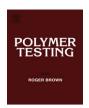
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Test method

The applicability of the Pennsylvania Notch Test for a new generation of PE pipe grades

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ABSTRACT

The failure mode termed long time brittle failure (due to slow crack growth – SCG) limits the lifetime of plastic pipes. There are now two main accelerated tests (PENT and FNCT) that enable us to estimate the lifetime of HDPE used in plastics pipes. For unimodal grades, the time to failure corresponding to these accelerated tests is about ten hours, for bimodal grade PE 100 it is roughly a thousand hours and for the new PE 100 RC grade is greater than 1 year. For the HDPE pipes grade with higher resistance against SCG, some modification or, alternatively, new tests should be developed that make it possible even for these materials to obtain the results required in a relatively short time. In this contribution we focus mainly on the PENT test and the possibility of the utilization of a structural analysis as well as a numerical approach for prediction of the lifetime of a new generation of PE grades.

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1. Introduction

Polyethylene has many critical applications in terms of pipes for conveying gas, water, sewage and chemicals. The long-time mechanism of failure is controlled by a process of slow crack growth (SCG). A crack initiates in the region near a stress concentration such as a void, notch or dirt particle, and grows under low stresses that occur during service [1]. N. Brown and his co-workers [2] developed an accelerated test now known as the Pennsylvania Notch Test (PENT) that enables us to produce the same type of brittle fracture that occurs in pipes after a long service time due to SCG. This test later became an international standard [3]. The PENT test can be performed on specimens moulded from the resin or specimens taken from the finished product (e.g. a polymer pipe). The failure processes start with a craze that is initiated near a stress concentration when the specimen is loaded. Then, the craze grows and the base of craze rupture slow crack extends. The PENT test has been used to improve the SCG resistance of resins [2]. A manufacturer can easily modify their method of resin production and use the test to evaluate the effect of the technology on SCG resistance, since failures in PENT tests occur about 20-30 times faster than in current hydrostatic pressure tests on pipes.

Large-scale usage of polyethylene pipes to convey gas began in about 1965. The first generation pipes are termed PE32, PE40 and PE63 and the second-generation are termed PE63 or PE80. This classification is based on the minimum required strength (MRS), which has to be applied for long-term loaded PE pipes operating at a temperature of 20 °C for at least 50 years (EN 12201-1). The third generation of PE is referred to as PE100. The new types of PE100 or PE100 RC should reach their lifetime over a period of 100 years [4].

This type of PE is bimodal (for comparison with conventional unimodal PE, see Fig. 1). Keeping in mind that for unimodal grades the time to failure according to the accelerated tests is about ten hours [2,5], for bimodal grades it is roughly a thousand hours and for the new PE100 RC

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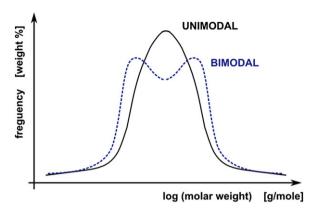


Fig. 1. Structural difference between bimodal and unimodal PE.

grade is greater than 1 year. With the development of a new generation of PE pipe grades the question arises as to whether the PENT test is a reliable tool for the prediction of its working lifetime. There are other mechanical tests that can provide quick answers regarding resistance against SCG, such as the Full Notch Creep Test (FNCT) [6–9], Cracked Round Bar (CRB) test [10,11] or the modified tensile test [12,13]. In some papers, good correlation between PENT, FNCT and modified tensile tests has been found [7,12,13].

It is known [14,15] that the lifetime of HDPE is influenced by a large number of structural and morphological parameters. The primary form of these is chain structure: (i) molecular weight and its distribution, (ii) number, type and distribution of short and long chain branches and (iii) type and number of unsaturated bindings. Molecular weight and its distribution are usually estimated through Gel Permeation Chromatography (GPC). It is possible to determine the type and the number of short chain branches from measuring Nuclear Magnetic Resonance (NMR) spectra, but this procedure is rather expensive. The fractionation technique (TREF-Temperature Raising Elution Fractionation) has been proved to be good but highly expensive and time consuming for characterisation of the chemical structure of macromolecules in terms of branching distribution vs. chain length. Therefore, reliable but less expensive methods have been developed [16,17] which permit the separation of the macromolecules with respect to their crystallizability (SIS - Stepwise Isothermal Segregation). The average density of chain branches can be estimated on the basis of Differential Scanning Calorimetry (DSC) after the SIS procedure. In 1999 D. Gueugnaut and D. Roussellot [17] published a modified version of the SIS technique ("rapid" SIS/DSC). A so-called "drift" molecular parameter is calculated from the DSC crystallization on the basis of a classic Avrami approach.

The primary morphological effect of short chain branches is (i) decrease of the lamellar thickness and (ii) increase of number of tie molecules. It is generally believed [18] that the slow process of craze opening involves the disentanglement of the tie molecules, which leads to fracture of the fibrils.

It is a well known fact that the polymer matrix undergoes structural changes during processing as a result of thermomechanical degradation (scission of the polymer chains, grafting, crosslinking). The structural changes can be detected by SIS/DSC as well as by modified SIS/DSC.

A combination of various structural methods [19] enables us to correlate the results of mechanical tests (FNCT, PENT, tensile tests) and structural parameters. Furthermore, this correlation is suitable for selection of the relevant structural parameters that are responsible for resistance against SCG. For unimodal material, very good correlation was found between the relevant structural parameters and accelerated tests [2,14,19]. Nevertheless, the relationship between structural and mechanical parameters does not exhibit the same trend for different grades of HDPE. For PE 100 and PE 100 RC, there are only limited experimental results published [4,20].

Based on a combination of experimental observations and fracture mechanics, SCG can be determined by the PENT test [21]. Using the assumptions of linear elasticity, the loading conditions of a PE structure can be described by the stress intensity factor K_I [10,21]. Based on knowledge of K_I and slow crack kinetics, the lifetime of the pressure pipe can be estimated. In the present contribution we summarize our own experiences of using accelerated tests (PENT tests) and the structural approach as well as numerical simulation for predicting the lifetime of unimodal and bimodal material. The aim is to open discussion about how to proceed in predicting the lifetime of the new generation of bimodal grades.

2. PENT test

The PENT test [3] – Pennsylvania Notch Tensile Test to measure the Resistance to slow crack growth of resins is utilised mostly in the USA. The FNCT test [6] – Full Notch Creep Test (determination of environmental stress cracking of polyethylene) developed by Hessel and Mauer is spread mostly in Europe. In contrast to the FNCT test, the PENT enables following the kinetics of slow crack growth (SCG) and estimates some relevant parameters: (i) time for SCG initiation – t_i , (ii) rate of SCG – d(COD)/dt and (iii) time to failure – t_f . Typically, a single edge notch (SEN) tensile specimen is used and dependence of COD vs. time is measured, see Fig. 2.

The single edge notch tensile (SEN) specimens can be taken either from compression-moulded plaques or from pipes, see Fig. 3.

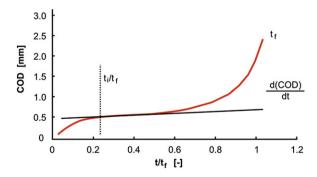


Fig. 2. Crack opening displacement COD of the SEN specimen vs. time.

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