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Prediction of strain rate sensitivity of high density polyethylene using integral transform of dynamic mechanical analysis data

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ABSTRACT

Recent interest in understanding the effect of strain rate on mechanical properties has motivated this study to develop a correlation between frequency domain dynamic mechanical analysis (DMA) results and elastic modulus values that are obtained from a separate set of elaborate tensile tests conducted over a wide range of strain rates. Using the time-temperature superposition principle and the integral relations of viscoelasticity, the DMA results are converted into a time-domain relaxation function in order to predict the strain-rate dependent modulus. The transformation technique is validated with experimental results for high density polyethylene (HDPE) resin and is found to be accurate over a wide range of strain rates. Cross correlation between DMA results and tensile test results over a wide range of strain rates can help in substantially reducing the requirement for tests that are needed to characterize the material behavior with respect to strain rates, temperature and loading frequency.

1. Introduction

Despite the wide availability of dynamic mechanical analysis (DMA) results on polymers and polymer matrix composites, such data have rarely been applied to design of structures and components because frequency-domain results obtained through this method are not directly applicable to most engineering problems. For thermosets, dynamic mechanical analysis is principally used to find maximum use temperature and glass transition temperature (T_g) [1,2], which can determine the suitability of the material for application in a particular environment. However, for thermoplastics which are used above T_g, such as high density polyethylene (HDPE), and whose mechanical response is highly time-dependent, such information is less useful.

The storage and loss moduli obtained from DMA provide a measure of energy stored and lost, respectively, in a material when a cyclic loading-unloading profile is applied. Numerous DMA studies on polymers can be found. DMA is considered the most sensitive method to locate thermal transitions [3–7] including those in crystallization and resin curing. When combined with other spectroscopy methods, information from DMA can reveal activation of different modes of motion of the polymer chains [8–10]. DMA is also used to gain information on the temperature sensitivity of the behavior of polymer blends [11,12], pharmaceutical and biomedical materials [13], and micro- [14–16] and nanocomposites [17–25]. Most of these studies have reported storage modulus E', loss modulus E'', damping parameter tan δ, and T_g. However, the relation of E' and E'' to elastic modulus at different strain rates is not developed in these studies, which is a major current limitation in using the DMA results in mechanical design. In order to develop this relation, the DMA data needs to be transformed into a time-domain representation which can yield more readily useful information about the material behavior.

Measurement of properties at widely varying strain rates is often complicated by the limited crosshead displacement speed ranges attainable within one testing setup or by a particular method. In addition, very low strain rate tests are time consuming and expensive to conduct, making it difficult to test multiple specimens at multiple strain rates and temperatures to develop a comprehensive understanding of mechanical properties of the material. Augmenting these present limitations, it is also noted that the correlation between results obtained from tensile or compressive tests with DMA results have not been established to develop a comprehensive understanding of the time and temperature dependent behavior of materials.

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In this work, the frequency-domain storage modulus function obtained by DMA is inverted to obtain the time-domain relaxation function, which is then used to obtain the linear viscoelastic response of the materials at a given strain rate. Various material properties can be found from this transformed data, such as secant (or tangent) modulus at a given strain, and energy absorption at a given elastic strain. Various exact and approximate relations exist for converting this function to creep compliance as well [26,27]. Using the time-temperature superposition (TTS) principle, a series of frequency sweeps at different temperatures are combined to yield the isothermal frequency response over a frequency range that is wide enough to ensure convergence of the transform over the desired strain rates. This transform technique is validated by comparing the predictions with reported values for HDPE in the literature and good agreement is found over a wide range of strain rates. HDPE was chosen for study in this work due to its widespread use in manufacturing industrial products. HDPE is also used extensively as a matrix resin in polymer matrix composites [28,29]. However, the method developed in this work is expected to be applicable to other polymers as well.

2. Experimental

2.1. Materials

Virgin HDPE of HD50MA180 grade procured from Reliance Polymers, Mumbai, India is studied in the present work. The HDPE has a melt flow index of 20 g/10 min (190 °C/2.16 kg). The resin is in granular form of 3 mm diameter and has a mean molecular weight of 97,500 g mol^{-1}.

2.2. Sample preparation

HDPE specimens are fabricated using an industrial scale horizontal type polymer injection molding (PIM) machine (Windsor, 80 ton capacity). Operating and processing parameters of the PIM machine were optimized in a set of earlier studies [28–31] and are set at 160 °C temperature and 30 kg/cm^2 (2.9 MPa) pressure. Samples of dimensions 60 × 12.7 × 3.3 mm^3 (length × width × height) are molded for use in the DMA study.

2.3. Dynamic mechanical analysis

Dynamic mechanical analysis is conducted using a TA Instruments (New Castle, DE) Q800 DMA. Specimens of nominal dimensions 60 × 12.7 × 3.3 mm^3 are tested in the dual cantilever configuration with a span length of 35 mm. Testing is conducted in the strain control mode with a maximum displacement of 25 μm.

DMA testing is conducted in two phases: (a) using the temperature sweep mode at constant frequency and (b) using the frequency sweep mode at constant temperature. In the temperature sweep test, the temperature is ramped from 35 °C to 130 °C at a rate of 1 °C/min with the deformation occurring at a constant frequency of 1 Hz. Testing is halted once E' reaches a value of 20 MPa to prevent total melting of the specimen inside the DMA chamber. At least five specimens of each type are tested in this phase. In the frequency sweep testing, the temperature is stepped from 35 °C to 120 °C in increments of 5 °C. At each temperature step the specimen is soaked for 5 min to ensure thermal equilibrium. The dynamic properties are measured at 20 discrete frequencies logarithmically spaced between 1 and 100 Hz at each temperature step. At least three specimens of each type are tested in this phase. The results of temperature and frequency sweeping are combined using the time-temperature superposition (TTS) principle to generate master curves describing the behavior of HDPE over a wider range of frequencies.

3. Results

3.1. Temperature sweep

A representative set of results of the temperature sweep for E', E'', and tan δ for one HDPE specimen are shown in Fig. 1. As T_g of the HDPE is about −110 °C [32], the experiments in the present work are conducted entirely in the rubbery region and the dynamic properties with respect to temperature do not show step changes or peaks which may indicate phase transitions. Results on E' are extracted at three arbitrarily selected temperatures in the rubbery plateau region of 60, 80 and 100 °C and are presented in Table 1.

The trends of E'' with respect to temperature are presented in Fig. 1a for HDPE resin. E'' values extracted at three representative temperatures are presented in Table 1. The peak in E'' at around 50 °C corresponds to the α-relaxation in HDPE, which is associated with softening of the interface between crystallites and the amorphous phase [32,33]. Tan δ results are presented in Fig. 1b and the values extracted at selected temperatures are presented in Table 1. This property, also known as the damping parameter, loss factor or loss tangent, is the ratio of the E'' to E' and represents the relative magnitudes of the elastic and viscous behavior of the material. Previous studies have reported similar trends for E' and E'' for polyethylene in the similar temperature range [32,33]. Although only the α-relaxation peak is partially observed in the test

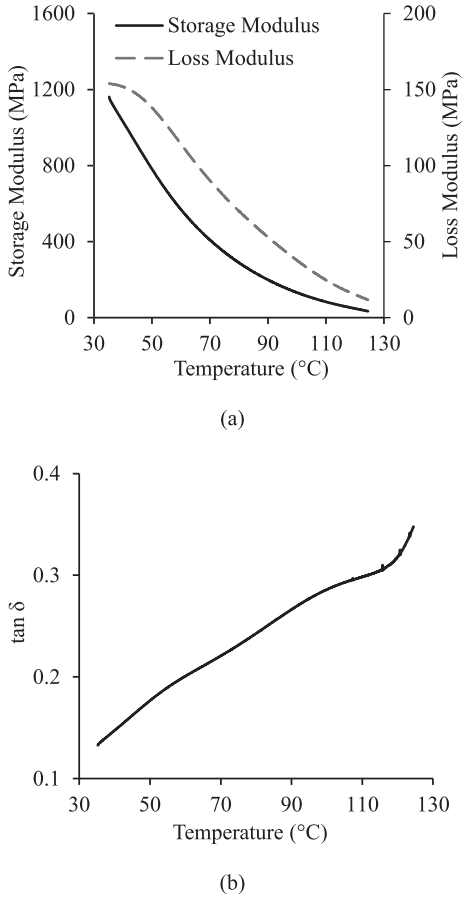

Fig. 1. (a) Storage and loss moduli and (b) tan δ results for HDPE resin from DMA temperature sweep at 1 Hz.