

MORPHOLOGICAL CONSIDERATIONS ON SLOW CRACK GROWTH IN POLYETHYLENE PIPES UNDER DIFFERENT PRODUCTION CONDITIONS

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KEYWORDS

polyethylene, slow crack growth, morphology, processing

ABSTRACT

In this work the influences of different production conditions on physical properties of polyethylene pipes were investigated. Pipes of the dimension dn 50x4,6 mm (dn 1.97x0.18 in) were produced by conventional extrusion and injection moulding as well as compression moulding of sheets of the same thickness. The used polyethylenes were bimodal PE 100 ones with butane copolymerisation which are used in the field for the transport of gaseous fuels under pressure.

It was shown, that the injection moulded pipes show significant higher times in 2NCT results (of about 2-3 times) and in internal pressure tests (of about 9-11 times) compared to the extruded pipes of same dimension. Investigations of the fracture surfaces show a different SCG-failure behaviour. The injection moulded pipes show a zone structure with different layers. In instrumented Charpy impact tests this behaviour could be also found, especially at higher test temperatures.

Morphological the extruded pipes show the well known increasing gradient of crystallinity from the outer- to the inner-pipe wall. The injection moulded pipes show a homogeneous crystallinity with lower crystallinities on the outer and inner side for some polyethylenes.

INTRODUCTION

Samples were tested in 2-Notch Creep Test (2NCT), a test geometry which makes it possible to achieve fracture results including the sensitive regions of the outer- and inner-pipe wall region with the highest internal stresses and orientations. Furthermore notched Charpy impact tests were performed and fracture mechanics values calculated.

Internal pressure tests were done on whole pipe specimens. For correlations with the morphology of the pipes crystallinity and lamellar thickness distributions were calculated based on DSC experiments.

2NCT

Slow crack growth (SCG) was measured by means of an adaptive to the Full Notch Creep Test (FNCT): the 2-Notch Creep Test (2NCT). This notching geometry offers the advantage, that the sensitive zones of samples, made of pipe specimens, are conserved: the inner- and outer-pipe wall layers (1-4,16,17). This test geometry makes it possible to achieve fracture results including the sensitive regions with the highest internal stresses and orientations.

The results indicate a better SCG-behaviour of the injection moulded specimens compared to the extruded ones at a factor of about 2-3 (Fig. 1). The fracture surfaces, shown in Figure 2, clearly indicate a zone structure with different layers for the injection moulded specimens, which are responsible for the higher failure times.

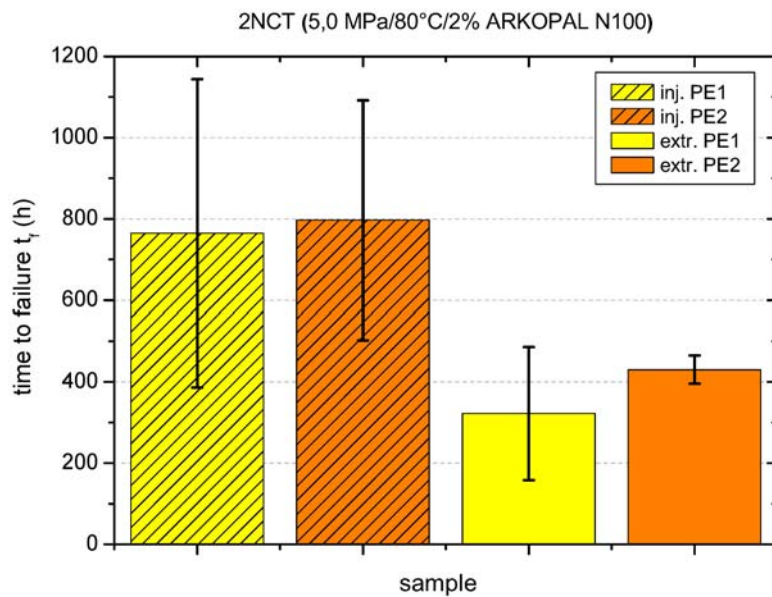
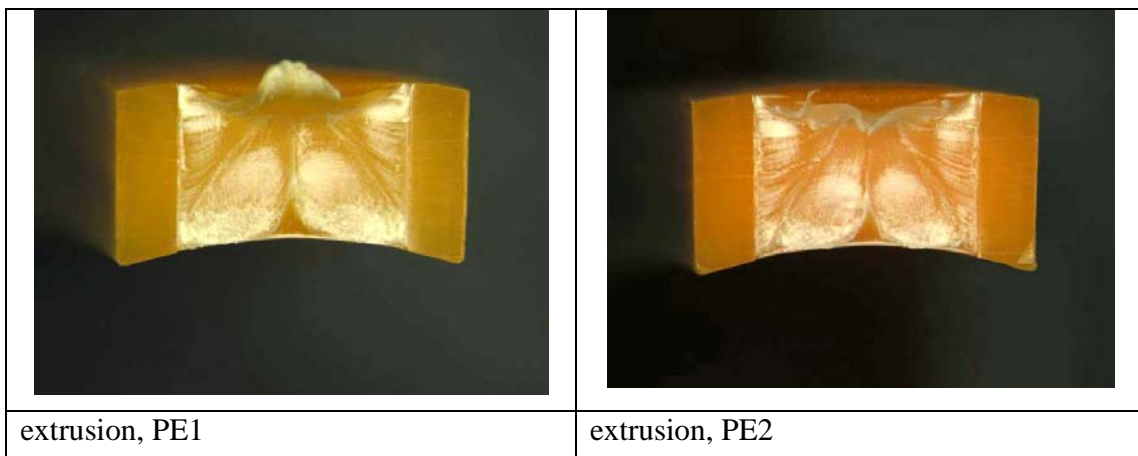


Fig. 1: 2NCT results (5,0 MPa at 176°F) of the injection moulded and extruded pipe specimens



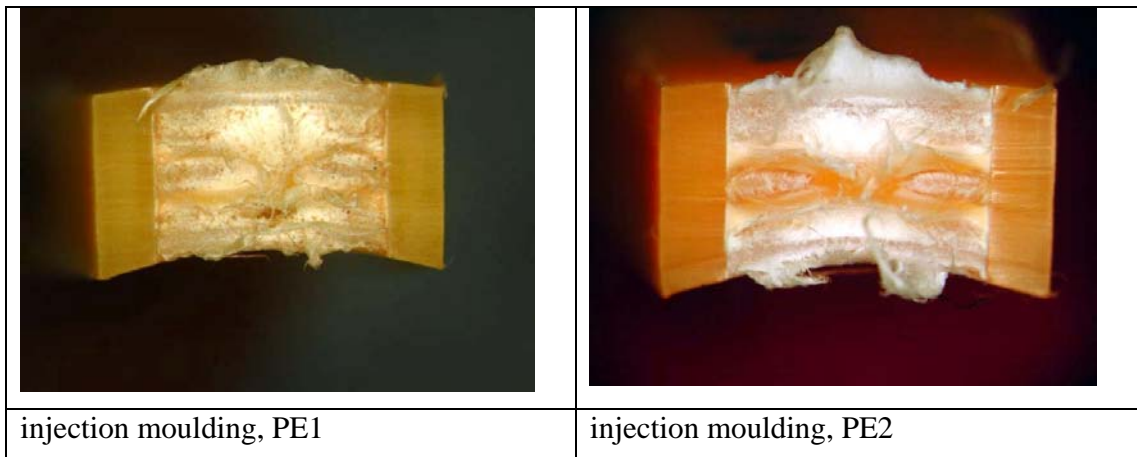


Fig. 2: 2NCT fracture surfaces of the injection moulded and extruded pipe specimens

Notched Charpy Impact Tests

Notched Charpy impact tests were performed on an instrumented 4J pendulum. Specimens were notched with a razor blade along the pipe wall thickness on one side (analogue the 2NCT experiments but only with one notch for 3-point bending tests, 3PB). Tests were performed at 0°C, 23°C and 80°C (32°F, 73°F and 176°F) and fracture mechanic values calculated, which already have been successfully applied for polyethylenes (5-9).

The results are shown in Figures 3 and 4. The injection moulded pipes show a stronger increasing toughness with higher test temperatures, which can be seen in Figure 3 for notched Charpy impact strength (a_{cN}) and J-integral according to Merkle and Corten (J^{MC}). This causes a lower decreasing gradient of stress intensity factor K_{IC} with increasing temperature (Fig. 4).

The fracture surfaces of the samples are shown in Figures 5-8.

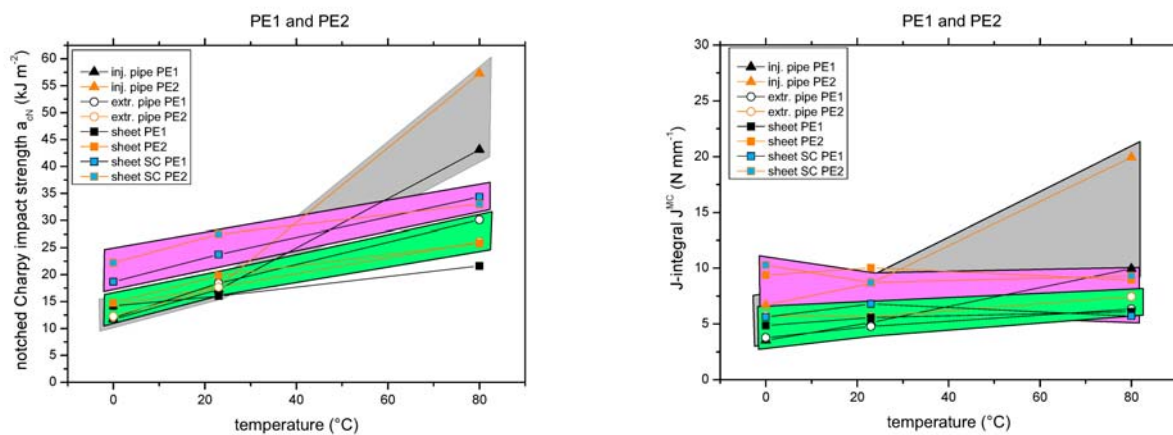


Fig. 3: Notched Charpy impact tests: evaluation of the notched Charpy impact strength a_{cN} and the J-integral J^{MC}

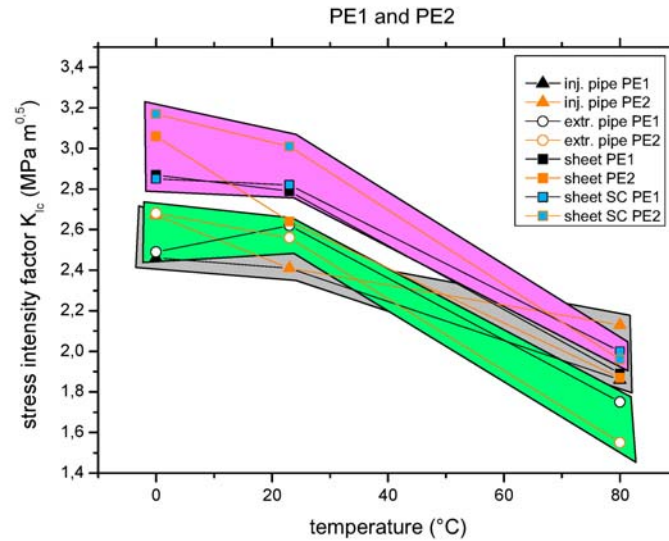


Fig. 4: Notched Charpy impact tests: evaluation of the stress intensity factor K_{IC}

Figures 5 and 6 show fracture surfaces of compression moulded sheets (normal and slow cooled) which were evaluated for comparison with the pipes. The sheets show a homogeneous fracture behaviour along the thickness of the sheet (only weak plastic zones). With higher test temperature the ductility (coarser fibrillation, whitening of the fracture surfaces) increases as expected.

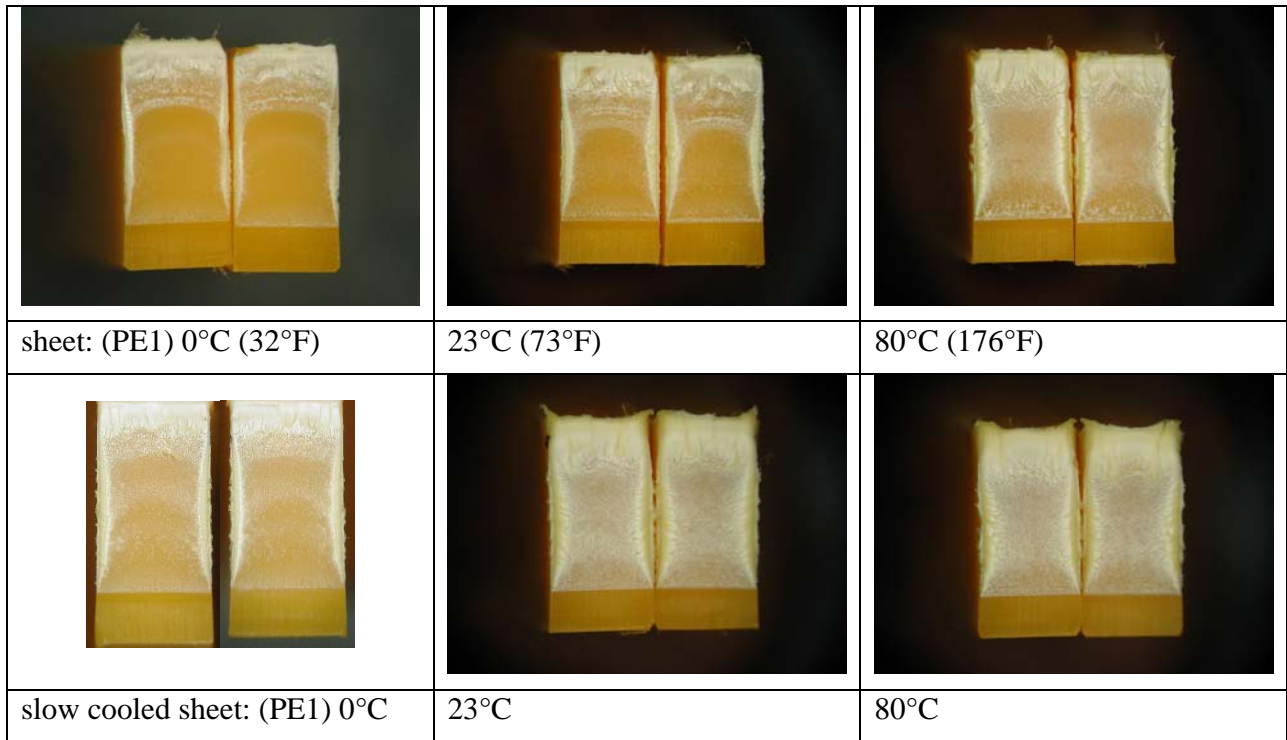


Fig. 5: 3PB fracture surface of PE1, sheet samples at 0°C, 23°C and 80°C

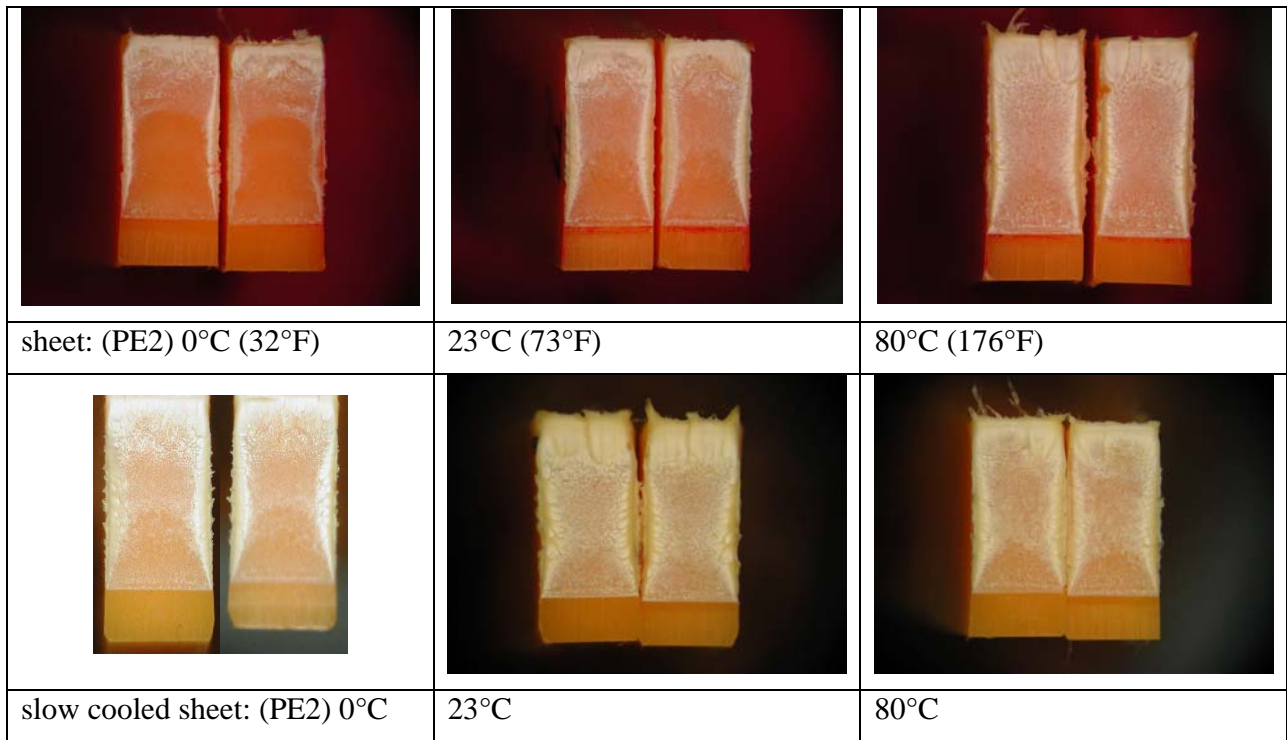


Fig. 6: 3PB fracture surface of PE2, sheet samples at 0°C, 23°C and 80°C

The fracture surfaces of the pipe samples show completely different fracture behaviour. The extruded pipe specimens show the conventional behaviour with the usual test temperature dependency (increasing ductility). The injection moulded pipe specimens show a special kind of zone structure, which increase with higher test temperatures. Formation of big ductile bands in the outer- and inner-pipe wall zone can be found. This is caused by the different processing conditions (injection moulding with ring runner vs. extrusion) where different shearing- and cooling-conditions are present.

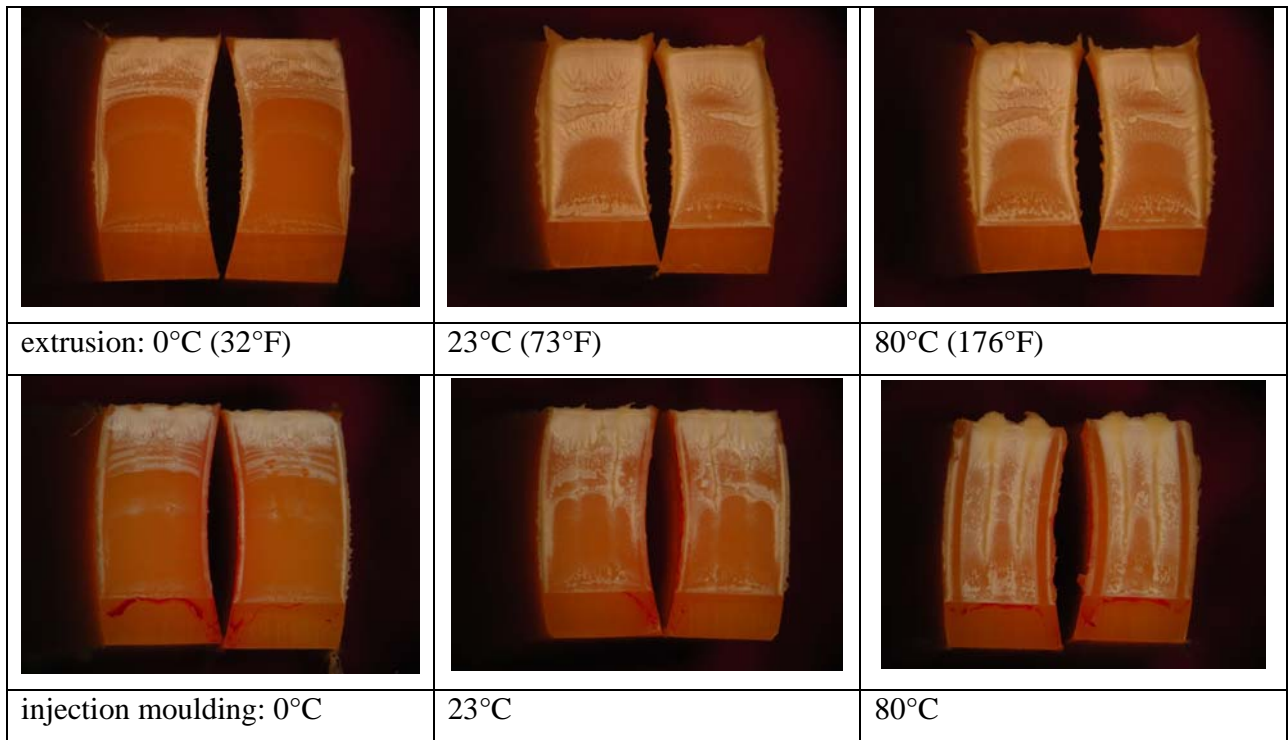


Fig. 7: 3PB fracture surface of PE1, extruded and injection moulded pipe samples at 0°C, 23°C and 80°C

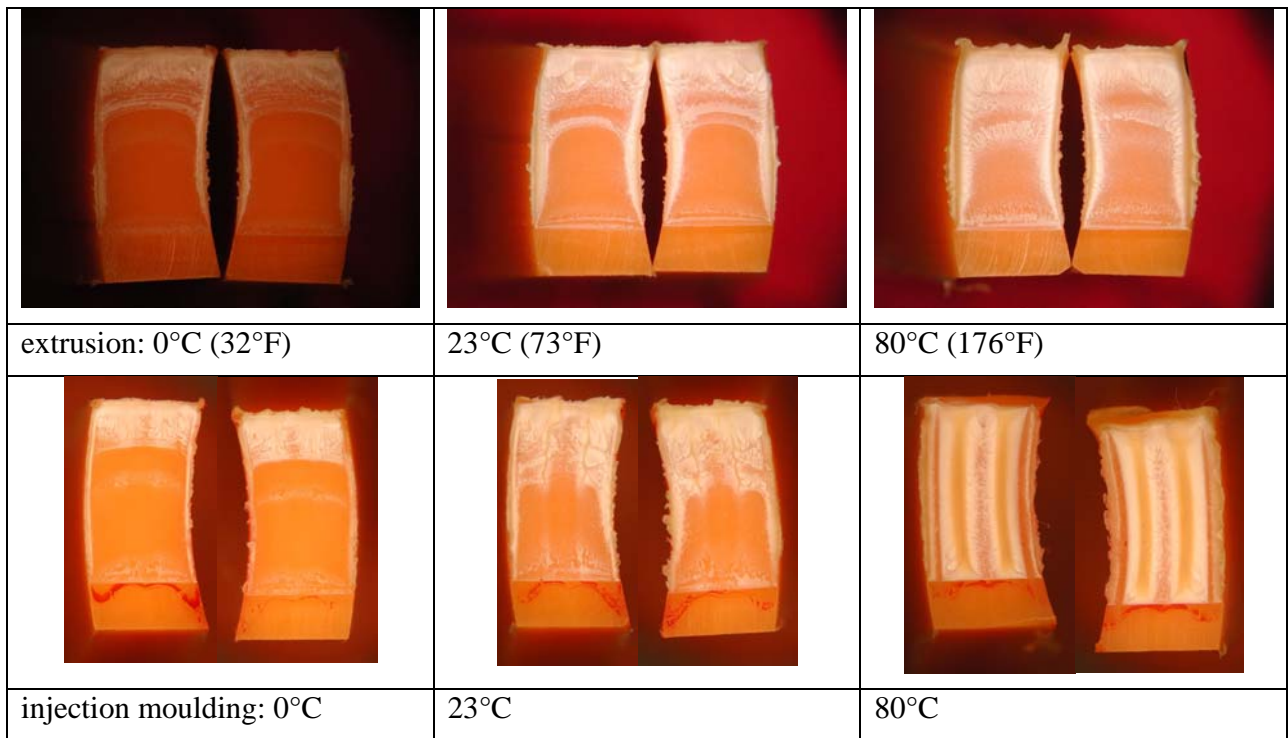


Fig. 8: 3PB fracture surface of PE2, extruded and injection moulded pipe samples at 0°C, 23°C and 80°C

Internal pressure tests

Internal pressure tests in water were performed at different stress levels at 20°C (68°F) and 80°C (176°F) on whole pipe specimens (10). The results are shown in Figure 9. They indicate higher failure times for the injection moulded pipe specimens compared to the extruded ones at a factor of about 9-11. This highlights the extremely different long term behaviour, as already shown in 2NCT, caused by the different processing conditions.

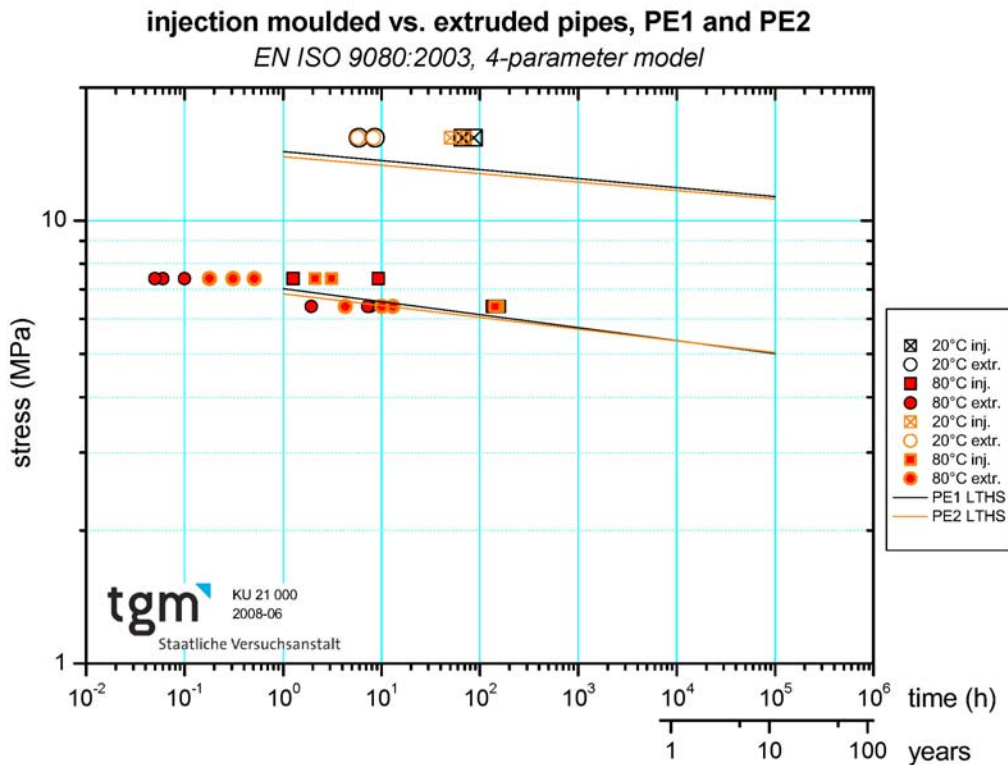


Fig. 9: internal pressure test results of the pipe specimens

MORPHOLOGICAL INVESTIGATIONS

Crystallinity was calculated of the DSC measurements for the pipe wall regions (outer, middle and inner zone) to show the morphology of the different processing conditions. Based on the DSC measurements also the lamellar thickness distributions of the crystalline thickness L_c for the pipe walls were calculated and correlated with the fracture results obtained in this work.

Crystallinity

Crystallinities for the extruded pipes show the typical behaviour with an increase to the inner pipe wall zone (indicated green). The injection moulded pipe specimens show a more homogenous distribution along the pipe wall. For reference compression moulded sheets were evaluated of the same batch and thickness of the pipe specimens. They show a homogeneous crystallinity (no gradient) which increases with lowering of the cooling conditions (sheet SC...slow cooled).

These results indicate the different morphology based on the processing conditions, which results in significantly different results of the slow crack growth behaviour and internal pressure results (11).

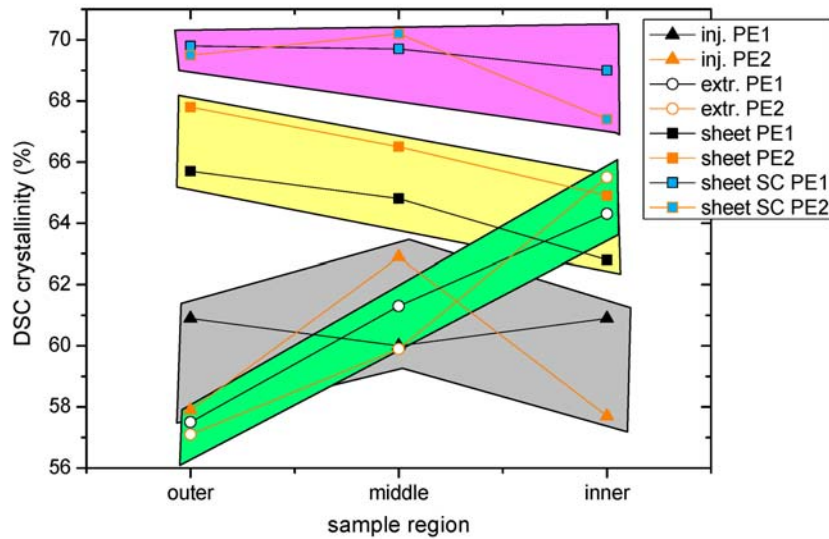


Fig. 10: Crystallinity of the pipe wall and sheet specimens

Lamellar thickness distribution

The lamellar thickness distribution was calculated based on the DSC results (12-14). The results are shown in Fig. 11 for the extruded and injection moulded pipe zones. There is no significant difference evident.

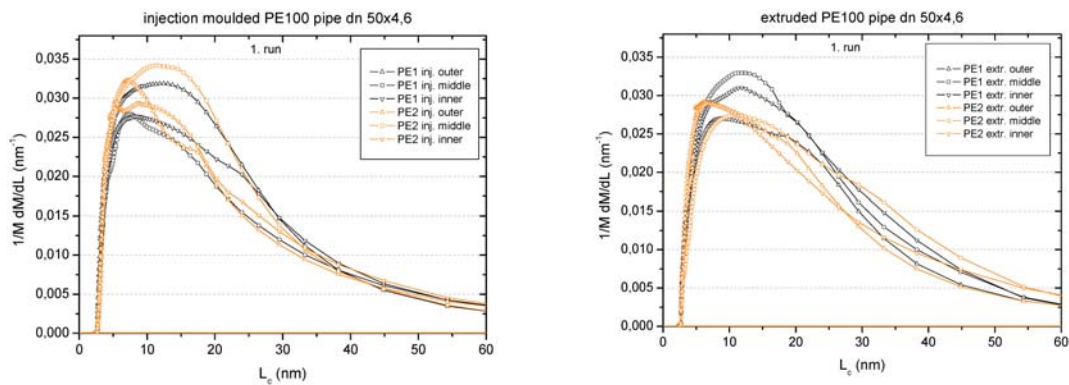


Fig. 11: Lamellar thickness distributions for the injection moulded (left) and extruded pipe specimens (right)

CONCLUSIONS

It was shown, that the injection moulded PE100 pipes show significant higher times in 2NCT (of about 2-3 times) and in internal pressure tests (of about 9-11 times) compared to the extruded pipes of same dimension (dn 50x4,6 mm, dn 1.97x0.18 in).

Investigations of the fracture surfaces show a different SCG-failure behaviour. The injection moulded pipes show a zone structure with different layers (bands in the inner and outer region). In instrumented Charpy impact tests this behaviour could be found too, especially at higher test temperatures.

Morphological the extruded pipes show the well known increasing gradient of crystallinity from the outer to the inner pipe wall (15-18). The extruded pipes show a homogeneous crystallinity with lower crystallinities on the outer and inner side for some polyethylenes.

This indicates a different long term behaviour of the pipes which is strongly depended on the processing conditions (17-18). Therefore care has to be taken when evaluating long term behaviour of materials, especially ones for fittings were pipes could have been injection moulded for ISO 9080 classification. This results have to be taken into account for further revisions of the concerning standards as well when comparing results of samples based on divergent processing conditions.

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