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DETERMINING THE RESIDUAL QUALITY OF PE PIPES USING THE STRAIN HARDENING TEST

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SHORT SUMMARY

Samples machined directly from pipe show that orientation has a large influence on the strain hardening modulus. No effects arising as a result of residual stresses were found. Samples machined in the axial direction from a hydrostatic pressurised pipe showed no effect as a result of this mechanical degradation. For now, also no clear effect on the <Gp> is observable for samples machined in tangential direction, but more research is needed to confirm this.

KEYWORDS

Strain Hardening Test, SHT, polyethylene raised resistance to cracking, PE 100RC, Slow Crack Growth, SCG, orientation, residual stresses

ABSTRACT

The strain hardening test (SHT), carried out in accordance with ISO 18488, is a very quick and reliable test to determine the disentanglement capacity of the tie molecules in polyethylene (PE). The resulting strain hardening modulus (<Gp>) is an intrinsic property related to the slow crack growth (SCG) resistance. The power of this simple tensile test is especially obvious when testing PE 100RC material, which has an extremely high SCG resistance and makes laying pipes without a sand bed possible.

Previous studies have shown that the SHT on re-melted samples is a resin test and gives no information about the residual quality of the pipe. In this study, test samples were machined directly from PE pipe to circumvent re-melting. Two effects were taken into account, namely orientation and residual stresses.

The PE molecules in a pipe are oriented as a result of the extrusion process. The machining direction of the test bars may therefore influence the results. Samples were machined in the axial, tangential (parallel to the pipe circumference), and radial (from the inner wall to the outer wall) pipe direction. Uneven cooling of the extruded PE pipe results in residual stresses. Test bars machined axially from the outer wall, the wall centre and the inner wall were also tested.

Orientation appears to be of great influence. The axial and tangential samples had a higher <Gp>, while the radial samples had a lower <Gp> than re-melted samples. In the axial and tangential machining directions, there was almost no difference in <Gp> between the locations in the wall described above. This indicates that residual stresses do not influence the <Gp>.

This knowledge was used to evaluate the residual quality of old first-generation PE pipes, which had been additionally mechanically degraded using hydrostatic pressure. The results for samples machined directly from pipe were compared with re-melted samples and with the results from the Pennsylvanian Notch Test (PENT).

Samples machined in the axial direction from the hydrostatic pressurised pipe showed no effect on the <Gp> as a result of this mechanical degradation. More research using pipes with longer hydrostatic loading times is needed to obtain better trend with samples machined in tangential direction. For now, no clear effect on the <Gp> is observable for the samples machined in this direction. More research is also needed to explain why the axial samples had a higher <Gp> than the samples prepared from granulate, while the tangential samples sometimes had a higher and sometimes a lower <Gp>.

INTRODUCTION

The strain hardening test (SHT), carried out in accordance with ISO 18488 [1], is a very quick and reliable test to determine the disentanglement capacity of the tie molecules in polyethylene (PE) [2,3,4]. The resulting strain hardening modulus (<Gp>) is an intrinsic property related to the slow crack growth (SCG) resistance [5,6,7]. The power of this simple tensile test is especially obvious when testing PE 100RC material, which has an extremely high SCG resistance and makes laying without a sand bed possible. The good performance of the SHT in discriminating between different PE types and the low interlaboratory scatter has been published previously [8,9].

The previous study showed that the SHT on re-melted samples is a resin test and gives no information about the residual quality of the pipe [9]. In this study, test samples were machined directly from PE pipe to circumvent re-melting. As a first step, the effect of two parameters on the <Gp> were investigated, namely orientation and residual stresses. The <Gp> as a function of the hydrostatic loading time was subsequently determined in order to simulate mechanical degradation.

EXPERIMENTAL METHOD

The strain hardening test (SHT) is a modified tensile test performed at 80 °C (176 °F) on specially prepared thin samples. When the samples are prepared from PE granules, 0.3 mm (0.0118 inch) thick samples are punched from a compression moulded sheet that has been pressed and annealed at 120 °C (248 °F) for one hour in accordance with ISO 18488 [1].

Since the temperature used for compression moulding is 180 °C (356 °F), the granules in fact become molten. The hypothesis is that this completely removes the orientation, the residual stresses and the thermal (and thus mechanical) history of the sample. This would mean that the SHT, as currently described, cannot be used to determine the quality of the pipe material, but only the original resin quality.

To prevent this thermal reset, samples were prepared directly from pipe. In this case samples were punched from a 1.0 mm (0.0394 inch) thick sheet that had been machined directly from the pipe wall. Machining was performed carefully to prevent plastic deformation as much as possible. Air was used to cool the samples during preparation. The maximum temperature at the surface of the sample during machining was 42 °C (107.6 °F) as measured with an infrared camera. No compression moulding or annealing was performed on these samples.

The samples were clamped and pulled at 20 mm/min while the elongation was carefully measured. A non-contact (optical) extensometer and a load cell of 500 N were 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 <Gp> [7,8].

SH samples were machined from different positions in a Ø630 mm PE100 pipe (wall thickness: 50 mm) that had been fabricated relatively recently, coded “PE 2007-228”, to determine the effect of orientation and residual stresses on the <Gp>. The positions vary in orientation (*zy*, *yx*, *yz* and *xy*, Figure 1) and in location within the pipe wall (outside (*o*), middle (*m*) and inside (*i*)). For each position, seven to ten SHT samples were prepared.

It must be noted that preparing samples in the tangential (*yz*) direction is only possible for pipes with a diameter greater or equal to 160 mm, because it is not possible to machine samples that fulfil the dimensional requirements of ISO 18488 from smaller diameter pipes.

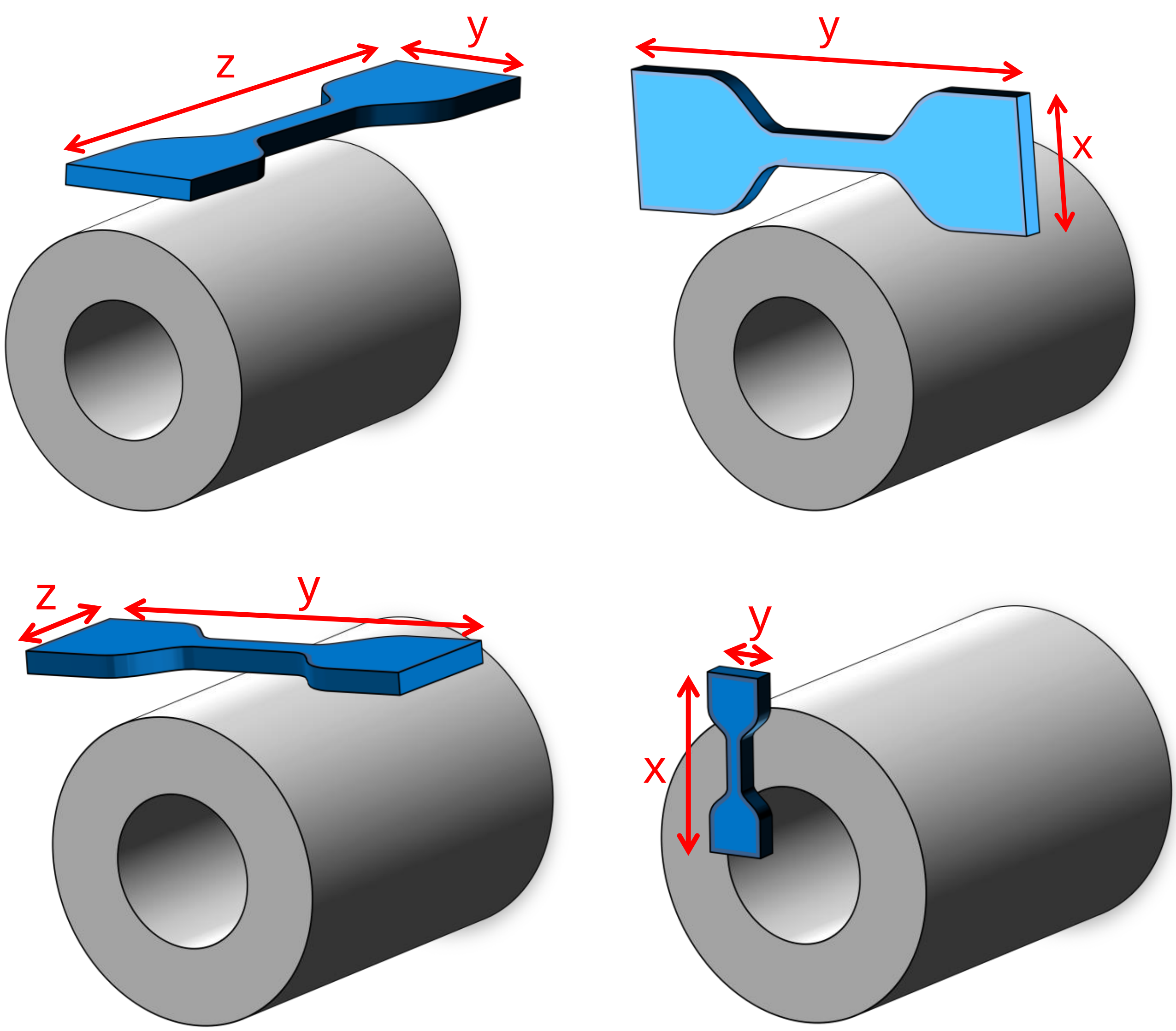


Figure 1. The different directions in a pipe: x is the radial direction (perpendicular to the pipe wall), y is the tangential direction (circumferential) and z is the axial direction (longitudinal). SH samples were machined in four directions. Other possibilities are outside the scope of this study.
Top left: SH sample zy (length of the specimen in axial direction and width in tangential direction)
Top right: SH sample yx (length of the specimen in tangential direction and width in radial direction)
Bottom left: SH sample yz (length of the specimen in tangential direction and width in axial direction)
Bottom right: SH sample xy (length of the specimen in radial direction and width in tangential direction)

Small plates were machined in various directions from the pipe as described above. Three plates were prepared for each direction. The dimensions were approximately 50 x 15 x 1 mm. After accurately measuring the dimensions in the X, Y and Z directions, the samples were heated in an air oven for 1 hour at 110 °C. These correspond to the test parameters as given in ISO 2505 [10], which is a standard for determining the longitudinal reversion. After heating and subsequent cooling, the dimensions in all three directions were recorded once again. The dimensional change was divided by the original dimension to determine the dimensional reversion.

To simulate mechanical degradation in practice, three segments from the two used first-generation PE pipes (coded “PE 2013-149” and “PE 2009-001”, Ø110 mm and Ø160 mm respectively) were hydrostatically pressurised to 3 MPa (435 psi) at 80 °C. The segments were stressed for 125, 250 or 500 hours. For the purposes of comparison, one segment was not subjected to hydrostatic pressure (0 hours).

Samples for the Pennsylvanian Notch Test (PENT [11]) and SHT were taken from the middle of the pipe segment, as far as possible from the ends of the pipe. The PENT samples from both PE pipes (four from each) were notched as described in ISO 16241 [11] and tested at 80 °C at a stress of 1.5 MPa (instead of 2.4 MPa [11]). The SH samples were prepared in accordance with ISO 18488 [1], while other samples were prepared directly from pipe. Fifteen samples from PE 2013-149 were machined in the *zy*-direction from the middle of the pipe wall. Seven samples from PE 2009-001 were machined in the *yz*-direction from the pipe wall.

The results were compared statistically using One-Way ANOVA. An overall significance level of 0.05 was used. The groups were compared using the Holm-Sidak method.

RESULTS AND DISCUSSION

<u>Orientation</u>

When a PE pipe is extruded, the polymer chains in the molten granulate will orientate themselves to a certain extent. This orientation is maintained when sheets are prepared directly from pipe. In the preparation procedure as carried out in accordance with ISO 18488, PE granulate is molten during compression moulding. The sheets are subsequently annealed. Once this has been carried out, the polymer chains have a very low orientation and are almost completely randomly oriented throughout the sheet. By comparing the strain hardening modulus (<Gp>) of the re-melted samples prepared in accordance with ISO 18488 with samples that have been machined directly from pipe, the effect of orientation can be evaluated.

Residual stresses are also introduced during pipe production. The freshly-extruded pipe is cooled with water from the outside, while the inner side and the middle of the pipe wall remain warm for a longer time. This asymmetric cooling results in residual stresses. On the outer side of the pipe wall the stresses are compressive, while tensile stresses will be present on the inner side.

Figure 2 shows the <Gp> for the investigated samples. The <Gp> for this pipe was 66.0 MPa when prepared and tested in accordance with ISO 18488. Samples machined in the axial direction (*zy*) had a higher <Gp> than the samples prepared from granulate, namely an average of 72.1 MPa. The samples prepared in the tangential direction (*yz*) also had a higher <Gp>, namely an average of 75.4 MPa. Both results also deviated statistically (One-Way ANOVA) from the <Gp> of the granulate. The other samples prepared in the tangential direction (*yx*) had more or less the same <Gp> (67.2 MPa) and did not deviate statistically. Only the samples machined in the radial direction (*xy*) had a lower <Gp>: 59.6 MPa (statistically different when compared to granulate).

The differences between the granulate, the *zy*-samples, the *yx*-samples, the *yz*-samples and the *xy*-samples therefore show that orientation has an influence on the <Gp>. The direction of preparation is thus crucial for the resulting <Gp>.

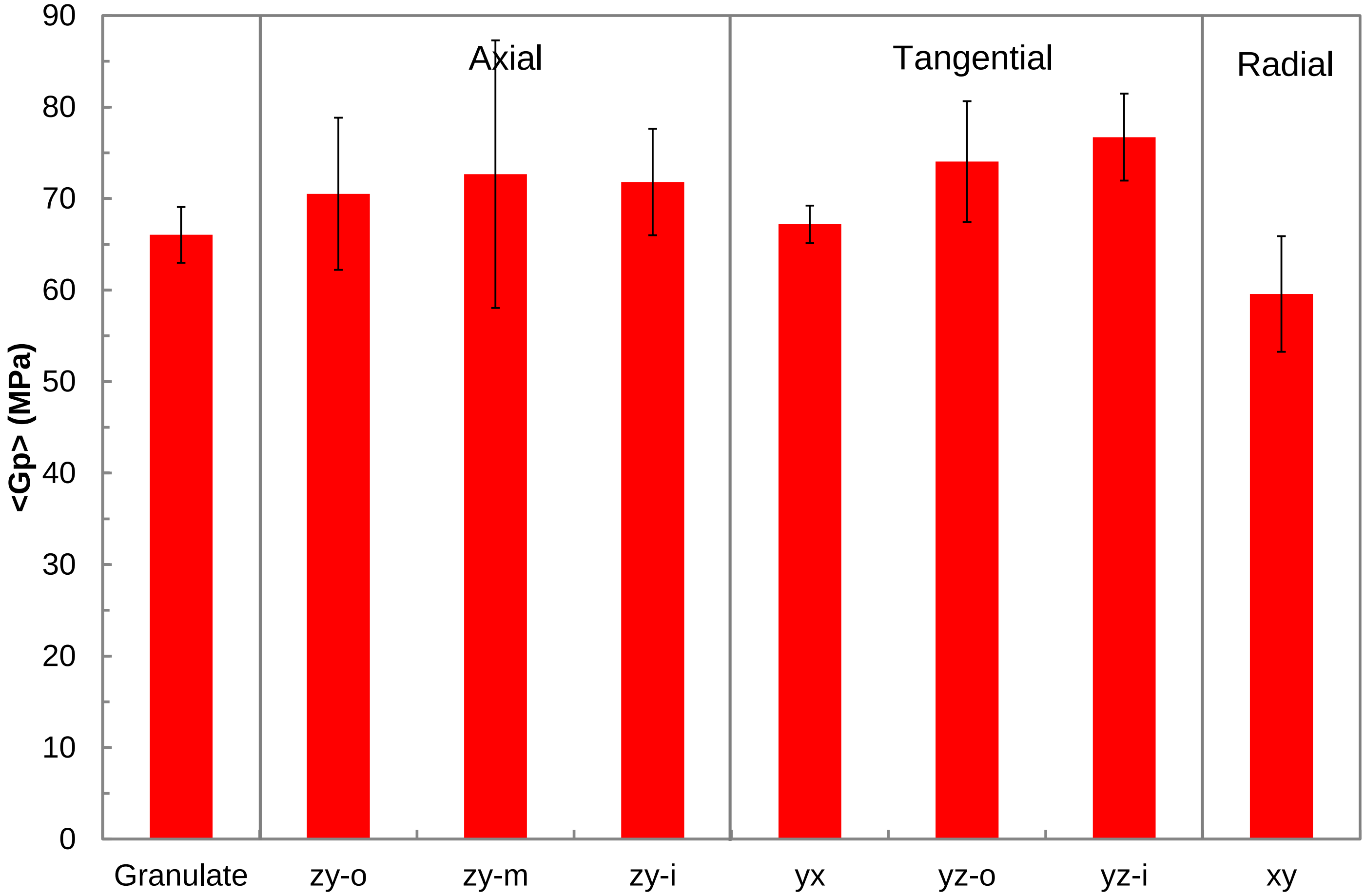


Figure 2. The strain hardening modulus (<Gp>) for various orientations (see Figure 1) and locations in the same pipe (PE 2007-228). x is the radial direction, y is the tangential direction and z is the axial direction. o is the outside, m is the middle of the pipe wall and i is the inner side of the pipe wall.

Residual stresses

Within one orientation, there was almost no difference in <Gp> observable between the *o*, *m* and *i* locations. The position within the pipe wall (inner side or outside), and thus the residual stresses, therefore seems to have no effect on the resulting <Gp>.

The lack of a measured difference for the SHT does not mean that there is no difference in residual stresses. The dimensional change was measured for samples from the outside and inner side of the pipe wall using the parameters for the longitudinal reversion test (see the experimental section). The results are presented in Table 1. The dimensional change for samples at the inside of the pipe wall was much smaller than samples from the outside (green and red cells respectively) due to fast cooling from the outside during the extrusion process. Note that the samples shrunk in the axial direction, whereas in the radial direction enlargement was found in all cases. This can be explained by the axial pull during the extrusion process. The axial elongation and the radial shrink are frozen in during the cooling step. When samples are heated again, this axial elongation is reduced once again, hence the shrinkage. The reverse is true for the radial direction.

Table 1. The dimensional reversion after 1 h in an air oven at 110 °C in three directions (X (radial), Y (tangential) and Z (axial)) for five different sample locations in PE 2007-228. A green background indicates that the value is relatively close to 0 % in that direction, while a red background indicates a relatively high (plus or minus) value in the same direction. A positive value indicates enlargement. A negative value indicates shrinkage.

	Dimensional reversion		
Sample location	**X (%)**	**Y (%)**	**Z(%)**
Axial outside (*zy-o*)	2.83	-1.41	-0.91
Axial inner side (*zy-i*)	0.87	-0.16	-0.62
Tangential outside (*yz-o*)	3.48	-1.32	-1.59
Tangential inner side (*yz-i*)	0.60	0.24	-0.67
Radial (*xy*)	1.77	-0.74	-0.92

Despite the measurable difference in residual stresses, the SHT did not measure any difference between the samples from different locations in the pipe wall. The preparation as carried out in accordance with ISO 18488 requires an additional annealing step at 120 °C for 1 hour. This will certainly decrease the residual stresses. It is quite possible that the test temperature of 80 °C is also sufficient to cause a relatively quick decrease in residual stresses due to relaxation. Moreover, at such high strains the residual stresses may be of no influence at all.

Note that these results do not reveal anything about the effect of residual stresses on the slow crack growth (SCG) process. The <Gp> is after all an intrinsic material property. It does not measure the resistance to SCG itself, but is related to it [6]. These results therefore only indicate that the SHT cannot measure any effects caused by residual stresses.

Mechanical degradation

This knowledge regarding the effects of orientation and residual stresses on the <Gp> was taken into account when preparing samples from pipe segments. These were mechanically degraded using hydrostatic pressure (see the experimental section).

The PENT failure time and the <Gp> of the PE 2013-149 pipes that were subject to hydrostatic pressure are shown in Figure 3. The results of the PENT (left axis on a logarithmic scale, light blue) and the SHT using granulate (right axis, open red squares) have been discussed in a previous paper [9]. This paper explains that the PENT is affected by the previously-imposed mechanical degradation, whereas the SHT is only a resin test.

The <Gp> (right axis, solid red squares) of the samples machined directly from pipe (*zy-m* location) is independent of the imposed mechanical degradation: the values remain more or less equal. This is similar to the results that have been found before using granulate. However, the difference is that the <Gp> values for the samples machined directly from pipe are higher than the samples prepared in accordance with ISO 18488. This corresponds to the results found earlier (see Figure 2).

Because the stress in axial (*zy*) direction is equal to half the stress in tangential (*yz*) direction, an effect on the <Gp> may be noticeable for samples machined in the tangential direction.

For this research a larger pipe, referred to as “PE 2009-001”, was used (see the experimental section). In this case, the <Gp> of the segment that was not subjected to hydrostatic pressure was already lower than the <Gp> for the samples prepared from granulate in accordance with ISO 18448. This outcome stands in contrast to the results presented in Figure 2, where the <Gp> is higher. This pipe was however old and had been used in practice for several decades. This means that mechanical degradation had already occurred. This could explain the lower-than-expected <Gp>.

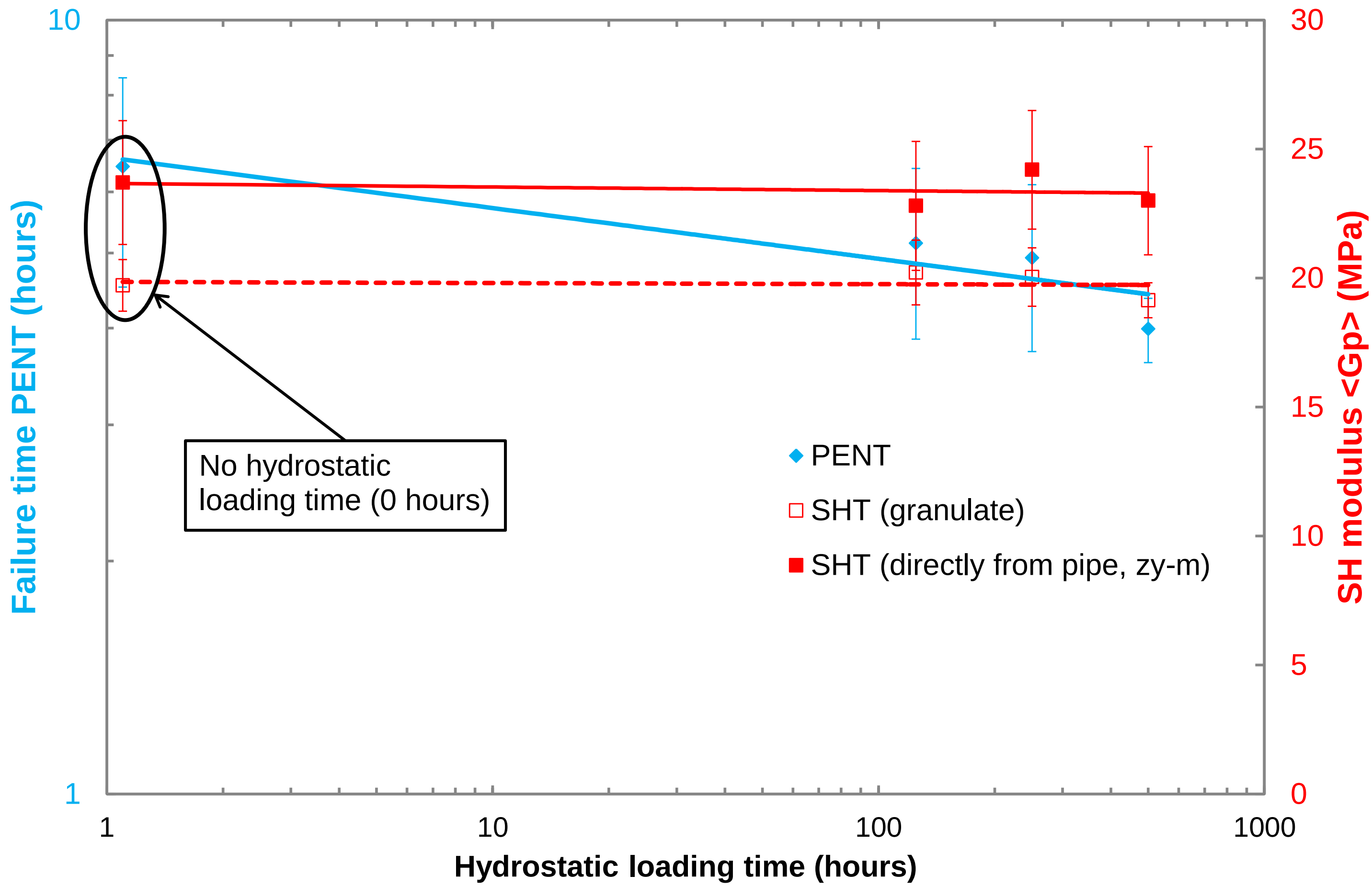


Figure 3. The failure time for the PENT (left axis on a logarithmic scale, light blue) and the strain hardening modulus of the SHT (<Gp>, right axis, red) for pipe "PE 2013-149". The hydrostatic loading time is shown on the horizontal axis (logarithmic). There is no "0" on a logarithmic scale; the value without hydrostatic loading is thus placed at 1.1 hours for clarity. The samples machined directly from pipe are taken from the middle of the pipe wall in the zy-direction (see Figure 1).

Four pipe segments were pressurised to 3 MPa at 80 °C and failed after 5, 30, 33 and 46 hours. The planned hydrostatic loading time 125, 250 and 500 hours was therefore not possible. To investigate the effect of further hydrostatic pressure, the *yz*-samples were machined from the pipe segment that failed after 46 hours. Similar as with the axial samples, the <Gp> does not decrease as a function of the hydrostatic loading time. Mechanical degradation may therefore not be the reason of the lower-than-expected <Gp>, although it must be noted that it was difficult to obtain a clear trend, because hydrostatic loading times up to 500 h were not possible.

It seems for now therefore that the fibril deformation and failure resistance, which are measured by the SHT, are not influenced by the mechanical history. Performing the SHT on *zy*- and yz-samples, which are not compression moulded or annealed, seem therefore not to measure the pipe quality, but are instead measuring the resin quality.

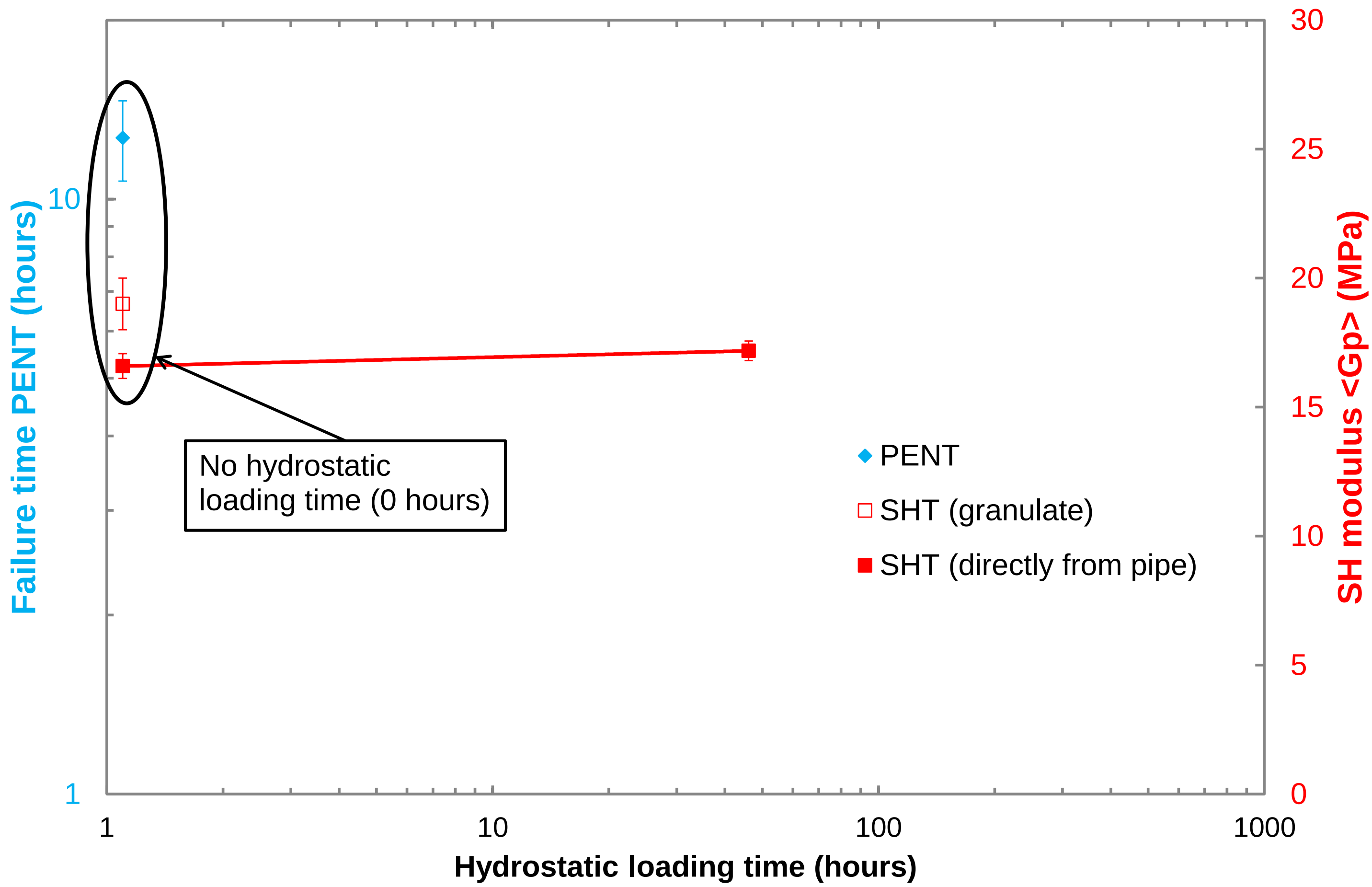


Figure 4. As Figure 3, but for PE 2009-001. Samples were machined in the yz-direction instead of the zy-direction (see Figure 1). The PENT and the SHT on granulate were only performed on the pipe not subjected to any hydrostatic loading. It is assumed that these tests respond identically to hydrostatic loading as presented in Figure 3.

It remains unknown why the axial (*zy*) samples from the Ø630 mm and Ø110 mm pipe had a higher <Gp> than the samples prepared from granulate (Figure 2 and Figure 3), while the tangential (*yz*) samples sometimes had a higher <Gp> (Ø630 mm pipe, Figure 2) and sometimes a lower <Gp> (Ø160 mm pipe, Figure 4). More research is needed to explain this behaviour.

Although the <Gp> of the samples machined in the axial direction (*zy*) was not affected by any mechanical degradation, the raw data hints at an effect due to the hydrostatic pressure. In Figure 5, a red circle indicates a change of slope in the SHT curve. The draw ratio (λ) for this change of slope was determined for all tested samples using the second derivative. The results are presented in Table 2. The λ at which the change of slope occurs for granulate remains more or less constant, while λ decreases for the pipe samples. This is only a trend, because statistically (using One-Way ANOVA) there is no difference between the groups. The trend is especially notable for the 0 h, 125 h and 250 h pipe samples.

This trend is not observed for “PE 2009-001” where the λ at which the change of slope occurs for the samples machined from tangential direction (*yz*) remains more or less constant (7.88 for the 0 h pipe samples and 7.93 for the 46 h pipe samples).

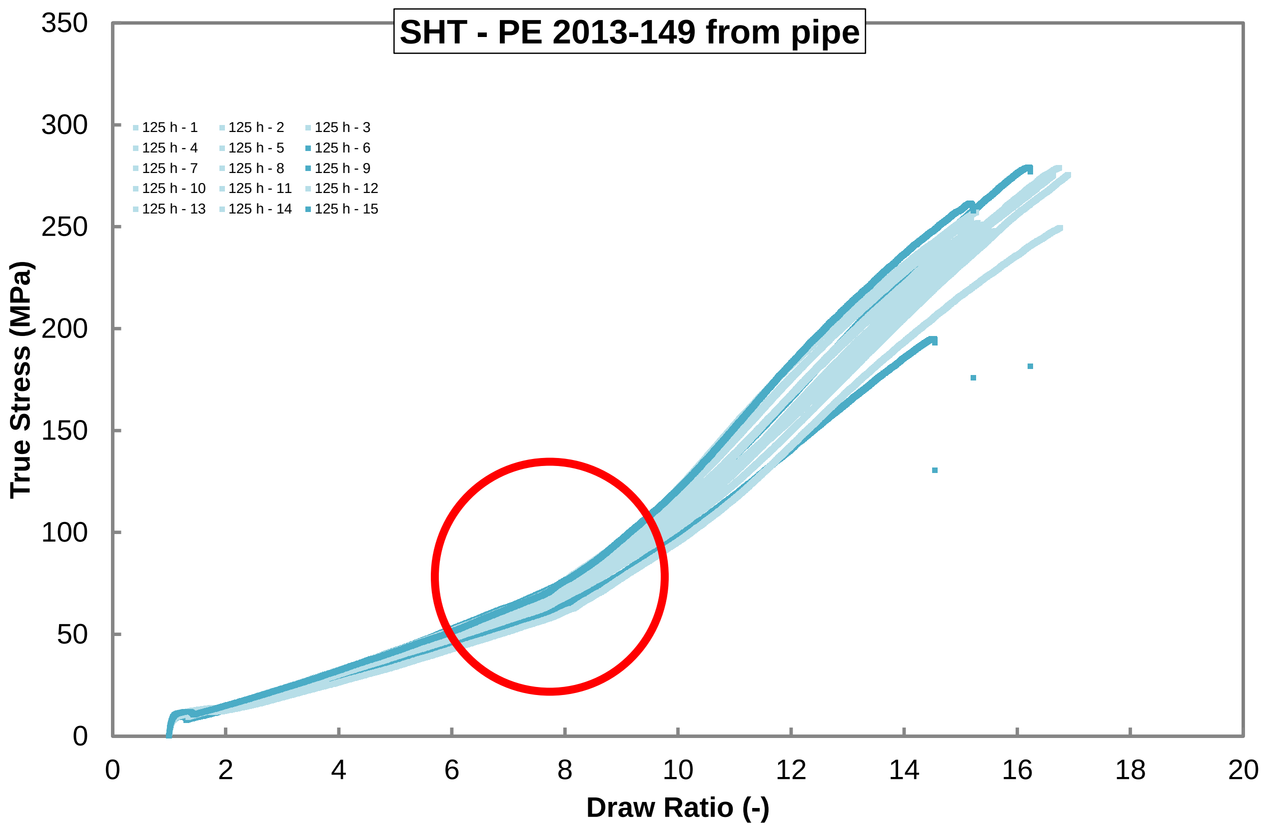
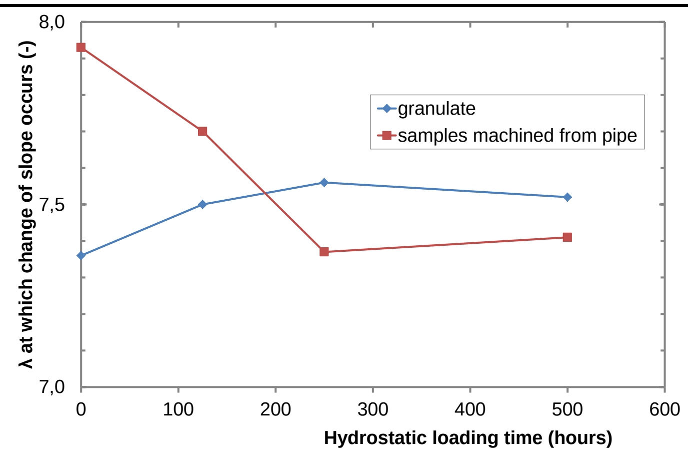


Figure 5. Raw data of samples prepared directly from PE 2013-149 that had been hydrostatically pressurised for 125 hours. Around λ=8 a change of slope can be observed (red circle).

Table 2. Average λ at which change of slope occurs in the SHT curve for various samples of PE 2013-149.

	λ at which change of slope occurs for	
Hydrostatic pressure time (h)	**granulate (-)**	**samples machined from pipe (-)**
0	7.36	7.93
125	7.50	7.70
250	7.56	7.37
500	7.52	7.41



An explanation for this change of slope is not yet available and it is possible that is it a merely a geometric effect that is independent of any mechanical degradation. It would however be interesting to investigate in future research if this result can be repeated or if it is just a coincidence.

CONCLUSIONS

Orientation appears to be of great influence on the strain hardening modulus when samples are machined directly from pipe. Recent axial and tangential samples had a higher strain hardening modulus, while radial samples had a lower strain hardening modulus when compared to re-melted samples.

The strain hardening modulus was not influenced by residual stresses. In the axial and tangential machining directions, there was almost no difference in strain hardening modulus between the inner side and outside of the pipe wall.

This knowledge was used to evaluate the residual quality of used first-generation PE pipes, which had also been mechanically degraded using hydrostatic pressure. The strain hardening modulus of samples machined directly from pipe in the axial direction remained constant and was independent of the imposed mechanical degradation. This result is similar to that previously found for re-melted strain hardening test samples prepared from granulate.

More research using pipes with longer hydrostatic loading times is needed to obtain better trend with samples machined in tangential direction. For now, no clear effect on the <Gp> is observable for the samples machined in tangential direction. More research is also needed to explain why the axial samples had a higher <Gp> than the samples prepared from granulate, while the tangential samples sometimes had a higher and sometimes a lower <Gp>.

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