Test method

Evaluating the influence of contacting fluids on polyethylene using acoustic emissions analysis

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A B S T R A C T

Identifying microstructural changes related to solvent effects in their early stages to prevent mechanical losses in polymers may be crucial, especially for applications where a leak is hazardous, as in fuel piping and storage tanks. Acoustic Emissions (AE) testing has proven to be a valuable tool for monitoring material failure, though mostly until now for composite systems. The present study examines the use of AE techniques to monitor and characterize the effects of two different chemical agents (i.e. an aqueous detergent and toluene) on the structure of a semi-crystalline polymer (high density polyethylene, HDPE) over different periods of exposure. Characterization of the aged polymer by tensile testing and X-ray diffraction was correlated with the signal-based and parameter-based analysis of AE information. Results presented reinforce the use of AE testing as a promising technique for understanding structural changes in polymers.

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1. Introduction

The service conditions for many polymers in everyday use often involve contact with fluids. When appropriately selected for the purposes of containment or transport, the polymer should exhibit dimensional stability, chemical resistance and low permeability to such fluids. However, the influence of a fluid can differ greatly based, not only on molecular structure of the polymer, but also the processing history of a manufactured part, as amorphous regions will display greater sensitivity to solvent effects in comparison to its crystalline state [1]. The extent and rate for sorption of a fluid into a polymer while in service will depend on many factors including compatibility, ambient temperature, surface integrity (ex. pores or microcracks) and exposure time. The outcome of absorbing a chemical is rarely beneficial as there is usually a decrease in mechanical properties, environmental stress cracking, variation in part tolerances, and

possibly disruption of other functional properties that may be important to the use of that polymer. Fortunately, if the polymer selection is done appropriately, these changes should be on a timescale matching the overall service life of the polymer; however, some uses have such critical importance to society that the service life must be known accurately as a failure would have far-reaching consequences, such as for the transport or storage of fuels. In such cases, more sensitive criteria for polymer selection must be realized based on informative testing or monitoring methods where detection of fluid sorption can be made on relatively short time scales before significant changes to structure have occurred. Acoustic emission (AE) methods are proposed herein as a suitable approach to both characterize the plasticizing effect of a fluid at the time of failure (ex. ASTM D1693) but may also actively monitor a polymer while in service. The present study focuses on polyethylene, which is a semi-crystalline polymer that is normally considered chemically inert to most solvents, and hence often selected for fluid storage and piping applications [2].

Signal-based AE analysis relates to the quantification of fracture modes in materials using the recorded signals to

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extract detailed information from an AE event. An acoustic emission in this respect refers to propagating elastic waves within a material, which can provide internal morphological information related to the events that initiated them, and often provide such insights long before damage is evident by other methods of characterization. Emitted waves are most often related to an elastic relaxation response after micro-crack formation, but other events of energy release such as interfacial de-cohesion for composites can also be involved. Generating these elastic waves requires circumstances where stresses concentrate locally in a material, generally precipitated by an external load, though other sources such as residual stresses related to processing, or swelling from solvent absorption, can be the cause. Hence, AE events may carry signatures that can be correlated with modifications generated by ageing conditions [3].

Fluid influences on the environmental ageing of polyester and epoxy composites have been more studied by AE techniques compared to any other class of polymer [3–6]. The work in that area has been primarily concerned with the use of AE analysis to reveal micromechanical information of the polymer-filler interface during deformation, or damage of the composite after its exposure to a chemical. In general, it has been reported that AE activity (based on peak counts) decreased with mechanical strength, while AE events (number of emitted waves that exceeded a threshold amplitude) increased as hydrolytic damage occurred to the matrix resin. In comparison, studying fluid interactions on non-reinforced polymers with AE techniques has been only sparsely covered in the literature. The only known study by Ishiyama et al. [7] examined the environmental stress cracking behavior of polymethylmethacrylate (PMMA) related to ambient humidity with the assistance of AE monitoring. The authors found that the number of AE events over time correlated well with the crack growth rate within PMMA. The brittleness of the material meant no AE events were detected until it failed. As polyethylene is a more ductile material compared to PMMA, AE events often occur prior to failure, although the signal amplitude is comparatively smaller.

The present study aims to identify descriptors from the AE signal that relate to the influence of two distinctly different fluids on polyethylene, a semi-crystalline ductile polymer, and determine if this analysis can be a sensitive approach to detecting damage mechanisms related to these fluids. The tests compared an aqueous solution containing a stress-cracking agent, often used in environmental stress crack testing, versus toluene, which is a moderate solvent to the polymer and considered representative of the aggressive environment in pipes and storage media for petroleum liquids. The study is part of a larger investigation into uses of AE techniques for polymer stability monitoring.

2. Experimental

2.1. Materials

High density polyethylene (HDPE) sheets of 3 mm thickness were purchased (McMaster-Carr) and cut into test specimens having dimensions of 100 mm × 30 mm.

The determined Young's modulus and ultimate tensile strength of the supplied resin was 2.2 GPa and 21 MPa, respectively. Test specimens used in the experiments were notched in a rig with a razor blade at a controlled speed of 0.25 mm/min to 1.4 mm depth on one face and 1 mm depth along its two edges; the procedure was intended to prevent premature initiation of a failure. All sample preparation was done following the specifications of ASTM Standard F1473, 2011. The fluids used in the study were toluene (HPLC grade, JT Baker) and a 10% aqueous solution of Igepal® CA-630 (ethoxylated octylphenol, a non-ionic surfactant by Solvay-Rhodia); Igepal is a known stress-cracking agent to polyethylene.

2.2. AE monitoring system

Two 300 kHz multi-resonant AE sensors (R30α, Mistras Group Inc.) were used in the AE investigation. The signal of one sensor was pre-amplified by 20 dB. No other conditioning was applied to the signals prior to being recorded. The signals of both sensors were sampled with a 10 MHz 12-bit 4-channel simultaneously-poling data acquisition card (National Instruments Corporation) attached to a computer running LabVIEW™ (Version 2013, National Instruments Corporation). A sampling rate of 1.5 MHz was used in these trials. The sensitivity and reproducibility of the AE setup with the HDPE test specimens was evaluated using the Hsu-Nielsen pencil lead break test mentioned in ASTM F976 prior to commencing experimental trials. Hit detection for recognition of an AE event used a threshold value of 6 mV based on the amplified sensor signal.

The recorded signals were studied using wavelet analysis. The energy of an AE event was estimated by digitally filtering the recorded signal using a Haar wavelet transform and then integrating the area under its waveform envelope (specifically MARSE, mean area under a rectified signal envelope). The acoustic 'fingerprint' of the signal, used to differentiate the influence of each fluid on HDPE, involved creating frequency-time plots based on Fast Fourier Transform (FFT) applied to Haar wavelet transform filtered signal. All processing routines of the signal were done using code developed in Python 2.7.6 with the pyWavelets 0.2.2 library.

2.3. Testing procedure

Notched test specimens were fully immersed in a large sealed container filled with either toluene or Igepal solution and left to soak at ambient conditions (23°C) for 1, 6, 24 or 72 h. A specimen for testing was removed, rinsed with water and dried prior to mounting in the universal mechanical testing system (Model 3366 with a 5 kN load cell; Instron Corporation, MA, USA). The two AE sensors were positioned on the same face above and below the notch, each at a minimum of 10mm distance, and held in place by separate grips. A couplant, recommended in ASTM E650, was not used as none could be found that did not promote slippage of the sensors during testing; however, the pencil lead break test revealed this was not an issue for collecting a good signal. A third AE sensor (14 kHz resonant frequency, R1.5, Mistras Group Inc) was attached to the test frame of