature (°C)	Material	
	N	**Q**
100	72 MPa	88 MPa
120	41 MPa	39 MPa

CONCLUSIONS

The SH test can easily distinguish different PE types and even the differences between PE100 and PE100 RC of five different resin manufacturers are very clear. Results of eight laboratories on three materials on the SH test are very similar, with a very low variation coefficient. SH results on five materials of the GERG are comparable to the results of other SCG tests, such as Cone, internal water pressure test, PENT, Notch test and FNCT in particular. ISO/TC138/SC5/WG20 has submitted the Strain Hardening (SH) test method as a New Work Item Proposal for making it an international test standard. Tentative results of SH tests on another semi-crystalline polymer (Polyoxymethylene - POM) show that the test parameters used for PE cannot simply be copied.

ACKNOWLEDGEMENTS

The authors wish to thank Matthijs Schrijver and Jolanda Brugman for their meticulous SH experiments. Thanks go to Linda Havermans (Sabic) and Rainer Kloth (Sabic) for their valuable and useful discussions; to the GERG members (E.ON Ruhrgas, GDF SUEZ and National Grid UK) for their support in this project and to the members of the Guideline Group (Borealis, E.ON Ruhrgas, Egeplast, Gerodur, Ineos, Intertek, Lyondell Basell, Pipelife, Sabic, SKZ, Total, TGM and Wavin) for their input (material and knowledge), discussions and suggestions.

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