



Post-mortem analysis of failure in polyvinylidene fluoride pipes tested under constant pressure in the slow crack growth regime

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Abstract

Under low pressure and high temperature (around 120 °C), α -poly(vinylidene fluoride) pipes submitted to burst pressure resistance tests fail after several thousands hours because of very small cracks. Optical and electronic microscopy are used to characterise the morphology of these cracks and to understand how they nucleate and propagate. Micro-cracks nucleate within the pipe wall just below the inner skin, propagate along the thickness and the generating line of the pipe. They first emerge on the inner surface of the pipe. Once the pressurised water penetrates the crack, the last stage of propagation before emerging on the outer surface is thought to be short. The influence of stress fields and microstructure parameters on the initiation and propagation of cracks is considered.

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1. Introduction

Poly(vinylidene fluoride), or PVDF, is a fluorinated polymer widely used in applications such as pipes, linings, machine parts or tanks in aggressive environments as for instance in chemical processes or in off-shore oil production or in environments with very high requirements of purity, i.e. ultrapure water piping in semiconductor manufacturing. In all these application the α -crystalline phase of PVDF is predominant, as

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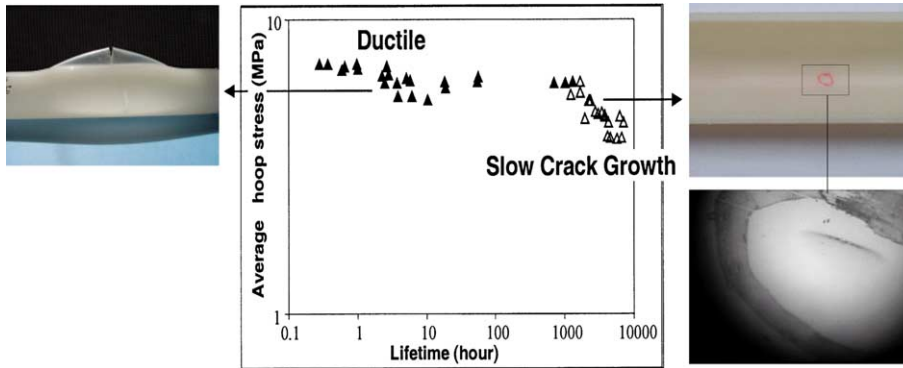


Fig. 1. Results from burst tests on α -PVDF pipes (external diameter 32 mm; average thickness 2.5 mm) (ISO 9080 standard).

it is the phase that is directly obtained from melt crystallisation. Its glass transition is at $-40\text{ }^{\circ}\text{C}$ and the melting point at $170\text{ }^{\circ}\text{C}$ [1,2]. In all applications described above the precise knowledge of the mechanical properties for very long periods of time, that is, in general 20 years or more is a prerequisite. In particular, the knowledge of the creep properties and the failure modes is important in order to establish safe design criteria. Since PVDF has a very high thermal ageing resistance, it is sometimes used in applications where mechanical performance at temperatures above $100\text{ }^{\circ}\text{C}$ is required. As with any thermoplastic material, creep properties become important upon approaching the melting temperature. Furthermore, new relaxation phenomena can give rise to additional failure modes. In the case of PVDF the motion of crystalline defects, the so-called α transition, is known to be activated above $100\text{ }^{\circ}\text{C}$ [3]. The general method employed to measure and describe creep and failure modes for thermoplastics is closely inspired by the actual pressure pipe application. To predict pipe lifetimes, burst tests under constant pressure and at a given fixed temperature are usually performed [4,5]. Results from such tests on a standard pipe grade PVDF in the α phase are presented in Fig. 1.

From this graph of applied hoop stress vs. lifetime or time to pipe failure two regimes can be distinguished. The high stress regime is characterised by a ductile failure mode – pipes burst after localisation of the deformation and creation of a blister – while, at low stresses, failure is due to the propagation of a very small crack. As in this last case no macroscopic plastic deformation is observed, this slow crack growth regime is sometimes called “brittle”. Polyethylene (PE) is known to exhibit a similar behaviour and the slow crack growth regime has been extensively studied for years in this material [6–9]. As the time necessary to activate the slow crack growth regime during burst tests is quite long (several months at least), most studies lead on PE focused on the development of short-term tests and the influence of microstructural parameters.

The aim of this paper, which is part of a study dedicated to slow crack growth in PVDF, is to characterise damage and crack morphology in pipes submitted to creep tests under constant pressure in the “brittle” regime. This study is aimed to give references for example to compare the damage obtained in any short-term test.

2. Experimental

2.1. Materials

The PVDF types studied here are commercial grades (Kynar[®] 740 and Kynar[®] 1000) elaborated by Arkema. Pipes were extruded with a typical geometry as pipes used in chemical applications meaning an

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