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Original Research

Mechanics of ABS Polymer under Low & Intermediate Strain Rates

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Abstract

Thermoplastics polymers like Acrylonitrile Butadiene Styrene (ABS) are often reinforced with nano/micro reinforcements to enhance their mechanical, thermal and electrical properties. However, the viscoelastic nature of these polymers results in their strong dependence on the applied strain rate and temperature sensitivity, leading to their characterization complexity. Hence it is paramount to study the strain rate-dependent mechanics of neat ABS. In this study, the effect of strain rate and temperature on Young's modulus of ABS polymer was characterized using a dynamic mechanical analyzer (DMA). Storage modulus curves at various temperatures and frequencies were transformed into a representative master curve at a specific temperature using the time-temperature superposition (TTS) principle. Based on this curve's storage modulus and frequency relation, an empirical fit function was developed and the strain rate values were extrapolated. Using integral relations of viscoelasticity, the results were further transformed to a time domain relaxation function to extract the strain rate-

sensitive Young's modulus at different loading rates. This method was validated by comparing the data with tensile tests conducted on ABS coupons as per ASTM D638-14. The results were acceptable over a wide range of strain rates and indicated a clear sensitivity of ABS to strain rate and temperature. The strategy used in this work can be employed to study the effect of reinforcement morphology in ABS thermoplastics using DMA.

Keywords
Acrylonitrile Butadiene Styrene (ABS); quasi-static strain rate; intermediate strain rate; dynamic mechanical analysis (DMA); elastic modulus; dynamic behavior of ABS

1. Introduction

Polymers are extremely strain rate sensitive due to their viscoelastic nature and morphology. Depending on the strain rate and temperature at which they deform, they can exhibit a wide range of deformation behavior from ductile to brittle to rubbery [1]. This leads to challenges in the characterization of their material response. A large number of experimental techniques exist in the literature to investigate polymer behavior at different rates of loading, including modified Split Hopkinson pressure bars [2] high-speed servo-hydraulic machines [3] and drop-weight impact machines [4, 5]. However, these fixtures are either expensive, complex, or have their own set of limitations regarding maintenance of dynamic equilibrium and constant strain rate along the gage length of the sample, an inertial effect due to fixture weight, and sample pre-loading, among many other factors. These test apparatus limitations are more pronounced in thermoplastics, since they are softer than thermosets and have a high loading rising time. To overcome these challenges and reliably characterize the strain rate-dependent behavior of thermoplastics, several recent studies have shown the potential of using the dynamic mechanical analysis (DMA) technique [6-11]. To this end, this work aims to understand the low to intermediate strain rate (1 s^{-1}-100 s^{-1}) and temperature-dependent behavior of Acrylonitrile-Butadiene-Styrene (ABS) polymer using the DMA technique.

Thermoforming is a general manufacturing process where plastic sheets are heated, molded and trimmed into useful three-dimensional parts. ABS thermoplastic provides an excellent solution for thermoforming due to its high sagging resistance during thermoforming [12], and thereby extensively used in the production of automotive, construction and material handling products. Such mass-volume production of ABS components from extruded sheets using thermoforming and other processes demands an in-depth understanding of the mechanics under uniaxial extension rates and temperatures to aid the design and manufacturing processes [13].

When subjected to elastic strains, thermoplastic polymers exhibit a viscoelastic behavior that combines the characteristics of both elastic solids and Newtonian fluids, as shown in Figure 1. As the deformation starts, the Hookean law of the linear relationship between stress and strain does not hold. Further, when the stress is released, the loci of the unloaded path do not trace over the loading curve, due to the viscous nature of these plastics. Hence, to completely understand the deformation behavior of thermoplastics, it is critical to investigate their viscous response, which influences properties like stress relaxation, creep and impact resistance. Typical tensile equipment

like hydraulic and screw-driven machines, focus only on the elastic component and are less sensitive to changes in the material microstructure in the viscoelastic range. DMA technique presents a unique solution to this problem and can determine the viscoelastic properties of thermoplastics by subjecting them to sinusoidal loading and measuring the resultant sinusoidal displacement. The loading curve is called as storage modulus and is a representation of stored elastic energy. This curve is analogous to Young's modulus related to the material's stiffness.

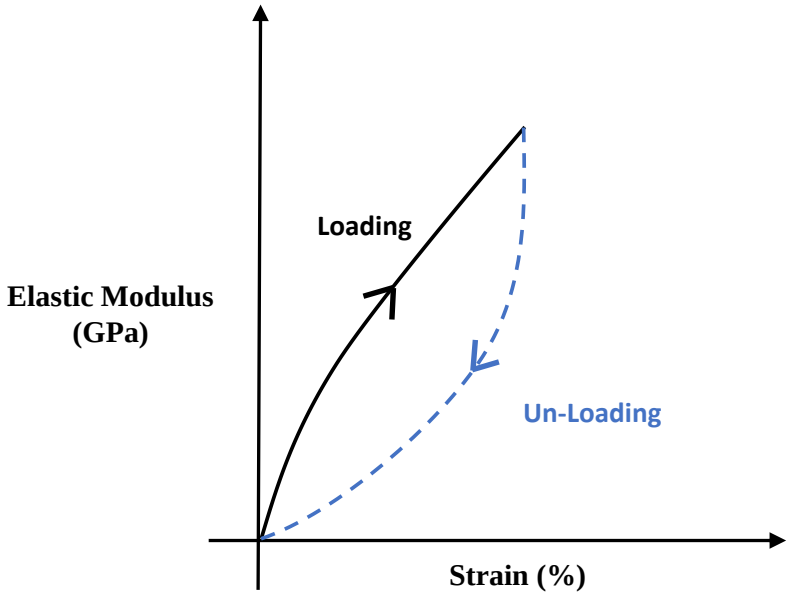

Figure 1 Schematic of loading and unloading curves of thermoplastic polymers under sinusoidal loading.

The phase difference between the loading and unloading sinusoidal curve is called the loss modulus and represents the energy loss. The energy loss is attributed to the conformational changes to polymer chains. Under displacement within the elastic phase, the initial resistance comes from the intermolecular resistance (Van der Waal's forces) between the molecular chains wherein it rotates and translates concerning one another while undergoing conformational changes to accommodate the newly displaced position. When the load is removed, the material returns to its original equilibrium position. However, the original conformational chain positions are changed and do not experience the similar resistance of the loading cycle.

DMA is an effective technique to determine thermoplastic viscoelastic parameters such as creep, stress relaxation, dynamic stress-strain relations, and transition temperatures. One major drawback however is that this method can only be used to characterize the deformation behavior within the quasi-static strain rates (<1 s^{-1}). Superposition principles have been employed in the literature to overcome this limitation and extrapolate the results to intermediate and high strain rates. The time-temperature superposition (TTS) principle based on the relation between stress relaxation time and temperature is more common [11, 14, 15]. This technique draws an equivalence between longer relaxation time at low temperatures to the relaxation behavior at high temperatures to predict the material response. Another effective technique to predict Young's modulus of thermoplastics at temperatures and strain rates beyond the capabilities of the DMA was introduced by Mulliken et al. [6]. The authors introduced the decompose/shift/reconstruct (DSR) method, in which the storage

modulus curves obtained via DMA were first decomposed into α and β curves. These curves were then shifted over higher decades of strain rates using experimentally derived shift factors before reconstructing them at any required loading rate.

Modulus curves for viscoelastic materials obtained using DMA are in the frequency domain and cannot be used directly to design structural components and other related engineering problems. Recent studies however have shown the transformation of frequency domain DMA data to time domain for yielding useful information on the material behavior [8-11]. These studies used the TTS method with the integral relations of viscoelasticity to convert the storage modulus from the frequency domain to the time domain. Another approach was developed by Dao-Long Chen [16] based on relaxation creep duality representation, a variant of the Prony series. The studies above have used neat polymers or their composites as test material. For this study, the authors have selected ABS, mainly because of its excellent energy absorption capabilities and adaptability to rapid manufacturing techniques. Several sources confirm ABS's chemical composition and property [17]. Several studies have characterized neat and reinforced ABS polymers at quasi-static strain rates [18-29] using a combination of DMA and traditional tensile fixtures. However, using servo-hydraulic machines, limited exploratory research was conducted to understand the effect of low and intermediate strain rate effects on ABS [30, 31]. At high strain rates, Nakai and Yokoyama [32, 33] have characterized ABS behavior from 700 s^{-1}-800 s^{-1} using SHPB. Further investigations into the tensile deformation response of ABS as a function of strain rate are required to develop a comprehensive understanding of the stiffness evolution.

This work aims to determine the temperature-dependent Young's modulus of ABS polymer under low and intermediate strain rates (1 s^{-1}-100 s^{-1}) using DMA. Storage modulus curves obtained at different quasi-static strain rates using DMA were extrapolated to intermediate strain rates using an analytical fit function. Storage modulus at various strain rates obtained in the frequency domain was then transformed into the time domain Young's modulus using integral relations of viscoelasticity.

2. Experimental Details

2.1 Materials & Manufacturing

ABS terpolymer (Cycolac™ Resin MG94) used in this study was obtained from Sabic®. Micro-extruder of 15 ccs. (DSM Netherlands) was used to manufacture the ABS tensile sample for DMA testing. At first, the ABS pellets were dried for 3 hours at 80°C to remove moisture. ABS pellets were fed to the extruder barrel that houses two intermeshing conical screws. A processing temperature of 240°C and conical screw speed of 100 rpm was maintained during the extrusion process. The polymer pellets were mixed for 10 minutes in the barrel. Further, the molten sample from the barrel was moved into a transfer cylinder maintained at the same ABS processing temperature. This molten sample from the transfer cylinder was pressed using a compression mold to make films of 0.3 mm thickness.

2.2 Dynamic Mechanical Analysis

The dynamic mechanical analysis on neat ABS specimens was carried out using Q800 from TA instruments. DMA was conducted under tensile testing configurations with samples of nominal

dimensions 18 mm × 5 mm × 0.3 mm. Testing was performed in the strain control mode with a maximum displacement of 20 μ. The temperature step/Frequency sweep test method was adapted in this analysis. In this test mode, the material sample was exposed to a series of increasing isothermal temperatures from 25°C to 95°C and the responses were collected at every 10°C interval. At each interval, the specimens were soaked for five minutes to ensure thermal equilibrium and were deformed at four different frequencies (1, 10, 100 & 1000 Hz). Four specimens were tested in this phase to ensure repeatability in data collection. The strain rates experienced by the samples corresponding to the DMA frequency were estimated based on (1) [6].

$$\dot{\varepsilon} = \frac{\left(\frac{d_o}{L_g}\right)}{X} \tag{1}$$

where, d_o and L_g are the prescribed displacement amplitude and sample gage length. The parameter X is defined as;

$$X = \frac{1}{4\omega},$$

where ω is the applied frequency at which the samples were excited.

3. Results and Discussion

Rectangular samples of ABS polymer were tested in the DMA, at temperatures lower than 105°C (glass transition temperature of ABS) to ensure no phase transition was experienced during the testing [34]. A representative storage modulus curve for ABS at a frequency of 1 Hz is plotted in Figure 2. The modulus curve experiences a dip near 100°C, which indicates the onset of a material phase transition during which ABS starts to melt and the storage modulus drops off exponentially.

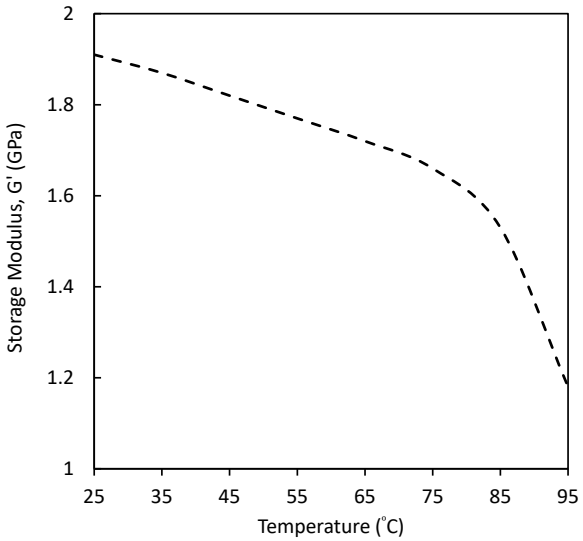

Figure 2 Storage modulus of ABS at 1 Hz.

To investigate the time and frequency-dependent storage modulus of ABS, four different frequency sweeps, ranging from 1-1000 Hz, were carried out 8 equal interval temperature steps

between 25°C-95°C. Representative DMA curves obtained from this analysis are shown in Figure 3. A direct relationship between the storage modulus and the loading frequency (rate of loading) can be observed from the figure, clearly indicating the strain rate sensitivity of ABS.

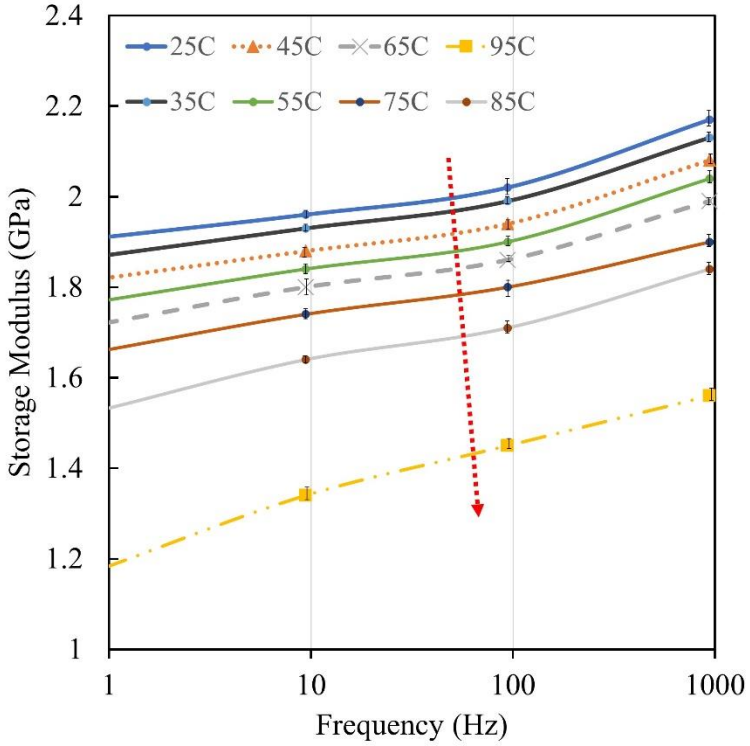

Figure 3 Storage modulus of ABS from frequency and temperature scan using DMA.

Furthermore, a reduction in storage modulus by ~30% was observed as the temperature of ABS was increased from 25°C-95°C.

3.1 Time-Temperature Superposition (TTS)

The time temperature superposition principle is based on the concept that increasing or decreasing the loading time on a material's properties to increasing or decreasing the temperature. This technique allows the superimposition of viscoelastic data obtained at a reference temperature upon the data obtained at a different temperature simply by using a shift factor.

$$E(t,T) = E\left(t_{ref}, T_{ref}\right) \tag{2}$$

Where

$$t_{ref=\frac{t}{a_T}}$$

a_T is the shift factor; t and T represent time and temperature respectively.

The conceptual premise for TTS is derived from the molecular relaxation or rearrangement of polymer chains within a thermoplastic. The rate of molecular relaxation in a polymer accelerates at higher temperatures (low strain rates). As such, the time these processes occur can be reduced by

conducting the experiments at higher temperatures and then transposing the data to lower temperatures (high strain rates).

Viscoelastic data like storage modulus can be collected using DMA by performing frequency multiplexing, where the material is analyzed under a series of frequencies at different temperatures. By selecting a reference temperature, a master curve is generated by shifting other data concerning time time or frequency, i.e., temperature above the reference shifts to lower frequencies and vice versa. The data can be shifted by using several techniques, with the Arrhenius equation (based on the activation energy concept) and the Williams-Landel-Ferry (WLF) equation (based on the free volume concept) being the most common. The WLF equation is generally preferred over the Arrhenius equation at low-temperature range and was used in this work. Using WLF, the shift factor is defined as shown in equation 3.

$$log\ a_T = \frac{-C_1(T - T_{ref})}{C_2 + (T - T_{ref})} \tag{3}$$

Where C_1 and C_2 are constants and T and T_{ref} are the measured temperature and reference temperatures. Determining the shift factor for a material using equation 3 makes it independent of the reference temperature. After fitting the WLF equation to the experimentally determined shift factor, the values for C_1 and C_2 for ABS were 6.9 and 145.8 respectively.

Once the master curve is determined using TTS, the storage modulus values chosen at the reference temperature are fitted to a second-order exponential function of the form shown in equation (4),

$$E'(\omega) = a * \exp(b * \omega) + c * \exp(d * \omega) \tag{4}$$

A, b, c, d are fit coefficients and ω is the frequency. This fit coefficient implies one smooth step transition in the storage modulus curve, as frequency goes to zero or positive infinity. The R-square value for the fit to the experimental data was above 0.99. Figure 4 shows the fit function plotted against the storage modulus.

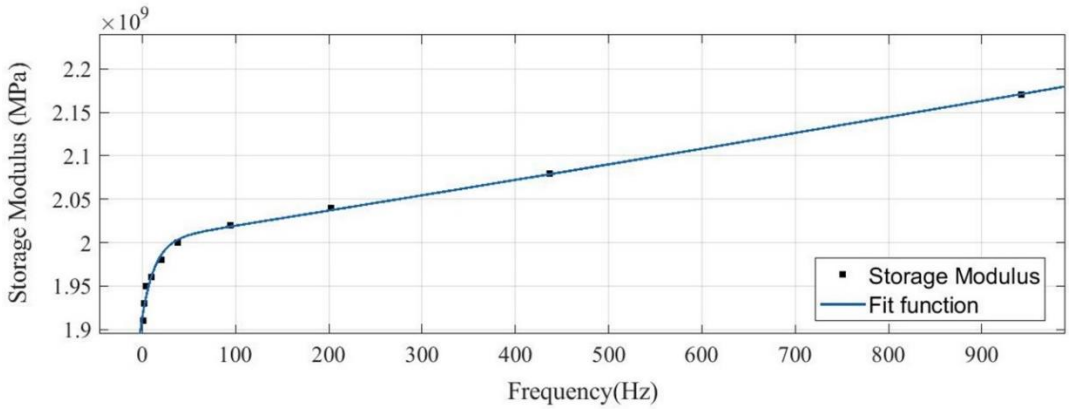

Figure 4 Comparison of exponential fit function with storage modulus from DMA.

A combination of TTS and fit function is used to generate the master curve and extrapolate the storage modulus beyond the frequencies tested using DMA. A representative curve of an extrapolated curve at 25°C is plotted in Figure 5.

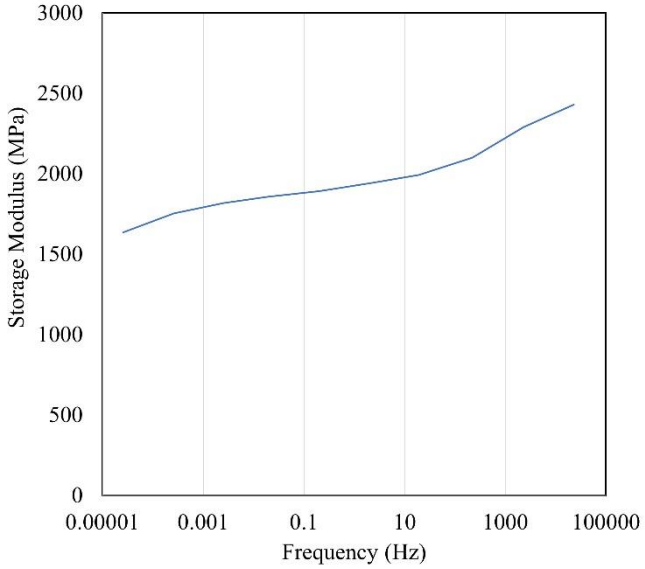

Figure 5 Extrapolated master curve at reference temperature of 25°C using TTS and proposed fit function.

3.2 Frequency to Time Domain Conversions

The viscoelastic parameters obtained in the frequency domain can be converted to more useful time domain functions for engineering applications using an appropriate transformation. Relaxation modulus E(t) or Young's modulus can be found from the storage modulus using equation 5 [35]

$$E(t) = \frac{2}{\pi}\int_0^\infty \frac{E'(\omega)}{\omega}\sin(\omega t)\, d\omega \qquad (5)$$

where 'ω' and 't' represents frequency and time respectively. The fitting function given in equation 5 was integrated to yield the time domain relaxation function.

Using this technique, Young's modulus was generated at four different arbitrary temperatures against a spectrum of strain rates ranging from 1 s^{-1} to 100 s^{-1} as shown in Figure 6. Young's modulus decreased with an increase in temperature, which exhibits a loss in stiffness. An approximate 30% reduction in modulus was observed over the entire temperature range, indicating a weaker temperature dependence commonly associated with molecular relaxations of the side chains or secondary relaxations. The gradual decrease in temperature also indicates that there was no phase change from a glassy to rubbery state which occurs only when there is an onset of chain mobility or segmental relaxation and is accompanied by a drastic decrease in the modulus values.

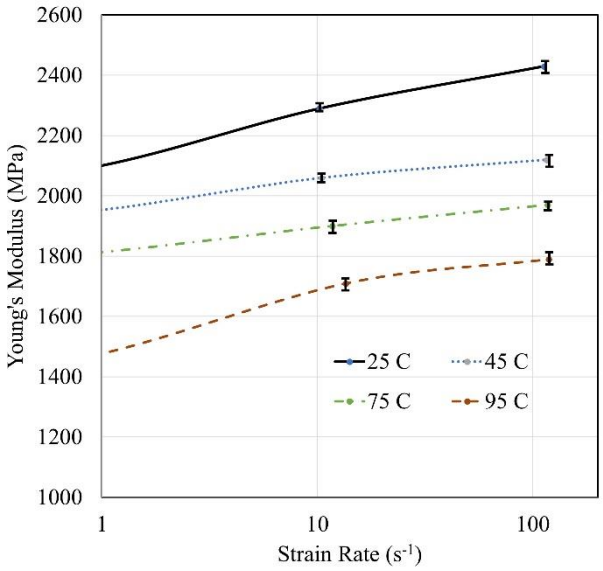

Figure 6 Young's modulus against strain rate at four different temperatures.

Figure 6 also indicated a clear correlation between the modulus and the loading rate. Young's modulus increased with an increase in strain rate for all the test temperatures. An average of 18% increase in modulus was observed in all the temperatures when the strain rate was increased from 1 to 100 s^{-1}.

It is important to note the accuracy of Young's modulus beyond the experimentally measured frequencies depends on the compatibility of the fit function in the extrapolated regions. Also, any presence of phase transitions or other material-specific changes is not captured using this specific fit function. These missing transition points can lead to the missing curvatures in Young's modulus curve plotted against the strain rates. One way to capture the transitions at higher strain rates is to conduct more DMA frequency sweeps at smaller temperatures than the reference. There are many techniques including high-rate servo hydraulic machines, modified Split Hopkinson Bars and drop-impact test fixtures, which were used in literature to predict the intermediate and high strain rate behavior of polymers, however these fixtures are either expensive or have inherent design flaws which affect the reliability of data. A fit function can provide a first-hand approximation of the polymer behavior at higher loading rates, facilitating the application design process.

3.3 Data Validation

Young's modulus data for ABS was verified against the experimental data to confirm the proposed scheme's validity. It is pertinent to mention that the larger goal of an investigation into the strain rate behavior of ABS, is to design a test fixture that can provide reliable test data for ABS (and other thermoplastics) at intermediate strain rates (from 1-100 s^{-1}). However, in the current work, the data available in the literature have been used for verification, as seen in Figure 7. As mentioned in section 1, there is a scarcity of rate-dependent tensile data for neat ABS. Only one study [27] reported the strain rate data for ABS, albeit till 40 s^{-1} at room temperature, which limits the data verification. However, to verify the current technique, the DMA test data was compared against the data obtained via a high-rate servo hydraulic machine.

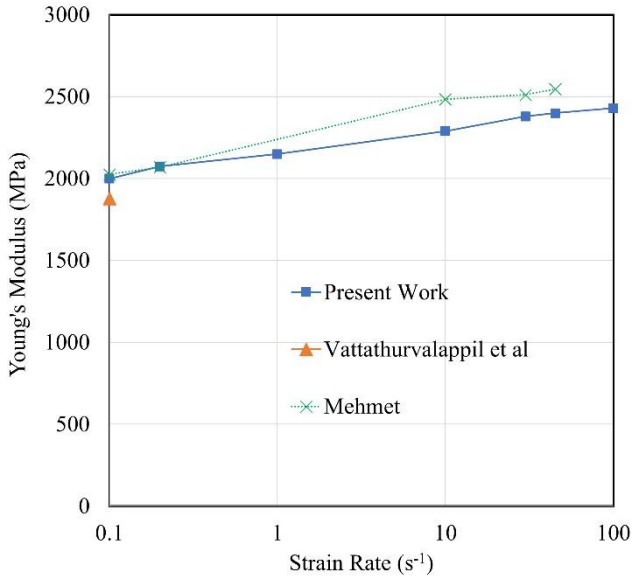

Figure 7 Comparison of Young's modulus with literature values for ABS.

Close proximity between the extrapolated strain rate data of ABS and the test data available in literature can be observed. In both cases, an increase in modulus was observed with the increasing strain rate. More importantly, less than 6% error was observed, validating that the DMA strain rate extrapolation can serve as an effective approximation technique for polymer behavior prediction at intermediate strain rates.

4. Conclusion

This work presented an approximation scheme to predict Young's modulus of ABS polymer at low and intermediate strain rates using DMA. A combination of TTS and second-order exponential fit function was employed to extrapolate the frequency spectrum, which corresponds to strain rate, beyond the limits of DMA. Further, the modulus data in the frequency domain was converted to the time domain using integral relations of viscoelasticity. The DMA approximation scheme was validated against experimental data available in the literature and a close agreement (~6%) was observed. Investigations into the strain rate dependency of the polymer modulus revealed nearly a 21 percent increase as the loading rate was increased from 0.1 s^{-1} to 100 s^{-1}. Furthermore, at all strain rates, an average decrease of 30 percent was observed in Young's modulus when the temperature increased from 25°C to 95°C. The proposed technique has an obvious flaw since material transition points cannot be accounted for via ad hoc extrapolation. Nevertheless, it can provide a reliable first-hand approximation for tensile polymer behavior at intermediate rates of loading. Further, this technique can also be extended to high strain rates by executing frequency sweeps at temperatures lower than the reference temperature (25°C).

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Author Contributions

Dr. Suhail Hyder Vattathurvalappil – Conception, Data collection, Data analysis, Article drafting. Mr. Syed Fahad Hassan – Data analysis, Article drafting. Dr. Mahmoodul Haq – Critical revision of the article.

Competing Interests

The authors declare no financial or non-financial interests that are directly or in-directly related to the work submitted.

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