A STUDY ASSESSING THE PERFORMANCE OF O-PVC IN PRESSURE PIPES

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Abstract

Bi-axially oriented PVC (referred to as O-PVC) pressure pipes have outstanding mechanical properties. Tensile strength and E-modulus increase significantly with the stretching ratio. On the other side, the elongation at break decreases with the draw ratio. O-PVC pipes have also much better impact resistance especially at low temperatures. Pressure and impact testing also demonstrated that the mechanical performance of O-PVC is not affected by notching.

Structural changes occurring in the PVC due to orientation were examined by thermal analysis and microscopy. It was shown that neither the total level of crystallinity nor the gelation levels were significantly different from conventional U-PVC. Further benefits of using O-PVC in pressure pipes are clearly demonstrated. The positive impact on environmental indicators such as energy and CO₂ release was also shown.

Keywords: pressure pipe, oriented Polyvinyl Chloride (PVC), stretching ratio, tensile properties, environmental indicator.

1. INTRODUCTION

In this paper, bi-axially oriented PVC (referred to as O-PVC) pressure pipe is compared against the most common alternatives, namely U-PVC and PE100. In literature, a lot of laboratory samples have been stretched and further studied (Brady¹, Gilbert and Hitt^{2,3,4,5,6,7,8}); whereas in the present paper industrial pressure pipes have been assessed.

It is generally accepted that PVC is a three-dimensional amorphous network of chains linked together by small crystallites. Typically in commercial S-PVC, the crystallinity accounts for 5% to 10% (Summers⁹, Gilbert et al^{4,8}, Hitt et al⁶, Kwon et al¹⁰) of the PVC structure. Gilbert et al⁴ has shown that the crystalline structure of PVC was an orthorhombic unit cell. It is also well known that there is a broad distribution of the crystal size and perfection that is reflected through the broad melting peak in Differential Scanning Calorimetry (DSC) covering temperature ranges from 110°C (230°F) till 230°C (446°F).

When stretching PVC, it behaves more like a semi-crystalline polymer than as an amorphous polymer since the performances in direction perpendicular to drawing are not decreased²; which is typically the case for amorphous polymers. With drawing, the crystallinity level is maintained whereas the order is improved. Stretching is causing crystalline orientation and alignment of the chains in the amorphous phase⁵. However, Gilbert⁶ has proven that it is the chain orientation in the amorphous phase that is mainly providing the increase in tensile strength.

The improvement of the mechanical properties of PVC by orientation is well known and has already been studied and reported widely (Anastassakis¹¹, Gilbert and Hitt^{2,3,4,6,7}, Bauer¹², Lowdon et al¹³, Marshall et al¹⁴, Chapman et al¹⁵, Holloway¹⁶).

The objective of this work was to investigate the draw effect and more specifically the draw ratio effect on mechanical properties such as tensile strength and impact strength. The influence of notching on the hydrostatic pressure and impact resistance of O-PVC was also assessed. Our goal was notably to develop a better understanding of the structure-property relationship. For this second part, we focused on the PVC materials. Differential Scanning Calorimetry (DSC), and observation of the fractures' surfaces with a 3D digital microscope allowed us to characterise the structure. DSC enabled us to assign the levels of gelation and of crystallinity, and to compare the thermal history across the wall thickness of U-PVC and O-PVC pressures pipes. An attempt was also made to characterise the orientation.

2. Experimental

2.1 Materials

This work investigated two different types of industrial PVC pipe materials: conventionally extruded U-PVC pipe and a bi-axially O-PVC supplied by Wavin (Hardenberg, NL). Both pipes have been produced using a K67 PVC resin from Shin-Etsu PVC (S67-04)(produced in Pernis, NL). In the present paper, when we make reference to the 'O-PVC', it means biaxially oriented PVC pipe manufactured by 'Wavin' in-line continuous process having a radial orientation of 1.9 and an axial draw ratio of 1.15.

2.2. Wavin process

In Wavin, this process is in use since 1996. The process is based on continuous extrusion. In-line orientation technology allows rapid production. Using standard U-PVC pressure pipe formulation, a pre-form is extruded by means of a specially designed die-head; which is then extended in both the hoop and axial directions over a mandrel (see Figures 1 and 2 below).





Figure 1: pre-form is pulled over the mandrel

Figure 2: orientation unit

The PVC pre-form must be oriented whilst the material is in a rubbery stage. Only in this case the pre-form is sufficiently flexible for the molecules to be oriented. This is the basic principle of in-line production of biaxially oriented pipe. To optimise mechanical properties, a temperature close to 90°C was proven to be the optimum

temperature for stretching since PVC has a peak elongation temperature of 90°C (Hitt⁷). Because of bad heat conductivity of polymers, it is very difficult but very important to heat the polymer to a uniform temperature during stretching. It should be noted that after stretching the greatest cooling rate will preserve the most orientation. After orientation of the pre-form over this mandrel, the diameter of the final biaxial pipes has almost doubled compared with the original pre-form.

As it is not possible to sample the pre-form pipe in production, a sample of oriented pipe is reverted back to pre-form by placing it in an oven at 150°C and the dimensions are measured before and after reversion. Detailed calculations to determine the radial and axial stretching levels have been reported earlier (Holloway¹⁶).

2.3 Mechanical testing

For assessing radial strength, test specimens were hoops, loaded by the split disk method as in ASTM D2290. For evaluating the axial strength, dumbbells have been cut in the pipe along the extrusion direction. Tensile testing was performed according ISO 527. Hydrostatic pressure resistance was measured according to the ISO 1167 norm. Impact test carried out was the staircase weight falling test performed according to ISO 11173 method. The test was carried out from temperatures ranging from $-20^{\circ}C$ ($-4^{\circ}F$) to $20^{\circ}C$ ($68^{\circ}F$).

2.4 Differential Scanning Calorimetry

2.4.1 Sampling

In order to study the morphology across the pipe thickness, specimens have been sampled as shown in Figure 3.



Figure 3: Macrotome sampling done across pipe section: the following layers have been extracted from inside surface to external surface: inside surface (I), inside-middle (IM), external-middle (EM) and external surface (Ex); respectively.

2.4.2 DSC

The 'macrotomed' samples, weighing approximately between 10 to 20 mg were placed in sealed aluminum DSC pans with holes to ensure that HCl gas could escape during the test. DSC tests were performed with a Perkin Elmer Diamond DSC system using a heating rate of 20°C/minute from 35°C to 240°C. The DSC cell was purged with dry nitrogen gas to create an inert atmosphere and to liberate HCl gas. The DSC was calibrated using Indium and Zinc standards.

3. Results and discussion

The effect of stretching PVC pipes in both radial and axial directions on tensile properties has been thoroughly investigated. First of all, the shape of the tensile

curve is modified when the PVC is stretched, regardless the stretching direction: radial or axial.



As can be seen on Figure 4, the tensile curve of U-PVC follows the typical pattern of amorphous polymeric materials. Initially we find a viscoelastic, time-dependent, response that is considered as fully reversible. For small loads the material behaviour is linear viscoelastic, while with increasing load the behaviour becomes progressively nonlinear. At the yield point the deformation becomes irrecoverable since stress-induced plastic flow sets in leading to a structural evolution which reduces the material's resistance to plastic flow: *strain softening*. Finally, with increasing deformation, molecules become oriented which gives rise to a subsequent increase of stress at large deformations: this is *strain hardening*.

Comparatively, O-PVC will break closer to what we have defined as the yield point (YP). The yield point is located at the cross section between the two tangents following the two different slopes of the tensile curve after the linear viscoelastic deformation (Figure 5). Orientation will increase resistance to crazing, suppress strain softening and necking of the samples. Only limited strain hardening - observed here between the Yield point and fracture - is still present and depends on the stretching level. Similar tensile curves have been reported earlier when an orientation level of 1.75 was at least applied (Bauer¹²). According to Bauer¹², this means that local yielding required for the fibrils formation and consequently craze initiation are not longer possible in O-PVC. Therefore O-PVC is much more likely to exhibit a ductile failure mode rather than a brittle failure.



| | Yield | Tensile | | | |
|------------------------------|-----------------|-----------------|--|--|--|
| | strength MPa | strength MPa | | | |
| U-PVC | 48 ± 2.4 | 49 ± 2.5 | | | |
| O-PVC ⁽¹⁾ | 70 ± 3.5 | 81 ± 4.1 | | | |
| PE100 | 25 ± 1.3 | 35 ± 1.8 | | | |
| (1) radial stret. factor 1.9 | | | | | |

Figure 6: Yield and Tensile strengths increase with stretching Tab



When comparing Figure 4 to Figure 5, it is also clear that the stress level achieved with O-PVC is much higher than that achieved with U-PVC. This characteristic will be discussed in more details below.





Figure 7: Strength increase is similar, whatever stretching type

Table II: Elongation at break

Both yield strength and tensile strength improve substantially with axial stretching ratio (Figure 6). The yield strength increased from 48 MPa for non oriented PVC to 77 MPa for an axial stretching ratio of 3.4; and the tensile strength increased from 49 MPa to 133 MPa for the same stretching level. Figure 7 shows that the stretching direction - would it be radial or axial – does not affect the tensile resistance performance. Considering O-PVC pipe compared to U-PVC pipe specifically, its tensile strength improved by at least 50%. This strength increase for a draw ratio of 1.9 is even higher than earlier predicted (Anastassakis¹¹, Gilbert and Hitt^{2,3,7}, Chapman¹⁵, Holloway¹⁶).



Figure 8: Tensile Strength not affected by diam. Fig. 9: Elongation (ε_b) decreases with stretching

Table I lists typical values of strengths including that of PE100 for comparison sake. As can be seen on that Table, tensile strength of O-PVC is higher than that of U-PVC, in its turn higher than that of PE100. Figure 8 shows that tensile strength remains unchanged whatever the pipe diameter.

Axial stretching is accompanied with a decrease in elongation at break that is levelling off for stretching ratios above 2.0 as illustrated in Figure 9. Table II provides standard values of elongation at break for PVC and PE materials. Typically O-PVC will break with an elongation at break of 60%. This reduction of elongation at break with stretching is aligned with most findings (Gilbert^{2,3}) and can be explained as follows: since the chains have already been oriented and extended in the rubbery

stage (close to T_g), their extensibility at room temperature will be further limited. Therefore elongation at break of O-PVC is reduced.



Figure 10: E-Modulus increases with stretching



As expected and also reported earlier (Brady¹, Chapman¹⁵), the E-modulus increases significantly with the stretching ratio as illustrated in Figure 10. The change in E-modulus can be understood in terms of chain alignment that occurs during mechanical stretching. It seems however that a minimum stretching is required to impact positively the modulus; on the other side, the modulus is levelling off at stretching levels close to 3.5.

The following equation was fitted to the E-modulus data:

 $E = (2900 - 1922) + \left[\frac{2 \times 1922}{1 + \exp(-1.6 \times \max(0, SR - 1.3))}\right]$ where SR is the stretching ratio (1)

The presence of an aligned network would be expected to improve impact resistance. As can be seen in Figure 11, impact strength of O-PVC is extremely difficult to measure because of the large energies required to produce fracture. It was indeed not possible to break the O-PVC with the loads commonly applied and therefore we have indicated that the energy needed to break the O-PVC pipes was higher than 370 Nm (arrow up). Figure 11 proves that the impact resistance of O-PVC is better than that of U-PVC, at all the temperatures tested. The gap is even bigger at temperatures below 0°C, emphasising the outstanding impact resistance of O-PVC. This is fully in line with previous reports highlighting the benefits of orientation on impact resistance (Anastassakis¹¹, Bauer¹², Gilbert3, Lowdon¹³, Holloway¹⁷, Chapman¹⁵). In most studies, it was shown that for a draw ratio of 2 x the impact strength was more than the double that of the undrawn material.

Figure 12 confirms that the impact strength of O-PVC is significantly better that of U-PVC. PVC-A, that is a PVC modified with acrylic modifier also exhibits excellent impact resistance (no failure, arrow up). Another interesting characteristic of O-PVC is that its impact performance is not affected by notching. Notched PVC-A showed however an intermediate impact resistance between U-PVC and O-PVC.



Hydrostatic pressure testing highlighted too that O-PVC pressure resistance was not affected by notching. Measurements performed on notched O-PVC pipes were indeed in line with those made on unnotched pipes as illustrated in Figure 13.



Figure 13: Notching does not affect pressure resistance of O-PVC

In order to understand the reason why the impact performances of O-PVC were improved and why notching did not affect mechanical performance such as hydrostatic pressure or impact resistance, we have investigated the O-PVC structure. Therefore, we have examined fractures' surfaces with a 3D digital microscope. In the case of O-PVC, a layered structure was observed as shown in Figure 14; which is not the case with U-PVC (see Figure 15).



Figure 14: O-PVC layered structure after break

Figure 15: U-PVC structure after break

The crack growth mechanism is slow down thanks to the reduction in stress concentration at the crack tip as the crack passes through a layer. Two mechanisms

are combined when a crack reaches a layer: the crack is blunted and further divided is small cracks on the one hand and, energy is dissipated as heat on the other hand. The layered structure in O-PVC explains why even in the case a crack has been initiated, it will not further propagate easily. In physical terms, additional energy is needed for crack growth in ductile materials when compared to brittle materials. Previous studies have also demonstrated that the extraordinary resistance to radial crack development in O-PVC pipes¹³ resulted in improved impact strength¹⁵.

Crystallinity and gelation have been assessed through the pipe thickness for both U-PVC and O-PVC by means of the Differential Scanning Calorimeter (DSC) technique. Sampling is described in the experimental section (Figure 3). An overview of the results is given in Table IV; where each measurement was made by averaging results from at least two specimens. DSC thermograms showed a glass transition temperature (Tg) around 86-87°C, followed by two endotherms ranging from 110°C to 220°C. As can be seen on Table IV, processing temperatures and gelation levels calculated according to equation (2) were found to be similar for both pipes. These results are also in line with Wavin data. Some inhomogenities - that can not be explained - have however been found within the pipe thickness of the U-PVC pipe.

 $Gelation \, level [\%] = \frac{\Delta H_A}{\Delta H_A + \Delta H_B}$

where ΔH_A : secondary cristallinity eq(2) ΔH_B : primary cristallinity

| Тр | I | IM | EM | Ex | Average |
|--|-------|-------|-------|-------|---------|
| | °C | | | °C | |
| U-PVC | 191.1 | 188.2 | 188.6 | 188.3 | 189.1 |
| O-PVC | 187.8 | 189.3 | 188.1 | 188.2 | 188.4 |
| Gelation level | | IM | EM | Ex | Average |
| $\Delta H_A / (\Delta H_A + \Delta H_B)$ | % | | | % | |
| U-PVC | 80.9 | 64.6 | 76.7 | 85.4 | 77 |
| O-PVC | 68.3 | 72.3 | 70.0 | 72.1 | 71 |
| Total crystallinity | I | IM | EM | Ex | Average |
| level ($\Delta H_A + \Delta H_B$) | J/g | | | J/g | |
| U-PVC | 5.0 | 2.9 | 5.9 | 8.2 | 5.5 |
| O-PVC | 5.7 | 6.0 | 5.4 | 4.3 | 5.4 |

Table IV: Processing T° (Tp), gelation level and total crystallinity obtained by DSC (coding, see Fig.3)

When observing the DSC thermograms, we noticed the presence of a Cold Crystallisation Peak (CCP) at the external surface of the U-PVC that can be explained by the fact that this surface is quenched when exiting the extruder die. All layers of O-PVC exhibit however a smaller CCP due to the fact that after cooling down the pipe is reheated for stretching. This CCP might also be attributed to the orientation as described by Fillot¹⁸ but this is not trivial in our case. The relaxation peak - also named superheating endothermal peak – was observed to be bigger for the internal surfaces than for the external ones of both U-PVC and O-PVC pipes; reflecting less free volume at the internal surfaces. The smaller quantity of free volume might be due to the slower cooling rate imposed to the internal surfaces; hence allowing a tighter packing of the PVC chains. We can also pinpoint that the differences in CCPs and relaxation peaks within the pipe thickness are larger for the U-PVC than for the O-PVC and this may be due to the very different heat pattern imposed to the internal and external surfaces of U-PVC.

4. Benefits for pressure pipes

O-PVC is a very strong and tough material. Compared to U-PVC, it exhibits improved tensile strength, higher impact resistance, slower crack growth due to slow crack propagation, mechanical properties that are not impaired by notching.... Thanks to these characteristics, O-PVC has a higher MRS (Minimum Required Strength) and a lower Safety factor (C=1.6 instead of 2 for U-PVC) than U-PVC, which results in a much higher HDS (Hydrostatic Design Stress) than U-PVC. As a consequence, a thinner O-PVC pipe might be produced for the same pressure class. An example is provided in Table V.

5. Environmental indicators

Some environmental indicators, such as CO_2 or energy indicators, were compared for pressure pipes made from different materials¹⁹ (see Figure 16). It came out that the total CO_2 released corresponding to 3 m pipe of U-PVC was similar to that of PE100. O-PVC releases however 80% of the CO_2 compared to above 2 pipes (thanks notably to the weight and thickness reduction). Most of the CO_2 emissions come from the use phase of the pipe (50 years). Differences during the use-phase are mainly caused by differences in internal diameter and roughness/waviness of the inside of the pipe. The O-PVC having for example a greater internal diameter – for the same external diameter – will result in lower pumping energies than the U-PVC. Similar conclusions could be drawn for the energy release.



| DN160/ PN12.5 | | U-PVC | O-PVC | | |
|-------------------------------|-----|-------|-------|--|--|
| MRS | MPa | 25 | 45 | | |
| С | - | 2 | 1.6 | | |
| HDS | MPa | 12.5 | 28 | | |
| SDR ^(a) | - | 21 | 46 | | |
| e ^(b) | mm | 7.6 | 3.5 | | |
| Weight saving | | | 53% | | |
| (a): Standard Dimension Batio | | | | | |

(a): Standard Dimension Ratio (b): Wall thickness



6. Conclusions

Thanks to above results, the excellent performance of biaxially oriented pipe named O-PVC was confirmed. Compared to traditional U-PVC pipes, O-PVC pipes have a higher tensile strength (+50%), impact strength (at least doubled) and pressure resistance and impact properties are not negatively affected by notching. These results confirmed that the influence of the draw ratio was similar on industrial pressure pipes than on previously studied laboratory samples.

In terms of morphology, DSC results did not highlight significant differences in terms of gelation or crystallinity level between O-PVC and U-PVC. The different thermal

history of the two pipes was reflected in DSC through the appearance of a clear Cold Crystallisation Peak (CCP) at the external surface of the U-PVC caused by the quenching of that surface and by a relaxation peak at internal surfaces of both pipes highlighting inside surfaces with less free volume in view of the lower cooling rates.

When used in pressure pipe systems/installations, an oriented pipe with half thickness of a U-PVC might be produced fulfilling all criteria for a given pressure class. This is achieved thanks to a higher Minimum Resistance Strength (MRS) of O-PVC combined with a lower safety factor (C). In this paper tensile testing has shown that a different mechanism of rupture is taking place in the case of O-PVC compared to the undrawn material, highlighting the absence of strain softening and crazing in the case of O-PVC. Observation of the fractures' surfaces confirmed the presence of a layered structure explaining the slower crack's propagation accounting for improved impact resistance.

In terms of environmental performance, O-PVC releases 20% less CO₂ and energy than conventional plastics used for the same application; i.e. U-PVC and PE100.

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