Short time test methods to predict long time slow crack growth behaviour?

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ABSTRACT

Determination of slow crack growth (SCG) on modern PE compounds, especially ones with hexene copolymerisation (PE100 RC) needs big amounts of times. Accelerated tests like the Full Notch Creep Test (FNCT) and the 2-Notch Creep Test (2NCT) can last some 1000 hours to generate brittle failure times. In this work two short time test methods which have been published in literature recently were evaluated.

Essential work of fracture (EWF) measurements and tensile tests at 80°C were done and the results compared with SCG-failure times and long time hydrostatics strength (LTHS) results. There seem to be some capabilities of both methods to give a first ranking for development work or quality control checks. The results and different possibilities of these methods are outlined in this work.

INTRODUCTION

The slow crack growth (SCG) behaviour of polyethylene is measured by different laboratory tests on whole pipe specimens as well as on test-specimens made out of pipes. Because SCG needs big amount of time these tests are accelerated by means of higher temperatures and particularly the use of wetting agents.

One method to measure the resistance to SCG is the Full Notch Creep Test (FNCT) [1,2]. An adaptive method with only two notches in opposite direction across the pipe wall is the 2-Notch Creep Test (2NCT). This special kind of notching conserves the outer- and inner-pipe wall zones, which contains the regions of maximal residual stresses and skin-core structures obtained during extrusion process [2-4].

Modern PE100 compounds with hexene copolymerisation (PE100 RC) can last some 1000 h in this accelerated test method until fracture.

Aim of this work was to give an overview of short time test methods and their capabilities to predict long time behaviours of PE pipe materials.

EXPERIMENTAL

In this study the essential work of fracture (EWF) method as well as a tensile test at 80°C was evaluated. Both methods have been published in literature in the last years to predict long time behaviour of PE pipe materials [5-7].
**Essential work of fracture (EWF)**

The Essential work of fracture (EWF) method uses a thin compression moulded specimen (about 0.5 mm) with two notches in opposite direction (double edge notch tensile, DENT specimen, see Fig. 1).

Specimens with different deep notches are prepared and short time tensile tests at 23°C carried out. Different stress-strain curves depending on the ligament length \( l \) are measured. Out of the tensile test results the parameters

- \( w_e \) ...essential work of fracture and
- \( \beta w_p \) ...non-essential work of fracture including the shape factor \( \beta \) are calculated.

This plastic work dissipation factor \( \beta w_p \) can be used to evaluate the ductile behaviour of PE materials.

![Diagram of DENT specimen](image)

**80°C tensile test**

The 80°C tensile test method uses thin compression moulded specimen (0.5 mm) which are tested unnotched. The tensile test in this method is performed at 80°C and 10 mm/min. Out of the stress-strain curve the true stress-strain curve is calculated. The last part of this true stress-strain curve represents the strain hardening zone of the test sample. Out of this last part the so-called strain hardening modulus \( G_p \) can be evaluated (Fig. 2) [6,7]. This value can be correlated to the resistance of craze fibrils under creep conditions [7].

![Diagram of true stress-strain curve](image)

Fig. 1: Principle of the EWF-method and evaluation of the results

Fig. 2: True stress-strain curve
**DSC analysis**

The investigated samples were analysed also by DSC and the crystallinity of the samples evaluated. The obtained morphological parameters are shown together with the results of the short time fracture test methods.

**RESULTS - Correlations with long time properties**

**σ\(_{LPL}\) classification for 50 years and 20°C**

The EWF results can be used to predict the long time ductile behaviour of PE-materials. This can be seen, if we consider the results of the long time hydrostatic strength (LTHS) tests on pipes (ISO 9080 extrapolation). The lower prediction limit (σ\(_{LPL}\)) values for 50 years at 20°C seem to correlate with the β\(_w\) results of the EWF measurements (Fig. 3).

The σ\(_{LPL}\) values also show a dependency on the crystallinity. The PE80 materials show a lower behaviour in both values. PE100 and PE100 RC materials show the corresponding same σ\(_{LPL}\) values but different groupings in the crystallinity levels (Fig. 4).
**Fig. 3:** Dependency of the $\sigma_{LPL}$ values on the non-essential work of fracture $\beta w_p$

**Fig. 4:** Dependency of the $\sigma_{LPL}$ values on the crystallinity $X_c$

**Slow crack growth behaviour (SCG)**

To predict long time slow crack growth behaviour the EWF method despite can not be used. There is no correlation between the essential work of fracture ($w_e$) and the plastic work dissipation factor ($\beta w_p$) evident with the failure times in 2NCT.

The results of the 80°C tensile test were evaluated together with the 2NCT results. It can be seen that the calculated Gp parameter describes the 2NCT failure times very well. With the
some hours lasting Gp experiment 2NCT failure times in the range of some 1000 h can be predicted with this method (Fig. 5).

The Gp behaviour as function of the crystallinity shows a similar tendency as the $\sigma_{\text{LPL}}$ one above. The PE80 types show a lower behaviour in both values (Gp and $X_c$). For PE100 and PE100 RC the Gp values are in a higher range in different groupings for $X_c$ (Fig. 6).

![Fig. 5: Dependency of the 2NCT values on the Gp parameter](image)

![Fig. 6: Dependency of the Gp parameter on the crystallinity](image)
CONCLUSIONS

The following statements can be concluded from the results obtained in this work:

- The EWF method seems to be able to predict the $\sigma_{LPL}$ behaviour of PE pipe materials.
- With EWF no prediction about the SCG behaviour can be given.
- The 80°C tensile test method allows a first prediction of the SCG behaviour of PE pipe materials. The groups PE80, PE100 and PE100RC can be distinguished in the ranking.
- A calculation of long time SCG values for PE100 and PE100 RC based only on 80°C tensile test results seem to be critical because there is too less difference in the Gp values.
- Both methods seem to be applicable to give a first ranking for development work or quality control checks.

REFERENCES

2. ÖNORM EN 12814-3 (2005-10-01), Testing of welded joints of thermoplastics semi-finished products – Part 3: Tensile creep test