Experimental Mechanics (2007) 47: 99–105
DOI 10.1007/s11340-006-8276-5

# Multiscale Creep Compliance of Epoxy Networks at Elevated Temperatures

**T. F. Juliano · M. R. VanLandingham · C. A. Tweedie · K. J. Van Vliet**

Received: 31 October 2005 / Accepted: 13 March 2006 / Published online: 12 May 2006


**Abstract** Epoxy networks are thermoset polymers for which an important structural length scale, molecular weight between crosslinks ($M_c$), influences physical and mechanical properties. In the present work, creep compliance was measured for three aliphatic epoxy networks of differing $M_c$ using both macroscale torsion and microscale depth-sensing indentation at temperatures of 25 and 55°C. Analytical relations were used to compute creep compliance ($J(t)$) for each approach; similar results were observed for the two techniques at 25°C, but not at 55°C. Although creep compliance measurement differed at elevated temperatures, there were clear correlations between $M_c$, glass transition temperature, $T_g$, and the observed time-dependent mechanical behavior via both techniques at 55°C, but these correlations could not be seen at 25°C. This work demonstrates the capacity of depth-sensing indentation to differentiate among epoxy networks of differing structural configurations via $J(t)$ for small material volumes at elevated temperatures.

**Keywords** Spherical indentation · Linear viscoelastic · Shear bending

T. F. Juliano · M. R. VanLandingham (✉)
United States Army Research Laboratory,
Aberdeen Proving Ground,
Aberdeen, MD 21005, USA
e-mail: mark.vanlandingham@arl.army.mil

C. A. Tweedie · K. J. Van Vliet
Department of Materials Science and Engineering,
Massachusetts Institute of Technology,
Cambridge, MA 02139, USA

## Introduction

Depth-sensing indentation is a technique used to measure mechanical properties of small volumes of materials [1, 2]. Routine indentation data analysis yields time-independent elastic modulus and hardness values. Additionally, a number of efforts have been made to measure time-dependent mechanical properties on polymeric materials using indentation with some success [3–9]. However, comparisons to measurements made at the macroscale is an important check of such microscale measurements to ensure that volumes of material not able to be tested on the macroscale, including thin coatings or composites, may be adequately characterized using indentation techniques.

Epoxy resins are used in a number of applications including coatings, adhesives, composites, and electrical insulation [10–13]. The composition and network structure of these cross-linked materials can vary greatly, yielding a range of different properties. Variation of the mechanical and thermal properties of epoxy networks can be achieved by modifying the molecular weight between crosslinks, $M_c$ [14]. In the previous work of Crawford and Lesser [14], reductions in the values of $M_c$ in a series of model aliphatic epoxies produced the expected increase in glass transition temperature, $T_g$, as well as an increase in the critical yield stress measured above $T_g$. In the current work, three of the aliphatic epoxy systems studied by Crawford and Lesser are used to characterize network viscoelastic behavior on the macroscale by conventional means and on the microscale using depth-sensing indentation.

## Theory

To measure the creep compliance within the linear viscoelastic regime, time-dependent equations based in elasticity theory of a rectangular beam found in [15] are utilized. A shear stress, $\tau$, is applied to the samples and the resulting shear strain as a function of time, $\gamma(t)$, is measured. Constrained boundaries are assumed. The viscoelastic creep compliance, $J(t)$, is defined as

$$J(t) = \left|\frac{\gamma(t)}{\tau}\right|, \tag{1}$$

Shear strain as a function of time is

$$\gamma(t) = \frac{\phi(t)}{l}\sqrt{\left(\frac{k}{2}\right)^2 + \left(\frac{w}{2}\right)^2} \tag{2}$$

and shear stress is

$$\tau = \frac{3M}{wk^3}\left(1 - \frac{192k}{\pi^5 w}\tan h\left(\frac{\pi w}{2k}\right)\right)^{-1}\sqrt{\left(\frac{k}{2}\right)^2 + \left(\frac{w}{2}\right)^2}, \tag{3}$$

where $\phi$ is the measured angular displacement of the bar, $l$ is the sample length, $k$ is the sample thickness, $w$ is the sample width and $M$ is the measured applied torque to the bar.

For measuring $J(t)$ using depth-sensing indentation, a time-dependent version of Hertz's elastic theory is considered [16]. Hertz's original equation for elastic displacement into a surface is

$$h_e = \left(\frac{3P}{4E^*}\right)^{2/3}\left(\frac{1}{R}\right)^{1/3}, \tag{4}$$

where $P$ is the load applied to the indenter, $R$ is the spherical radius of the indenter tip, and $E^*$ is the reduced modulus of the indented material, defined as

$$E^* = \left[\frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i}\right]^{-1}, \tag{5}$$

where $\nu$ and $E$ are the Poisson's ratio and elastic modulus of the indented material, respectively, and the subscript $i$ refers to properties of the indenter. Substituting equation (5) into (4) and assuming that the material property term is much greater than the indenter property term in equation (5), elastic modulus is given by

$$E = \frac{3P(1-\nu^2)}{4\sqrt{R}h_e^{3/2}}. \tag{6}$$

Assuming the material is isotropic and the Poisson's ratio is time independent, shear modulus is related to elastic modulus by

$$G = \frac{E}{2(1+\nu)}. \tag{7}$$

Because shear compliance is the inverse of the shear modulus for an elastic material (i.e., $J = G^{-1}$), combining equations (6) and (7) leads to the relation

$$J = \frac{8\sqrt{R}h_e^{3/2}}{3P(1-\nu)}. \tag{8}$$

In a traditional creep compliance test, the stress is held constant with time and the strain is monitored. However, for indentation creep testing, the load, $P$, which is estimated to be proportional to the indentation stress, is held constant and $h_e$, which is related to strain, is monitored with time. Restating equation (8) as a function of time,

$$J(t) = \frac{8\sqrt{R}h_e^{3/2}(t)}{3P_0(1-\nu)}. \tag{9}$$

This relation has also been classically derived using more rigorous approaches [17, 18], and applicability to viscoelastic property measurement has been discussed [17–22]. In general, equation (9) is only valid in the linear viscoelastic regime. Hence, only indenter geometries capable of generating the small contact strains necessary to maintain this condition, e.g., large spheres, are suitable for measurement of creep compliance to maintain linear viscoelastic deformation within the force and displacement resolution of available instrumentation.

## Experimental

Depth-sensing indentation measurements were made on three model aliphatic epoxy systems, previously studied by Crawford and Lesser [14]. Each epoxy was cured with different proportions of the two aliphatic curing agents ethylenediamine and *N,N′*-dimethylethylenediamine. After mixing the amines with the epoxy resin, networks were cured between two glass plates, which were treated with a silating agent to prevent adhesion, being spaced approximately 3 mm apart. After full cure was determined via differential scanning calorimetry, the epoxies were cut with a diamond wheel dicing saw to pieces suitable for both torsion and indentation measurements. The three aliphatic epoxies considered herein exhibited $M_c$ values of 596 g/mol (M596), 818 g/mol (M818) and 1,452 g/mol (M1452) and $T_g$ values of 107, 86 and 67°C, respectively. Complete details of processing and standard characterization measurements for these materials can be found

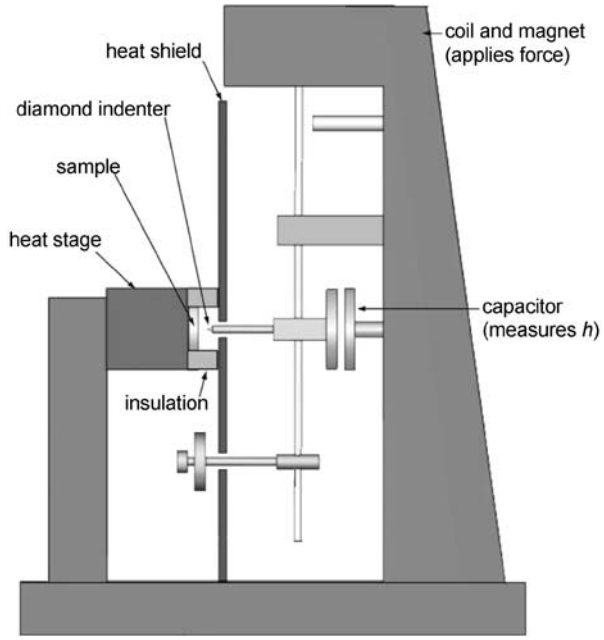


**Fig. 1** Setup of the depth-sensing indenter, equipped with temperature control, used for creep compliance experiments. In this design, heat applied to the tip and sample will rise into the air and not into the electronics, providing more robust measurement accuracy at elevated temperatures

in [14]. Root mean square surface roughness at room temperature was measured via contact mode scanning probe microscopy (3DMFP, Asylum Research) and ranged from 29.6 nm (M1452) to 79.4 nm (M596).

One sample for each epoxy material was tested in torsion in an Advanced Rheometer 2000 (TA Instruments, Delaware, USA). The bars were approximately 3 mm thick, 12 mm wide, and 50 mm long, as measured via calipers. In order to erase possible aging effects prior to testing, each epoxy sample was annealed inside a nitrogen-purged oven at ~20°C above the corresponding $T_g$ for 30 min. After annealing, the samples were allowed to cool to room temperature in open air. Prior to operation, the rheometer was calibrated for measurement of instrument inertia, geometry inertia, air bearing friction, rotational mapping, and zero gap measurement. Torsion samples were fixed in place using two clamps that were tightened by screws with a torque wrench. To ensure that tests were conducted in the linear viscoelastic deformation regime, an oscillatory strain sweep at 1 Hz was performed on each sample at room temperature to determine the threshold at which the shear modulus began to change with strain; this corresponded to about 0.15% shear strain. Even greater linear viscoelastic strains should be permissible at higher temperatures. Therefore, to be conservative, the maximum applied shear strain was targeted at all temperatures to be 0.1%. The applied normal force on the sample was controlled at 0.1 ± 0.1 N, which was the lowest value the machine would allow.

To find the shear stress that corresponded to 0.1% strain for all testing conditions, a stress-relaxation test, in which strain was maintained constant, was performed for a duration of 2 min at temperatures of 25 and 55°C. The actual temperature deviation was a maximum of 0.2°C from the commanded temperature. The approximate shear stress values throughout the test were noted. In between tests, at least 5 min was allotted for the sample to completely recover. Creep tests were then performed at shear stress values corresponding to 0.1% shear strain, with three trials for each condition. At the shear stress values selected, shear strain was 0.1% or below.

Indentation creep testing was performed using a depth-sensing indenter (Micro Materials, Ltd., Wrexham, UK) over a range of temperatures. This system has been shown to offer low drift and reliable results at higher temperatures on a number of materials [23]. A schematic of the instrument is shown in Fig. 1. Experiments were conducted at polymer surface temperatures of 25 and 55°C, as measured via a thermocouple on the surface. These temperatures were chosen because rheology experiments indicated that changes in creep compliance were detectable at 55°C for the epoxies considered. Each sample was mounted on a temperature controlled stage via a rigid, high temperature cement. The system was allowed to equilibrate for at least 15 min at all temperatures before experiments began, and then was stable during testing to within 0.2°C of the desired temperature.

Creep compliance experiments were conducted with a ruby spherical indenter (radius $R$ = 500 μm), measuring the change in depth $h(t)$ over 100 s upon

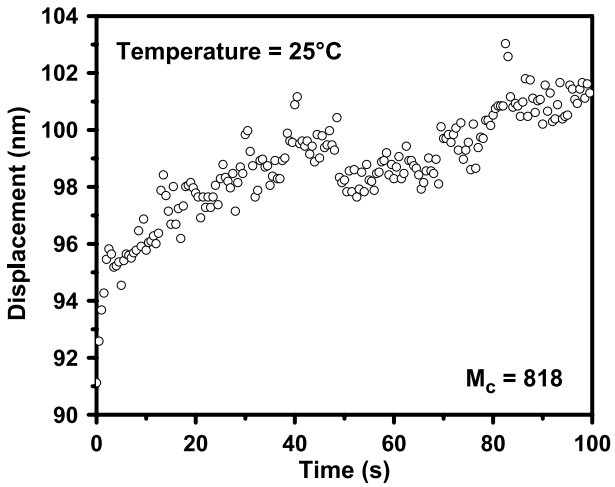


**Fig. 2** Typical displacement data as a function of time for a hold at a load of 3.8 mN on sample M818 at 25°C

application of a step-load (loading time of 0.5 s) of magnitude $P_{max}$. Creep compliance was calculated via application of equation (9) to measured $h(t)$; output for a typical test is shown in Fig. 2. Both load and displacement into the sample surface were measured continuously, and the instrument was operated in load-control. Each sample was indented in triplicate at each temperature to a contact strain level of 0.38 ± 0.002% (corresponding to 90 nm depth with contact strain defined as $\varepsilon = 0.2a/R$, where $a$ is the radius of contact at the sample surface). This level of contact strain is well below 0.8%, which has been determined to be within the linear viscoelastic regime for these three samples [24]. Experimental iteration was used to determine the load $P_{max}$ corresponding to a displacement of 90 ± 4 nm for each epoxy. Although instrument signal stability was not compromised at temperatures less than 55°C as confirmed by similar tests on nonpolymeric samples with the same indenter radius, indentation creep compliance measurements at 75°C are not included herein because repeatability of $P_{max}$ ($h$ = 90 ± 4 nm) was insufficient to allow comparison of creep responses. This may have been due to effective roughening and increased molecular mobility of epoxy surfaces nearing $T_g$. This experimental limitation in elevated temperature indentation has not been found on amorphous polymers such as polystyrene with our current setup.

## Results and Discussion

Creep compliance measured via torsion at 25°C is shown in Fig. 3. The M1452 epoxy was the least compliant, while M596 was the most compliant. However, the differences between the three materials are within about 5% difference in $J(t)$ compared to the others. There are several parameters that can affect the integrity of the measurement, including securing the sample bar completely orthogonal to the direction of the normal load, degree of uniform dimensions of the sample throughout its width or thickness, temperature calibration, and force or distance calibrations on the machine. Also, all three materials are sufficiently below the respective $T_g$s, such that they all exhibit glassy behavior. Therefore, at 25°C, the differences in $J(t)$ among the three epoxies are minimal and are similar in magnitude to measurement uncertainties.

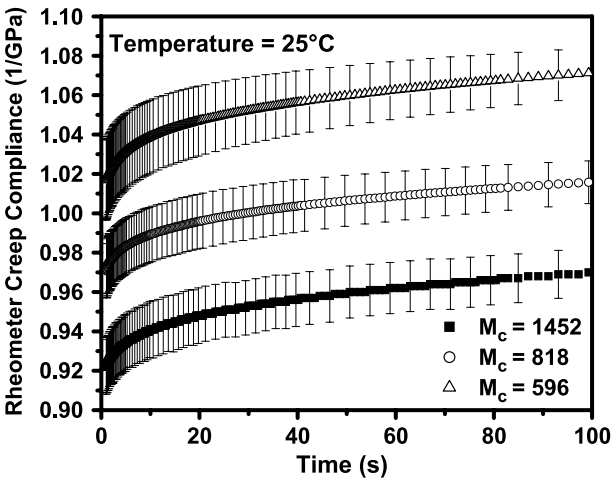


**Fig. 3** Creep compliance measurements of the three epoxy samples at 25°C from torsion tests. *Error bars* show one standard deviation between three separate test runs. Each sample has a creep compliance of about 1 $GPa^{-1}$ over 100 s of loading

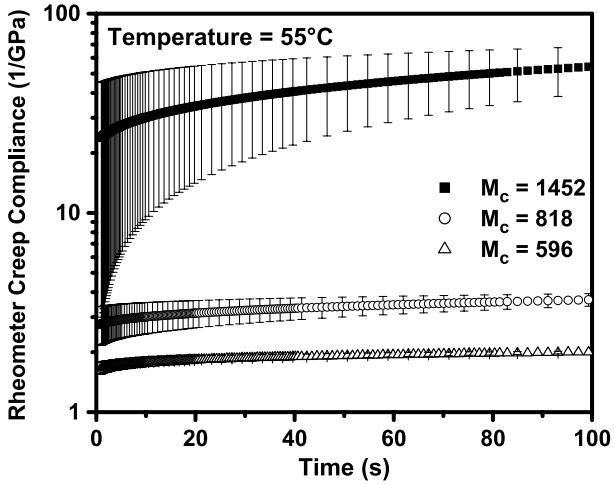


**Fig. 4** Creep compliance measurements of the three epoxy samples at 55°C from torsion tests. *Error bars* show one standard deviation between three separate test runs. M1452 is clearly more compliant than the other samples, followed by M818

At 55°C, the networks began to exhibit significant differences in $J(t)$ behavior, as shown in Fig. 4. Values of $J(t)$ differ from one material to the next by at least 100%. In particular, M1452 exhibits a much higher degree of time-dependence under loading as compared to at 25°C. This shift in behavior occurs below the determined $T_g$ of 67°C. However, the glass transition temperature represents a range of temperatures about which the molecular side chains gain mobility, and mechanical softening can occur when the temperature is less than $T_g$. Tests performed at 75°C, shown in Fig. 5, distinguish the differences between the time-dependent mechanical responses of these materials to an even greater degree. Even though 55°C is below the $T_g$ of all samples and M818 and M596 were at least 30°C below their corresponding $T_g$ values, the effect of $M_c$ on $J(t)$ becomes evident, as $J(t)$ increases for higher $M_c$ values; i.e., lower cross-link density correlates with increased mechanical compliance. This is consistent with the trend found previously for natural rubbers of

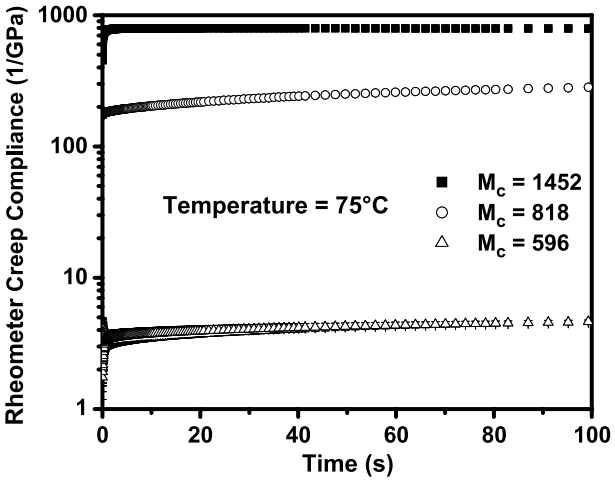


**Fig. 5** Creep compliance measurements of the three epoxy samples at 75°C from torsion tests. *Error bars* show one standard deviation between three separate test runs. For this condition, creep compliance measurements between the three samples are distinguished much more than at 55 or 25°C

varying $M_c$ [25, 26], as well as for glassy polyimide systems [27]. The creep compliance rate, $dJ(t)/dt$ also increased for each material relative to 25°C. Note that as $J(t)$ increased by an order of magnitude with increasing temperature, the variance in acquired data also increased considerably. This additional variance may be due to the resolution of the applied torque or the influence of the normal load on the sample (up to 0.2 N), as the material begins to transition from glassy to rubbery.

Creep compliance measured via depth-sensing indentation at 25°C is shown in Fig. 6. The values of $J(t)$ for the epoxies were similar and could not be statistically separated outside of the data scatter. This result was further verified using a different indentation instrument (Nano Indenter XP, MTS Systems). The average values of $J(t)$ for each sample measured at the macroscale via torsion and at the microscale via indentation were within ~10%. However, creep compliance rate and thus the change in creep compliance over the 100 s hold period were markedly different. Whereas torsion data showed an increase over 100 s of only about 0.03 $GPa^{-1}$, indentation detects about a 0.1 $GPa^{-1}$ increase—about a factor of three higher than that measured at the macroscale. This difference may be due in part to drift in instrumentation signals and/or to the difference in applied strain levels between the two techniques. For indentation creep compliance measurements, the total increase in $h(t)$ was on the order of 10 nm. The typical magnitude in drift of $h(t)$ over 100 s is ≤3 nm, and this measurement error could result in as much as a 50% increase in $J(t)$. In addition, contact strains imposed via indentation (~0.4%) exceeded those imposed via torsion (~0.1%), although previous testing indicated that linear viscoelasticity was maintained for contact strains less than 0.8%. However, a more significant issue might be potential errors introduced via indenter contact with surface asperities. Surface roughness on the order of tens of nanometers can affect the evolution of $h(t)$ when indenting with a sphere that has a micron-scale radius. As the sphere radius increases, the ratio of contact area to depth increases. Therefore, the differences observed between the creep of near-surface and bulk volumes for the epoxy samples is likely due to surface roughness and the use of a 500 μm radius spherical tip:

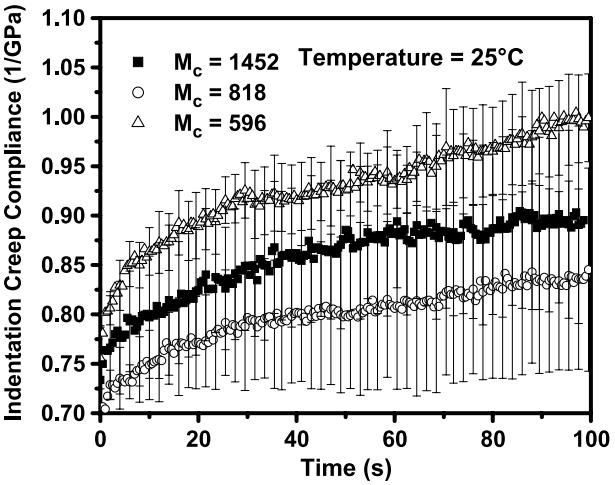


**Fig. 6** Creep compliance measurements of the three epoxy samples at 25°C using indentation. *Error bars* show one standard deviation between three separate test runs. Each measurement is within scatter of one another

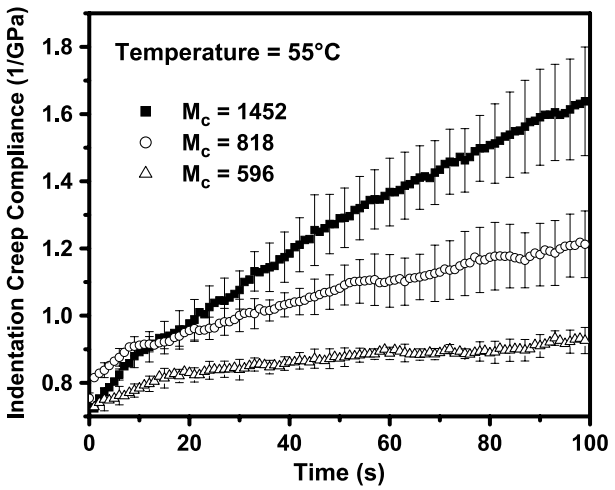


**Fig. 7** Creep compliance measurements of the three epoxy samples at 55°C using indentation. *Error bars* show one standard deviation between three separate test runs. As with the torsion measurements, M1452 is the most compliant sample, followed by M818 and then M596

**Table 1** Summary of testing data for both indentation and torsion measurements at 25 and 55°C, including average $J(t)$, standard deviation of $J(t)$, and approximate change in $J(t)$ over the 100 s hold

| | Epoxy M596 | | Epoxy M818 | | Epoxy M1452 | |
|---|---|---|---|---|---|---|
| Temperature | 25°C | 55°C | 25°C | 55°C | 25°C | 55°C |
| Rheometer $J(t)$ Ave ($GPa^{-1}$) | 1.04 ± 0.02 | 1.81 ± 0.08 | 0.99 ± 0.01 | 3.10 ± 0.50 | 0.94 ± 0.01 | 33.7 ± 19.7 |
| Rheometer $J(t)$ ($GPa^{-1}$) | 0.05 | 0.35 | 0.05 | 1 | 0.05 | 30 |
| Indentation $J(t)$ Ave ($GPa^{-1}$) | 0.80 ± 0.07 | 0.86 ± 0.03 | 0.93 ± 0.04 | 1.06 ± 0.06 | 0.86 ± 0.07 | 1.25 ± 0.08 |
| Indentation $J(t)$ ($GPa^{-1}$) | 0.15 | 0.25 | 0.20 | 0.40 | 0.15 | 1 |

a rough surface should be measured as being more compliant than a smooth one. A further possibility is that the epoxy surfaces are indeed different relative to the bulk material. For example, the crosslink density could be reduced at the surface (more network defects) or the silating agent used might alter surface properties. For the samples tested in torsion, machine mechanical or thermal drift and surface effects such as roughness or decreased crosslink density would not contribute significantly to the precision of the data due to the much larger sampled volume.

At 55°C, $J(t)$ measured via indentation, as shown in Fig. 7, is a clear function of $M_c$. As expected, the most compliant sample is M1452, followed by M818 and then M596. This trend agrees with that measured via torsion experiments at the same temperature. Also, as observed in the torsion experiments, the average standard deviation increases for each sample with temperature, as does the change in $J(t)$ relative to values measured at 25°C. The percent change in $J(t)$ over the 100 s hold period is similar between torsion and indentation measurements for all three samples at 55°C. This result suggests that the time-dependent or viscous component of the material response is similar when measured by either approach. However, there are some striking differences between the torsion and indentation data. Most notably, although the values for $J(t)$ are comparable between the two methods at 25°C, indentation $J(t)$ values at 55°C are at least a factor of two lower (M596) and as much as a factor of 30 lower (M1452) compared to the torsion measurements at 55°C.

A synopsis of measurement results is shown in Table 1. This comparison may suggest that at 55°C, the molecular mobility for microscale, near-surface volumes of the epoxy networks is not nearly as pronounced as it is for macroscale volumes. However, measurement of creep in polymers via indentation at elevated temperature has not been previously considered. Therefore, no similar data exists in the literature to which results can be compared, and the amount of error associated with using equation (9) on the resulting data is unknown. This relation does not include contributions from frictional or adhesive forces, which could alter the measurement of the surface compliance. In addition, although structural isotropy over the deformed epoxy volume is assumed, elevated temperature may alter the degree of isotropy within constrained areas of the epoxy networks. Finally, if decreased crosslink density affected the results at 25°C at the free surface of the epoxy, a similar increase in creep compliance or in compliance rate of the near-surface volumes would be expected relative to the bulk measurements, which was not observed.

In summary, comparison of macroscale and microscale measurements of $J(t)$ indicates that microscale or near-surface indentation measurements of $J(t)$ qualitatively capture the effects of structural and physical properties on creep compliance. An appropriate temperature range must be considered. However, microscale measurements analyzed using currently available contact models will quantitatively underestimate $J(t)$ as the material behavior deviates from primarily elastic behavior.

## Conclusions

Both torsion and depth-sensing indentation techniques were used to measure creep compliance, $J(t)$, for three aliphatic epoxies, each with different molecular weight between crosslinks, $M_c$, using analytical relations of experimentally measured variables. At room temperature, all three epoxies were sufficiently below their glass transition temperature and exhibited limited creep behavior. Differences in $J(t)$ values between epoxies were minimal and did not correspond to differences in $M_c$. The magnitudes of the $J(t)$ values measured via these two methods were quantitatively similar, indicating that cubic-micron scale volumes behave in a similar fashion to the bulk epoxy. However, surface roughness issues can cause discrepancies when using large spherical probes, which are required to achieve linear viscoelastic contact conditions. As temperature was increased, $J(t)$ measured via either approach indicated significant differences as a

function of $M_c$ and corresponding $T_g$. At 55°C, similar trends but differing magnitudes of $J(t)$ were identified. Most notably, creep compliance of the bulk polymer M1452 measured via torsion increased significantly at a temperature roughly 12°C below its $T_g$, whereas the increase in creep compliance of the M1452 surface measured via indentation was significantly less. These issues could indicate that the available viscoelastic contact analyses require further refinement to account for pronounced nonlinearity or viscosity, as is anticipated in polymers characterized near $T_g$.

**Acknowledgments** The authors would like to thank Mr. Kevin Calzia and Dr. Alan Lesser of the University of Massachusetts at Amherst for their help in providing samples and for their invaluable advice. Thanks are given to Dr. Daniel O'Brien of the U.S. Army Research Laboratory for his counsel on the torsion measurements and valuable comments on the manuscript. T.F.J. was supported by an Oak Ridge Institute for Science and Education (ORISE) post-doctoral fellowship to perform this work. C.A.T. gratefully acknowledges support from the National Science Foundation Graduate Fellowship program.

## References

1. Oliver WC, Pharr GM (1992) An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. J Mater Res 7:1564.
2. Oliver WC, Pharr GM (2004) Measurement of hardness and elastic modulus by instrumented indentation: advances in understanding and refinements to methodology. J Mater Res 19:3–20.
3. Briscoe BJ, Fiori L, Pelillo E (1998) Nano-indentation of polymeric surfaces. J Phys, D, Appl Phys 31:2395–2405.
4. Berthoud P, G'Sell C, Hiver JM (1999) Elastic-plastic indentation creep of glassy poly(methyl methacrylate) and polystyrene: characterization using uniaxial compression and indentation tests. J Phys, D, Appl Phys 32:2923–2932.
5. VanLandingham MR, Villarrubia JS, Guthrie WF, Meyers GF (2001) Nanoindentation of polymers: an overview. Macromol Symp 167:15–43.
6. Sakai M (2002) Time-dependent viscoelastic relation between load and penetration for an axisymmetric indenter. Philos Mag, A, Phys Condens Matter, Struct Defects Mech Prop 82:1841–1849.
7. Fischer-Cripps AC (2004) A simple phenomenological approach to nanoindentation creep. Mater Sci Eng, A Struct Mater: Prop Microstruct Process 385:74–82.
8. Oyen M (2005) Spherical indentation creep following ramp loading. Fundamentals of nanoindentation and nanotribology III. Materials Research Society, Pittsburgh, Philadephia.
9. VanLandingham MR, Chang NK, Drzal PL, White CC, Chang SH (2005) Viscoelastic characterization of polymers using instrumented indentation. I. Quasi-static testing. J Polym Sci, B, Polym Lett 43:1794–1811.
10. Hand RJ, Ellis B, Whittle BR, Wang FH (2003) Epoxy based coatings on glass: strengthening mechanisms. J Non-Cryst Solids 315:276–287.
11. Lau K-t, Lu M, Lam Chun-ki, Cheung H-y, Sheng F-L, Li H-L (2005) Thermal and mechanical properties of single-walled carbon nanotube bundle-reinforced epoxy nanocomposites: the role of solvent for nanotube dispersion. Compos Sci Technol 65:719–725.
12. Chisholm N, Mahfuz H, Rangari VK, Ashfaq A, Jeelani S (2005) Fabrication and mechanical characterization of carbon/SiC-epoxy nanocomposites. Compos Struct 67:115–124.
13. Prolongo SG, del Rosario G, Urena A (2006) Comparative study on the adhesive properties of different epoxy resins. Int J Adhes Adhes 26:125–132.
14. Crawford E, Lesser AJ (1998) The effect of network architecture on the thermal and mechanical behavior of epoxy resins. J Polym Sci, B, Polym Phys 36:1371–1382.
15. Timoshenko SP, Goodier JN (1970) Theory of elasticity. Third ed. McGraw-Hill, New York.
16. Hertz H (1881) On the contact of elastic solids. J Reine Angew Math 92:156–171.
17. Lee EH, Radok JRM (1960) The contact problem for viscoelastic bodies. J Appl Mech 27:438–444.
18. Ting TCT (1966) The contact stresses between a rigid indenter and a viscoelastic half-space. J Appl Mech 33:845–854.
19. Hunter SC (1960) The Hertz problem for a rigid spherical indenter and a viscoelastic half-space. J Mech Phys Solids 8:219–234.
20. Graham GAC (1965) The contact problem in the linear theory of viscoelasticity. Int J Eng Sci 3:27–46.
21. Yang WH (1966) Contact problem for viscoelastic bodies. J Appl Mech 33:395.
22. Lu H, Wang B, Ma J, Huang G, Viswanathan H (2003) Measurement of creep compliance of solid polymers by nanoindentation. Mech Time-Depend Mater 7:189–207.
23. Beake BD, Smith JF (2002) High-temperature nanoindentation testing of fused silica and other materials. Philos Mag, A, Phys Condens Matter, Struct Defects Mech Prop 82:2179–2186.
24. Tweedie CA, VanVliet KJ (2006) Contact creep compliance of polymers via nanoindentation. J Mater Res 21 (in press).
25. Plazek DJ (1966) Effect of crosslink density on the creep behavior of natural rubber vulcanizates. J Polym Sci, A-2 Polym Phys 4:745–763.
26. Farlie ED (1970) Creep and stress relaxation of natural rubber vulcanizates. Part I. Effect of crosslink density on the rate of creep in different vulcanizing systems. J Appl Polym Sci 14:1127–1141.
27. Nicholson LM, Whitley KS, Gates TS (2001) Crosslink density and molecular weight effects on the viscoelastic response of a glassy high-performance polyimide. Polym Prepr 42:398–402.