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Discrete Deconvolution to Extract Viscoelastic Material Properties from Spherical Nanoindentation

***[1]M. T. Abba, [1]M. Dauda and [2]S. R. Kalidindi**

[1]Mechanical Engineering Department, Air Force Institute of Technology, Nigerian Air Force Base, PMB 2104, Kaduna, Nigeria.

[2]The George W. Woodruff School of Mechanical Engineering, Georgia Institute of Technology, Atlanta, GA 30332 0405, USA.

m.tahir.abba@gmail.com*

Research Article

Abstract

This study looks at how discrete deconvolution can be used to extract the relaxation modulus and creep compliance of polymers from spherical nanoindentation load-displacement curves. It was used to extract viscoelastic material properties from three commercially available polymers; polycarbonate (PC), polymethyl methacrylate (PMMA), and low-density polyethylene (LDPE) using three different indenter sizes. The average instantaneous modulus obtained by nanoindentation was 2.96 GPa, 2.14 GPa, and 0.265 GPa for PMMA, PC, and LDPE respectively. The average creep compliance obtained was 0.43-0.034e^(-t/20)-0.057e^(-t/220), 0.49-0.015e^(-t/12)-0.013e^(-t/150), and 6.59-1.17e^(-t/15)-1.64e^(-t/146) for PMMA, PC, and LDPE respectively. The average relaxation modulus obtained was 2.34+0.28e^(-t/17)+0.34e^(-t/192), 2.02+0.067e^(-t/12)+0.055e^(-t/145), and 0.152+0.067e^(-t/12)+0.047e^(-t/111) for PMMA, PC, and LDPE respectively. It is shown that the properties extracted were consistent across the three indenter tip sizes and similar to other results found in literature. The results suggest that viscoelastic properties can be reliably extracted at the microscale for different types of polymers using spherical nanoindentation.

Keywords

Spherical Nanoindentation; Discrete Deconvolution; Polymers; Viscoelasticity.

Article History

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1. Introduction

With advances in technology and the push for advanced engineering materials, there has been considerable interest in the past few years in understanding the micro and sub-micron scale properties of viscoelastic materials. Polymer composites are popularly used for bone cement, bone replacement, cartilage replacement, screws, microcontrollers, microactuators, and in the design of stronger, tougher, and "greener" materials (Deville *et al.*, 2006; Dunlop & Fratzl, 2010; Ebina & Mizukami, 2007; Munch *et al.*, 2008; Ramakrishna *et al.*, 2001; Walther *et al.*, 2010). To truly appreciate the mechanical properties of composites, it is important to understand the properties at the microscale where the separate constituents interact, and not just the effective properties at the macroscale. Also successful application and design of devices at the microscale can only be guaranteed if their mechanical properties at the lower length scales are known. There are various tools that characterize mechanical response of materials at the microscale (Bhushan, 2001; Fischer-Cripps, 2002; Haque & Saif, 2003; Hemker & Sharpe, 2007), but different assumptions and data analysis procedures have led to disparate results on the same materials.

Nanoindentation is commonly used to characterize the local mechanical behaviour in material systems with heterogeneous microstructures (Bhushan, 1998). To be able to apply the indentation methods to polymer composites (Kim *et al.*, 2019; Rossi, 2018), one has to first develop analysis techniques that will work for viscoelastic materials. This paper extracts the viscoelastic material properties from the load-displacement data generated from spherical nanoindentation on materials exhibiting time-dependent response at room temperature. Most studies on nanoindentation focus on extracting the relaxation modulus or creep compliance by fitting the results to a mechanical model or a Prony series representation (Cheng *et al.*, 2005; Huang & Lu, 2006; Huang & Lu, 2007). Some researchers have used sharp, Berkovich or conical, tips to investigate the viscoelastic behaviour of different polymers (Briscoe *et al.*, 1998; Jakes *et al.*, 2012). It should be noted that in these cases the region deformed under the indenter is plastically deformed and as such the properties extracted are not representative of the original undeformed viscoelastic material. Some tests extract these properties by fitting the loading portion of the test to the viscoelastic solution (Cheng & Yang, 2009; Huang & Lu, 2007) whereas others extract them by ramping the load or displacement to a preset number and holding it for some time (Oyen, 2005; Peng *et al.*, 2012). The relaxation modulus or creep compliance can then be extracted from the hold portion.

In this paper discrete deconvolution has been used to extract the viscoelastic material properties from the load-displacement data on three commercially available polymers; polycarbonate (PC), polymethyl methacrylate (PMMA), and low-density polyethylene (LDPE) using three different indenter sizes. The properties obtained from nanoindentation are compared against results obtained from conventional uniaxial compression tests performed on the same polymers at a similar strain rate.

2. Constitutive Relationships

The constitutive equations, for stress and strain, that describe the fundamental viscoelastic material behaviour at any time ‘t’ is usually expressed as a convolution integral (Lakes, 2009):

$$\sigma(t) = \int_0^t E(t-\tau)\frac{d\varepsilon(t)}{d\tau}d\tau \quad (1)$$

with a complimentary relation as:

$$\varepsilon(t) = \int_0^t J(t-\tau)\frac{d\sigma(t)}{d\tau}d\tau \quad (2)$$

where σ(t) is the stress response or applied stress, ε(t) is the strain response or applied strain, E(t) is the relaxation modulus and J(t) is the creep compliance. The relationship between the relaxation modulus and the creep compliance can be represented as (Lakes, 2009),

$$\int_0^t J(t-\tau)E(\tau)d\tau = \int_0^t E(t-\tau)J(\tau)d\tau = t \quad 3)$$

If the material response to a step strain or stress can be determined experimentally, the response of a linear viscoelastic material to any load or displacement history can be found. Experimentally applying a step strain or stress is not feasible, so the usual procedure is to apply the stress or strain at the fastest rate that the machine allows and hold it at a pre-set value. This ramp loading can be taken into consideration when extracting the relaxation modulus or creep compliance. These are the implicit relationships. If specific analytical forms are known for the relaxation function or creep function, the explicit relationship can be formulated using Laplace transforms as will be developed for the viscoelastic nanoindentation case.

2.1 Prony Series

A general representation of the relaxation modulus can be obtained by connecting many Maxwell elements in series, and adding a spring in parallel with the whole array. The relaxation modulus will then have either of the two forms (Lakes, 2009):

$$E(t) = E_\infty + \sum_{i=1}^{N} E_i e^{-t/\tau_i} \quad (4)$$

$$E(t) = E_0 - \sum_{i=1}^{N} E_i \left(1 - e^{-\frac{t}{\tau_i}}\right) \quad (5)$$

where E_0 is the instantaneous modulus, E_∞ is the steady state stiffness of the system, and τ_i and E_i are the time constants and stiffnesses of the Maxwell elements. The sum of the exponentials is known as the Prony series and is generally used by finite element modelling software to define the properties of time dependent materials. Experimental data from stress relaxation tests can also be fit to equation (4) to determine the Prony series terms. For cases where only creep data is available, the creep compliance can be written in Prony series representation as:

$$J(t) = J_\infty - \sum_{i=1}^{N} J_i e^{-t/\tau_i} \quad (6)$$

$$J(t) = J_0 + \sum_{i=1}^{N} J_i(1 - e^{-\frac{t}{\tau_i}}) \quad (7)$$

Where, J_∞ is the steady state compliance of the system, J_0 is the instantaneous compliance, and J_i and τ_i are the compliances and time constants of the Maxwell elements. If only one function is known, the relationship between the relaxation function and the creep function, described by equation (3), can be used to obtain the other. The shear and bulk relaxation moduli can also be represented in a similar form if either time series is known. The Prony series is just an example of the most common method of describing viscoelastic behaviour and is normally used to model a wide range of materials and relaxation times by using the appropriate number of elements.

An important study was carried out by Oyen (2005) to study the assumption of step loading creep conditions. Experimentally, a step loading condition is impossible to implement, therefore it is important to take into account the ramp load before the hold. Oyen obtained the analytical solution for this case and that if a mechanical model is used, a “ramp correction factor” for the exponential decay terms is the only difference between a ramp loading condition and an analytical step loading condition.

2.2 Viscoelastic Nano indentation

In traditional indentation, an indenter of predefined geometry is pushed into a weaker sample by applying a set displacement or force. An optical image is then taken of the indent; the resultant imprint is measured and correlated to a hardness index number (Bhushan, 1998). With high resolution equipment, it is possible to continuously monitor and control the load and displacement of the indenter to produce load-displacement curves. This is known as depth sensing indentation or instrumented indentation testing and for sub-micron resolutions, nanoindentation. Nanoindentation has significant advantages over the traditional indentation, since the material properties can now be probed from depths as small as a few nanometers, using proper analysis techniques (Fischer-Cripps, 2002).

Theoretical studies on linear viscoelastic indentation started in the mid-1950s with the works of Lee (1956), Hunter (1960), Lee and Radok (Lee & Radok, 1960), Graham (1965), and Ting (1966). They developed an approach for finding the viscoelastic time dependent indentation solution in cases where the corresponding solution for the purely elastic case has already been solved. This is known as the viscoelastic correspondence principle. This principle for viscoelastic indentation was first derived by Lee and Radok (1960) and then later developed for more general cases by Ting (1966).

The solution to viscoelastic nanoindentation is given by using the viscoelastic operators from the constitutive relationships for viscoelasticity given by equations (1) and (2) and placing the equations in terms of load (P(t)) and displacement (h(t)) to get (Abba, 2015):

$$P(t) = \frac{4\sqrt{R_{eff}}}{3(1-\nu^2)}\int_0^t E(t-\tau)\frac{dh^{3/2}(\tau)}{d\tau}d\tau \quad (8)$$

for a prescribed arbitrary displacement history and

$$h^{3/2}(t) = \frac{3(1-\nu^2)}{4\sqrt{R_{eff}}}\int_0^t J(t-\tau)\frac{dP(\tau)}{d\tau}d\tau \quad (9)$$

for a prescribed arbitrary loading history. Where, the Poisson’s ratio (ν) is assumed to be constant, R_{eff} is the effective curvature of the two bodies in contact and E_{eff} is the effective modulus of the combined system.

$$\frac{1}{R_{eff}}=\frac{1}{R_1}+\frac{1}{R_2};\ \frac{1}{E_{eff}}=\frac{1-\nu_1^2}{E_1}+\frac{1-\nu_2^2}{E_2} \quad (10)$$

Equations (8) and (9) are commonly used to analyse load-displacement curves obtained from nanoindentation experiments on viscoelastic materials.

3. Discrete Deconvolution

The solution to the indentation problem on a linear viscoelastic material is in terms of a convolution integral. Since in a typical nanoindentation test, the displacement and load are measured, the relaxation modulus or creep compliance can ideally be deconvoluted from the viscoelastic solutions. The data obtained from the indenter is discrete, and therefore, the convolution has to be in a discrete form to be deconvoluted. The general form of the convolution integral is:

$$b(t)=\int_0^t A(\tau)x(t-\tau)\,d\tau \quad (11)$$
$$=\int_0^t A(t-\tau)x(\tau)\,d\tau$$
$$=A(t)*x(t)$$

where '*' is the convolution operator and x(t) is assumed to be the unknown parameter to be deconvoluted. To approximate the integral by a summation, let ΔT be the sampling interval, and then the discrete form is given by:

$$b(n\Delta T)=\sum_{m=0}^{n\Delta T}A(n\Delta T-m\Delta T)x(m\Delta T)\,\Delta T \quad (12)$$

Each function is now a discrete array and the nth element is the function evaluated at nΔT. Thus the discrete convolution can be written as:

$$b(n)=\Delta T\sum_{m=0}^{n}a(n-m)x(m) \quad (13)$$
$$=a(n)*x(n)$$

The summation can be expanded to get:

$$b(n)=\begin{bmatrix}a(0)x(0)\\ a(0)x(1)+a(1)x(0)\\ a(0)x(2)+a(1)x(1)+a(2)x(0)\\ \vdots\\ a(0)x(n)+a(1)x(n-1)+\cdots+a(n)x(0)\end{bmatrix}\Delta T \quad (14)$$

or in matrix form:

$$\begin{bmatrix}a(0) & & & \\ a(1) & a(0) & & \\ \vdots & & \ddots & \\ a(N) & \cdots & \cdots & a(0)\end{bmatrix}\begin{bmatrix}x(0)\\ x(1)\\ \vdots\\ x(N)\end{bmatrix}\Delta T=\begin{bmatrix}b(0)\\ b(1)\\ \vdots\\ b(N)\end{bmatrix} \quad (15)$$

$$AX\Delta T=B$$

where:

$$A_{ij}=\begin{cases}0 & i<j\\ a(i-j) & \text{otherwise}\end{cases}$$
$$X_i=x(i-1)$$
$$B_i=b(i-1)$$

An estimate $\hat{X}$ of the unknown parameter can be calculated by using a linear least squares approach to a solution:

$$\hat{X}=(A^TA)^{-1}A^TB \quad (16)$$

For the viscoelastic nanoindentation solution, once the creep compliance or relaxation modulus is extracted, the estimated solution can be fit to a Prony series to get an idea of the material's instantaneous and long-term moduli or compliance.

3.1 Extracting Viscoelastic Property

The creep test is performed by ramping the load to a specified value, holding the load for a set time, and unloading at the same rate, as shown in Figure 1. The relaxation test can be performed by controlling displacement instead of load, as shown in Figure 1. The data from the creep tests will be used to extract the viscoelastic property of the materials.

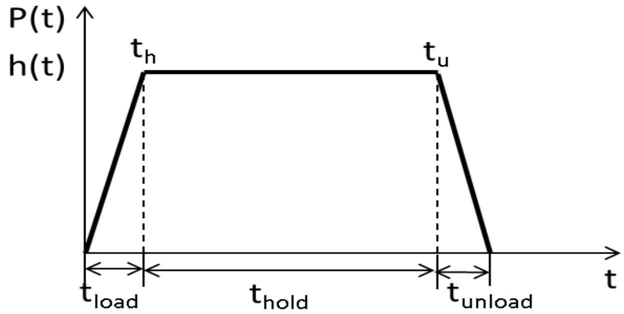

Figure 1: Schematic for performing a creep or relaxation test

When performing a ramp and hold test, the method presented by Oyen (Oyen, 2005) is used to analyse the data obtained. The loading conditions can be written as:

$$P(t)=kt \quad 0\le t\le t_h$$
$$P_{max}=kt_h \quad t_h<t\le t_u \quad (17)$$

where t_h is the time it takes to reach the maximum load and k is the loading rate. The second term in equation (17) is then a constant. This means that the viscoelastic equation for creep (equation (9)) must be solved twice, once for the ramp and again for the hold. The Prony series representation of the creep compliance, equation (6), can be used and the integrals solved to obtai:

$$h^{\frac{3}{2}}(t)=\frac{3(1-\nu)}{8\sqrt{R}}\int_0^{t_h}J(t-\tau)\,kd\tau \quad 0\le t\le t_h$$
$$=\frac{3(1-\nu)k}{8\sqrt{R}}\left[J_\infty t-\sum_{i=1}^{N}j_i\tau_i\left(1-e^{-t/\tau_i}\right)\right]$$
$$h^{\frac{3}{2}}(t)=\frac{3(1-\nu)}{8\sqrt{R}}\left[\int_0^{t_h}J(t-\tau)\,kd\tau+\int_{t_h}^{t}J(t-\tau)\,0d\tau\right] \quad (18)$$
$$h^{\frac{3}{2}}(t)=\frac{3(1-\nu)}{8\sqrt{R}}\int_0^{t_h}J(t-\tau)\,kd\tau \quad t_h\le t\le t_u$$
$$=\frac{3(1-\nu)k}{8\sqrt{R}}\left[J_\infty t_h-\sum_{i=1}^{N}j_i\tau_i e^{-t/\tau_i}\left(e^{-t_h/\tau_i}-1\right)\right]$$

The discrete deconvolution procedure is then used, where the convolution to be solved is given by equation (15) with the terms obtained from equation (18). The unknown in this case is either creep compliance represented by array X with the terms in matrix A derived from dP/dt as a constant value. The output in array B is then represented by the displacement as $h^{3/2}$. The hold portion of the nanoindentation test is used to get the creep compliance function. This method takes into account the ramping it takes to achieve the holding load rather than assuming a step load. The relaxation modulus can then be extracted by using equation (3) and deconvoluting the expression.

4. Materials and Methods

4.1 Sample Preparation

For nanoindentation, the specimens were obtained from a 31.75 mm diameter extruded rod for PMMA (density 1.19 g/cm^3; glass transition temperature 105°C), PC (density 1.25 g/cm^3; glass transition temperature 145°C), and LDPE (density 0.91 g/cm^3; glass transition temperature -125°C). For compression tests, the PMMA and PC specimens were extruded rods of approximately 15.875 mm diameter and 27.94 mm length and the LDPE specimens were extruded rods of approximately 25.4 mm diameter and 50.8 mm length. Each PMMA specimen was annealed at 110°C and each PC specimen was annealed at 150°C. All the specimens were annealed in a Thermo Scientific Lindberg/Blue M™ Moldatherm™ box furnace (Waltham, MA, USA) for two hours and then slowly cooled down to room temperature at a rate of 5°C/hr. The nanoindentation specimens were cut perpendicular to the extruded direction using an Allied TechCut 5™ (Rancho Dominguez, CA, USA) precision sectioning machine and then polished using silicon carbide paper of decreasing grit size (320, 800, 1200, 2400, and 4000 grit) using a Struers Tegramin-30 (Cleveland, OH, USA). Each polishing step except the 4000 grit was performed for 2 minutes, followed by washing to remove debris. The 4000 grit was performed for 6 minutes. This was followed by polishing with a 1 μm alcohol based diamond suspension (Struers DP-Suspension) for 20 minutes and a 0.05 μm colloidal silica suspension (Buehler MasterMet) for 20 minutes.

4.2 Uniaxial Compression Tests

Compression tests were performed on an MTI Phoenix Universal Testing Machine with a 20,000 lb. load cell. Tests were performed according to ASTM standard D695 for testing plastics (ASTM D695-10, 2010) at a speed of 0.05 in/min. Displacement was measured using a capacitance gage and all load-displacement data was converted to true stress-strain curves. At least five samples were tested for each material with the data reported as mean ± standard error.

4.3 Spherical Nanoindentation

The indentation tests were performed on an Agilent G200 (Keysight Technologies Inc., Santa Rosa, CA) Nano Indenter with an XP head. The indenter has a maximum load of 500 mN with a high load option of 10 N and a load resolution of 50 nN. The maximum indentation depth achievable is greater than 500 μm with a resolution of less than 0.01 nm. The indents are performed on the polished surfaces of the polymers and the spherical diamond tips used have a radius of 16 μm, 100 μm, and 1500 μm. Since changes in temperature can cause expansion or shrinkage of materials leading to errors in measurement, the indenter measures a thermal drift before each test by holding the indenter on the surface of the material and measuring any changes in displacement. All tests were performed after the measured indenter drift rate reached and maintained a value of 0.05 nm/s.

To extract the viscoelastic properties ramp and hold tests were performed using the 16 μm, 100 μm, and 1500 μm indenter tips. For each material the load was increased to three different loads at a constant loading rate and that load was held for 300 seconds and then unloaded at a constant unloading rate. The viscoelastic properties were extracted from the hold portion.

5. Results and Discussion

5.1 Uniaxial Compression Tests

The load and displacement values from the compression tests were converted to true stress strain curves, as shown in Figure 2, and the results analysed (Abba, 2015). The moduli, measured from the initial linear portion of the true stress-strain curves, were 1.9 ± 0.031 GPa, 1.42 ± 0.031 GPa, and 0.154 ± 0.0014 GPa for PMMA, PC, and LDPE respectively. While the compressive modulus values for PMMA and LDPE are acceptable compared to reported values of 2.1 GPa and 0.165 GPa respectively, the value for PC is at least 0.5 GPa lower than the expected 2 GPa. This disparity may primarily be due to the thermo-mechanical history that the material experienced before uniaxial compression testing. These results will be compared to the indentation properties extracted.

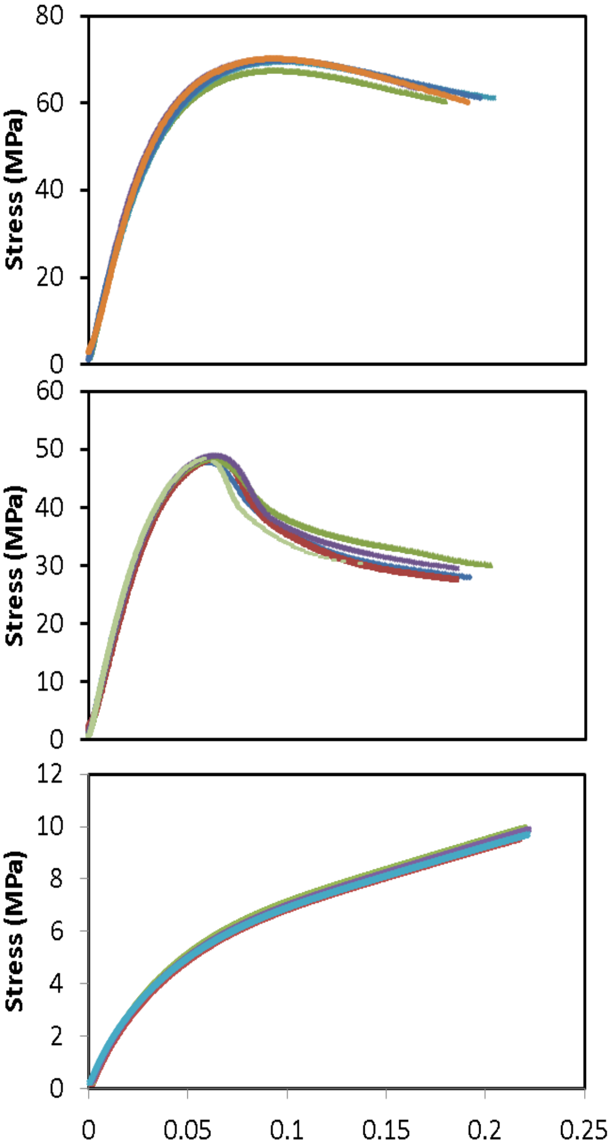

Figure 2: True stress-strain curves from compression tests on a) PMMA, b) PC, and c) LDPE

5.2 Spherical Nanoindentation

The results from the creep tests using the 100 µm radius indenter are shown in Figure 3. LDPE has more pronounced viscoelastic properties with the largest increase in displacement, while PC has the least, making it close to an elastic material. These results will be used to extract the viscoelastic properties. The same tests were also performed using the 16 µm and 1500 µm radius indenter at different loads.

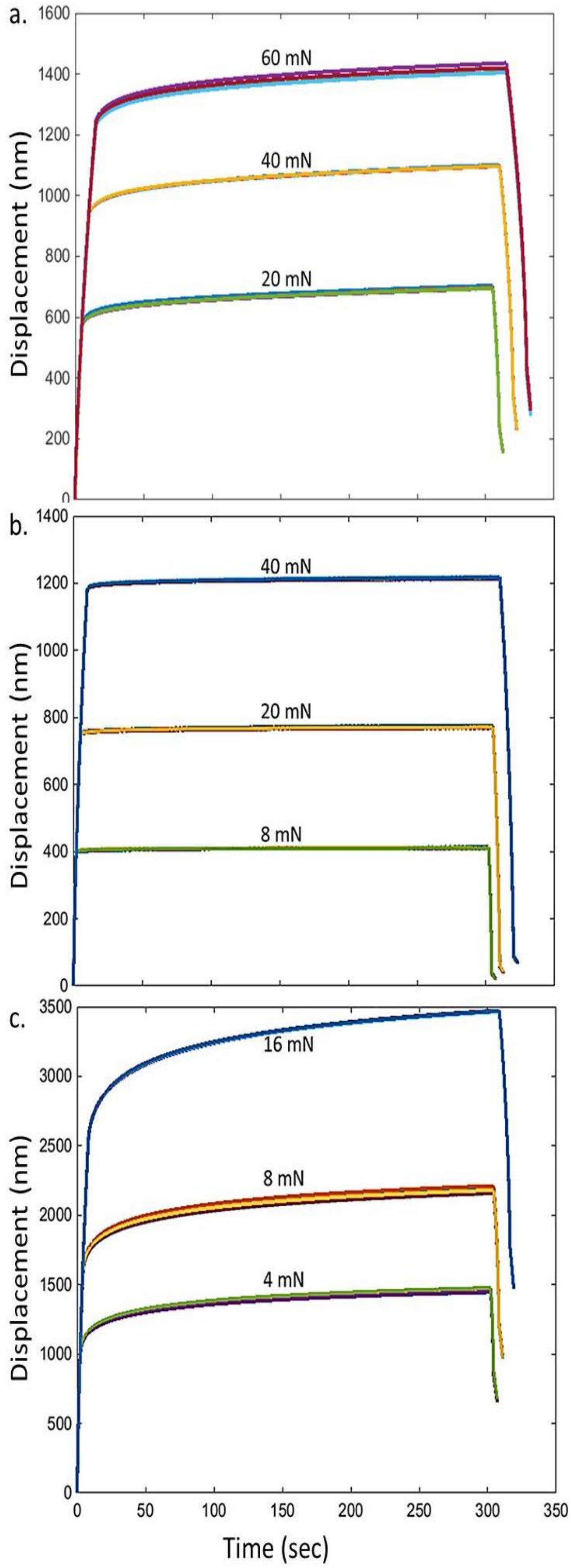

Figure 3 - Displacement vs time for creep tests performed on a. PMMA at holding loads of 20 mN, 40 mN, and 60 mN, b. PC at holding loads of 8 mN, 20 mN, and 40 mN, and c. LDPE at holding loads of 4 mN, 8 mN, and 16 mN.

5.3 Viscoelastic Properties

The relaxation modulus and corresponding creep compliance were extracted using the methods in section 3. The viscoelastic properties can be represented in terms of a two-term Prony series as:

$$E(t) = E_{\infty} + E_1 e^{-\frac{t}{\tau_{k1}}} + E_2 e^{-\frac{t}{\tau_{k2}}}$$

$$J(t) = J_{\infty} - J_1 e^{-\frac{t}{\tau_{j1}}} - J e^{-\frac{t}{\tau_{j2}}} \quad (19)$$

Where the terms in front of the exponentials are the Prony series terms, E_∞ is the long term relaxation modulus, J_∞ is the long term creep compliance, and τ is the time constant. The instantaneous creep compliance, J_0, can be obtained by subtracting the Prony series terms from the long term creep compliance and the instantaneous relaxation modulus, E_0, can be obtained by adding the Prony series terms to the long-term relaxation modulus. These results are presented in Table 1. The compressive moduli obtained by uniaxial compression was compared to the instantaneous moduli obtained by nanoindentation. The average instantaneous modulus obtained by nanoindentation was 2.96 GPa, 2.14 GPa, and 0.265 GPa for PMMA, PC, and LDPE respectively. These results show that the compressive moduli were lower than the indentation moduli for all three materials. This is expected as the zone of indentation is constrained by the material surrounding it. This effect usually leads to an increase in modulus and yield strength (Spitzig & Richmond, 1979). Also a much smaller volume of material is being probed leading to more local properties compared to the average properties obtained by uniaxial compression. The average ratio between the compressive moduli and indentation instantaneous moduli was 1.6 for PMMA, 1.5 for PC and 1.7 for LDP. This ratio can be used to approximate the expected indentation instantaneous modulus for new viscoelastic materials if the compressive modulus is known.

Table 1: Extracted creep compliance and relaxation modulus for the materials studied using three different indenter tip radii

S/N	Material	Indenter Size (µm)	Creep Compliance (1/GPa)	Relaxation Modulus (GPa)
1	PMMA	16	$0.43-0.032e^{-t/18}-0.060e^{-t/216}$	$2.32+0.26e^{-t/16}+0.37e^{-t/186}$
2		100	$0.45-0.044e^{-t/15}-0.076e^{-t/225}$	$2.23+0.37e^{-t/13}+0.44e^{-t/187}$
3		1500	$0.41-0.025e^{-t/25}-0.034e^{-t/220}$	$2.46+0.20e^{-t/23}+0.22e^{-t/202}$
4	PC	16	$0.47-0.012e^{-t/15}-0.010e^{-t/136}$	$2.11+0.059e^{-t/15}+0.047e^{-t/133}$
5		100	$0.49-0.013e^{-t/9}-0.014e^{-t/151}$	$2.03+0.059e^{-t/9}+0.058e^{-t/146}$
6		1500	$0.52-0.021e^{-t/11}-0.016e^{-t/162}$	$1.92+0.084e^{-t/11}+0.061e^{-t/157}$
7	LDPE	16	$6.48-1.22e^{-t/17}-1.58e^{-t/154}$	$0.154+0.072e^{-t/13}+0.046e^{-t/118}$
8		100	$6.76-1.12e^{-t/14}-1.61e^{-t/138}$	$0.148+0.057e^{-t/11}+0.043e^{-t/106}$
9		1500	$6.54-1.18e^{-t/15}-1.73e^{-t/146}$	$0.153+0.072e^{-t/11}+0.051e^{-t/108}$

The average creep compliance obtained from Table 1 is $0.43 - 0.034e^{-\frac{t}{20}} - 0.057e^{-t/220}$, $0.49 - 0.015e^{-\frac{t}{12}} - 0.013e^{-t/150}$, and $6.59 - 1.17e^{-\frac{t}{15}} - 1.64e^{-t/146}$ for PMMA, PC, and LDPE respectively. The average relaxation modulus obtained from Table 1 is $2.34 + 0.28e^{-\frac{t}{17}} + 0.34e^{-t/192}$, $2.02 + 0.067e^{-\frac{t}{12}} + 0.055e^{-t/145}$, and $0.152 + 0.067e^{-\frac{t}{12}} + 0.047e^{-t/111}$ for PMMA, PC, and LDPE respectively. The relaxation moduli obtained by Huang and Lu (2006) were $E(t) = 2.3343 + 0.1607e^{-0.1t} + 0.2574e^{-0.01t}$ for PMMA and $E(t) = 1.4531 + 0.0681e^{-0.1t} + 0.1359e^{-0.01t}$ for PC. The results for PMMA are comparable to the ones obtained in this work, with that of PC being lower as observed with the compressive modulus. To the best of our knowledge there is no reported indentation creep compliance or relaxation modulus for LDPE.

5. Conclusions

Viscoelastic properties were extracted from spherical nanoindentation using three different indenter tip sizes on commercially available polymers. This was carried out by loading the material at a constant loading rate and then holding a specified load for 300 seconds before unloading. Using a loading ramp that has a constant loading rate was convenient because an analytical form of the viscoelastic equation that does not include an integral can be formed. This was picked over a displacement controlled method since that results in a relationship that cannot be solved analytically. The creep compliance was obtained by discrete deconvolution of the numerical form of the viscoelastic indentation equation. The relaxation modulus was obtained by discrete deconvolution of the relationship between the relaxation modulus and the creep compliance. Properties obtained were compared against all three tips and with results from compression tests on the same materials.

The average instantaneous modulus obtained by nanoindentation was 2.96 GPa, 2.14 GPa, and 0.265 GPa for PMMA, PC, and LDPE respectively. The average creep compliance obtained from was $0.43 - 0.034e^{-\frac{t}{20}} - 0.057e^{-t/220}$, $0.49 - 0.015e^{-\frac{t}{12}} - 0.013e^{-t/150}$, and $6.59 - 1.17e^{-\frac{t}{15}} - 1.64e^{-t/146}$ for PMMA, PC, and LDPE respectively. The average relaxation modulus obtained was $2.34 + 0.28e^{-\frac{t}{17}} + 0.34e^{-t/192}$, $2.02 +$

$0.067e^{-\frac{t}{12}} + 0.055e^{-t/145}$, and $0.152 + 0.067e^{-\frac{t}{12}} + 0.047e^{-t/111}$ for PMMA, PC, and LDPE respectively. While the indentation viscoelastic material properties were consistent across all the indenter sizes, the ratio between the instantaneous indentation modulus and the compressive modulus was about 1.6 for all three materials. The results obtained in these studies allow us to draw several important conclusions for the mechanical characterization of viscoelastic materials using spherical nanoindentation.

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