A dynamic microindentation device with electrical contact detection

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We developed a microindentation instrument that allows direct measurement of the point of contact for reasonably conductive samples. This is achieved in the absence of a contact load using a simple electrical circuit. Force is measured using an optical interrupter to measure the deflection of a cantilever beam. Displacement is achieved using a piezoelectric motor and is measured using an independent optical interrupter. Force and displacement measurements are accomplished in real time, allowing the specification of arbitrary waveforms. The instrument was rigorously validated by comparing mechanical property measurements from the indenter with results from traditional dynamic mechanical analysis. Details of the construction and feedback control schemes are given explicitly. © 2009 American Institute of Physics. [DOI: [10.1063/1.3043428](https://doi.org/10.1063/1.3043428)]

I. INTRODUCTION

Indentation is a rapid straightforward technique for measuring mechanical properties of a broad range of materials. Extensive theoretical foundations exist for the measurement of linear elastic mechanical properties via indentation.^{1–5} More recently, the focus has shifted to nonlinear elastic and linear viscoelastic materials.^{6–33} However, the instrumentation required is generally expensive and specialized for a certain class of materials. Accordingly, we have developed an indentation device that was robust, simple to construct, inexpensive, and convenient for testing a variety of material types without sacrificing the accuracy of results.

Measurement of forces in the micro-Newton range has traditionally required expensive, complex, and fragile load sensors. Newer techniques, such as atomic force microscopy, tend to rely on coupling optical and magnetic devices with more traditional mechanical transduction methods.^{12,34–36} Many recent indentation devices have taken advantage of the wealth of classical theory available for the mechanics of cantilever beams for measuring force.^{37–40} These instruments measure force using specialized and computationally intensive image analysis after the fact. Unfortunately, such methods do not allow real-time force measurements, rendering true dynamic testing (e.g., creep or relaxation tests) impossible. Semiempirical methods have thus been adopted for attempting to quantify the viscoelastic parameter of materials using indentation.¹²

One recent instrument used a simple photodetector array to detect the deflection of a plate and infer the force in real time to allow the measurement of dynamic forces for animal

movement.⁴⁰ We have adapted this idea to indentation, measuring force by using a cantilever beam to vary the voltage output of an optical interrupter. An optical interrupter is an infrared light source situated opposite a photodetector. When the light from the source was blocked from the detector, the voltage output of the system varied proportionally. This allowed force measurements using a cantilever beam without the need for image analysis. Since the force was known in real time, feedback control was used to enable the specification of arbitrary force or displacement waveforms, thereby allowing the quantitative determination of viscoelastic material parameters using creep, relaxation, and dynamic (oscillatory) testing methods.¹⁰

Another problem with the indentation of soft nonlinear materials is the determination of the point of contact. It has been shown that if the exact point of contact is not accurately identified, the material properties established using this technique will not necessarily converge to the correct values.⁹ Thus, we have developed a simple method for quantitatively defining the point of contact by simply applying a voltage to the sample and the indentation probe. When the two meet, the circuit is grounded and the contact is quantitatively established. This method is suitable for application for any reasonably conductive sample, including many biological materials.^{41,42}

II. APPARATUS

A microindentation device was constructed as follows. Displacement was achieved using a piezoelectric motor (PDA130D, EDO Electro-Ceramic Products, Salt Lake City, UT) to drive an alumina beam (Fig. 1). The position of the alumina beam was monitored using an optical interrupter (H22A1, Fairchild Semiconductor, South Portland, ME) where the source was interrupted by a flag attached to the

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FIG. 1. (a) Photograph and (b) schematic of the microindentation apparatus. The piezoelectric motor drives the alumina beam. The linear movement is ensured by the linear bearing. The displacement of the beam is measured by the optical interrupter (left), while the other end of the beam is equipped with the force sensor (detailed in Fig. 2).

alumina beam driven by the motor. Force was measured using an identical optical interrupter, where the source was interrupted by a simple plastic cantilever beam (Fig. 2). The beam deflection altered the output of the optical interrupter by obstructing the detector from its source. A polished aluminum flat punch of 250 μm diameter was mounted on the cantilever beam. The motor was controlled, and displacement and force sensor voltage signals were acquired with 16 bits of precision using LABVIEW V8.0 software in conjunction with a PCI card (PCI-6221, National Instruments Corp., Austin, TX). Since both displacement and force were measured, either could be selected as the control variable for the feed-

FIG. 2. Schematic of the force sensor (a) at the point of contact and (b) at an arbitrary indentation depth. At the point of contact, (c) the blocker's position dictates the offset of the force sensor, whereas the material properties (elastic modulus and moment of inertia) and dimensions of the deflection beam dictate the sensor's sensitivity. As the probe delves deeper into the sample, it causes deflection of the beam. This deflection causes the beam to enter the (d) path of the optical sensor, thereby changing the output voltage.

back loop (Fig. 3). All loop functions, including recording of force and displacement data, were performed at a frequency of 1 kHz. Using LABVIEW's timing profiler capabilities, we determined that the limiting factor in decreasing the feedback loop's time increment was recording the data. Therefore, if a computer with faster writing capabilities was employed, the loop time could be decreased further.

The force sensor operates as a simple cantilever beam. At the point of contact, the blocker's position dictates the offset of the force sensor, whereas the material properties (elastic modulus and moment of inertia) and dimensions of the deflection beam dictate the sensor's sensitivity. As the probe delves deeper into the sample, it causes deflection of the beam. This deflection causes the beam to enter the path of the optical sensor, thereby changing the output voltage. An optical interrupter is simply a light source situated opposite a photodetector. When some light from the source is blocked from the detector, the voltage output of the system varies proportionally.

FIG. 3. Feedback loop block diagram for the control of (a) displacement or (b) force. Note that both displacement and force were measured dynamically and that the motor control voltage is the control parameter in both control schemes. Symbols in the block diagrams are error ϵ , command voltage V , probe velocity v , probe position x , measured force F , displacement u , and beam deflection at the probe tip u_{beam} . The method for determining each of these quantities is described in the text.

The cantilever beam used to measure force for low-modulus materials naturally has a low bending rigidity, resulting in a low natural frequency for the beam (in this case, 50 Hz). This in turn caused oscillations at higher frequencies in dynamic testing. Another potential drawback of this design is the potential for the change in the angle of the probe relative to the sample surface. In experiments where sufficient friction exists at the probe-sample interface, the probe will remain normal to the surface. However, if slippage occurs, then deviation from the normal may result. Since indentation models necessarily assume a perpendicular contact, care must be taken to ensure that only small deviations from this occur to minimize the error in measuring mechanical properties. This may be achieved by changing the length of the probe, the material of the cantilever beam, or the shape of the blocker attached to the cantilever beam. The initial contact angle of the probe was ensured using a level fixed to the indentation device. For the materials studied herein, the change in the angle from the normal (as calculated using the beam deflections given in Ref. 43) was always less than 3° and was therefore not considered to have a significant impact on the results. This assumption is reinforced by agreement with results from DMA.

A. Feedback control

Once contact was established, the indentation device was controlled by a feedback loop. This allowed the indenter to follow any arbitrary displacement $[u_s(t)]$ or force $[F_s(t)]$ waveform. Figure 3 gives the block diagram for displacement-controlled indentation (A) and for force-controlled indentation (B).

The error ϵ is defined as the difference between the set point of the control variable (u_s or F_s for displacement or force control, respectively) and the true value from the last loop iteration, or

$$\epsilon_i = u_{s,i} - u_{i-1} \quad \text{or} \quad \epsilon_i = F_{s,i} - F_{i-1}. \quad (1)$$

A kinetic proportional-integral-derivative algorithm⁴⁴ is augmented with backward finite-difference differentiation and multiple-application trapezoidal integration⁴⁵ to increase the accuracy without significantly increasing the computation requirements. The voltage V output by the controller to the piezoelectric motor is then

$$V_i = \left[K_P \epsilon_i + K_D \frac{\epsilon_i - \epsilon_{i-1}}{\Delta t} + K_I \frac{t_1 - t_0}{2n} \left(\epsilon_n + 2 \sum_{j=i-n}^i \epsilon_j + \epsilon_i \right) \right] + V_d, \quad (2)$$

where i is the current iteration; n is the number of terms considered in the integral; K_P , K_D , and K_I are gains specified for the proportional, derivative, and integral portions, respectively; and V_d is a dead zone of ± 1 V in the motor drive circuit within which the motor did not move. The sign of V_d is the sign of the error for iteration i .

Assuming that the motor's output velocity v is linearly dependent on the control voltage,

FIG. 4. Schematic of cantilever beam (a) load F and length dimensions a , b , and $\ell = a + b$ and (b) cross-sectional width $\bar{w}$ and thickness $\bar{t}$ for the determination of beam deflection for feedback control using Eq. (6). For clarity, figures are not to scale.

$$v_i = \frac{\partial x}{\partial t} \approx c_m (V_i - V_d), \quad (3)$$

where c_m is a proportionality constant relating the velocity to the voltage and x is the position of the beam. This assumed relationship gave satisfactory results during indentation experiments, where the voltage range was very small. This relationship fails when the time for one iteration of the loop is long, causing large errors and, therefore, large voltages. The position of the beam is then measured using the position optical interrupter and could be estimated as

$$x_i = x_{i-1} + \int_{t_0}^{t_1} v_i dt = x_{i-1} + \Delta x, \quad (4)$$

although the true position is computed from the measured voltage using the linear calibration fit rather than calculated. The indentation depth u_i is then

$$u_i = u_{i-1} + \Delta x - u_{\text{beam}}, \quad (5)$$

where u_{beam} is the deflection of the cantilever beam at the point where the probe is fixed. This deflection was

$$u_{\text{beam}} = \frac{Fa^3}{E\bar{w}\bar{t}^3}, \quad (6)$$

where F is the force exerted on the probe by the material; a is the distance from the fixed edge of the cantilever to the probe (Fig. 4); and $\bar{E}$, $\bar{w}$, and $\bar{t}$ are the elastic modulus, width, and thickness of the cantilever beam, respectively.⁴³

The exact form of the force F depends on the constitutive behavior of the material being indented. For example, the force generated by indentation of a linear elastic material is

$$F = \frac{2ERu}{1 - \nu^2}, \quad (7)$$

where E and ν are the sample material's elastic modulus and Poisson ratio, respectively, and R is the probe radius.² Again, force is directly measured using a calibration of the cantilever beam rather than calculated using this relationship. In principle, one could measure a broad range of elastic moduli by simply changing the material or shape of the deflection beam.

B. Calibration

The position sensor was calibrated by monitoring the output voltage from the optical interrupter while measuring

FIG. 5. Calibration for the motor position sensor. The sensor was comprised of a blocker attached to the piezoelectric motor's alumina drive beam, which varied the detected intensity of a stationary optical interrupter with the drive beam's position. The position data are relative to the position at which the blocker first changed the voltage output of the optical interrupter. The vertical dashed lines (---) indicate the location of hard stops installed on the alumina beam after calibration to ensure operation only within the linear region of the position sensor's dynamic range, giving up to 450 μm of useful displacement. The solid line (—) indicates the best-fit line used for calibration. The equation of this line was used to relate the output voltage to the position of the motor. The least count of the micrometer was 10 μm , so the uncertainty in measurement of the position was assumed to be on the order of 5 μm .

the position using a digital micrometer (Fig. 5). All displacements used in experiments were performed within the linear range of this calibration.

The force transducer sensitivity was calibrated by placing washers around the indentation probe (Fig. 6). Each washer was first weighed using an analytical balance. The beam stiffness was sufficiently low that the mass of the beam caused a shift in the offset upon inversion. The offset is accounted for in the control software prior to testing by setting the offset equal to the force sensor's output at the time when contact was achieved. Since the linear elastic modulus was dependent only on sensitivity F/u (as shown below), not the offset, this shift in offset was unimportant as long as the output remained in the linear range. However, for the measurement of nonlinear parameters, determination of this offset before and after indentation was important. A linear calibration was implemented by fitting only the linear range of the response curve. All experiments occurred within this linear range. This was ensured by adjusting the beam such that the initial voltage offset was far from the threshold or saturation prior to each experiment.

The correlation between position and optical sensor output for determining displacement was highly linear across all allowable displacements (Fig. 5). The alumina beam was disallowed from moving outside this range by attaching mechanical stops to the beam. This approach yielded up to 450 μm of accurate displacement. This yielded a nominal resolution of 0.01 μm , which could be improved by simply altering the shape of the blocker attached to the alumina beam. However, signal noise limited the precision to around 0.05 μm . The least count of the micrometer used in the calibration was 10 μm , so the uncertainty in measurement of the position was assumed to be on the order of 5 μm .

The force sensor output voltage was a sigmoidal function of force with a large linear dynamic range (Fig. 6). This yielded a resolution of 0.03 μN . Again, this could, in prin-

FIG. 6. Calibration for the force sensor. The sensor was comprised of a blocker attached to the cantilever beam, which varied the detected intensity of a stationary optical interrupter with beam deflection. The data are relative to zero force with the cantilever beam initially bent such that it did not influence the voltage output of the optical interrupter until a load was applied. The vertical dashed lines (---) indicate the limits of the sensor's linear response. The cantilever beam was always adjusted prior to experimentation such that its initial voltage output fell within this range. Experiments yielding data falling outside of this range were discarded. This yielded a linear range of about 2300 μN of force. The solid line (—) indicates the best-fit line used for calibration. The equation of this line was used to relate the output voltage to the force applied to the cantilever beam.

ciple, be improved by altering the shape of the blocker attached to the cantilever beam. However, signal noise limited the precision to around 0.1 μN .

C. Contact determination

The determination of the point of contact is essential for accuracy when determining the mechanical properties of any nonlinear material using indentation.⁹ Real-time contact detection was implemented by applying a 3 V electrical potential across the probe and the sample. When this circuit was grounded (i.e., when the probe contacted the sample), the voltage decreased instantaneously by an amount that depended on the electrical properties of the sample material. Thus, contact was objectively determined when the voltage fell below a predefined threshold of 1 V. To minimize the electrical current through the sample, a 1 M Ω resistor was placed between the voltage source and the indentation probe. It should be noted that for a perfectly linear elastic behavior and a flat punch, the detection of contact is unimportant. However, its accurate determination is essential for quantifying nonlinear material parameters.

In practice, two methods were used to achieve contact. First, the indentation device was mounted on a three-dimensional micromanipulator. When it was appropriately positioned adjacent to the sample, the manipulator was used to slowly lower the indenter until the probe contacted the sample (as determined by observing the voltage output from the contact circuit). The second method was implemented in the control software to automate the process. Contact was achieved by automatically incrementing u_s by 2 $\mu\text{m/s}$ until the voltage of the contact circuit fell below the threshold value. Virtually identical results were obtained using these two methods. Once contact was determined, the desired displacement or force waveform was applied to the sample using the position and force measurements at the point of con-

FIG. 7. Example of the fitting methods for data analysis. (a) First, the data are fitted with polynomials of sufficient order to capture their character (i.e., second order for loading data and third order for unloading data). (b) The loading data are also fitted with a line whose slope is related to the material's stiffness. The coefficients of the polynomials are then used to determine the asymptotic slopes at (c) the onset of loading and (d) the onset of unloading. The slopes determined using these three methods were then used to calculate the elastic modulus for comparison with results from DMA.

tact as the initial condition. Since the point of contact was established prior to the initiation of any indentation protocol, pure creep, relaxation, and dynamic tests to determine the viscoelastic material parameters were possible.¹⁰

III. MATERIAL TESTING

A. Materials

Samples used for validation of the indenter were prepared from commercially available gellan (Kelcogel CG-LA; CP Kelco, Chicago, IL). Gels were prepared in a manner similar to the protocol suggested by the manufacturer. A stock solution of gellan was made up at 1% concentration (10 mg gellan/mL distilled water), heated in a water bath at 90 °C until dissolved, and then cooled to 50 °C. This stock solution was then added at 50 °C to a mix of the appropriate amount of distilled water containing 1 ml of ten times concentrated Dulbecco's phosphate-buffered saline (DPBS) (without calcium or magnesium ions), pH 7.4, to give final desired concentrations of 0.15, 0.20, 0.30, and 0.40 wt % gellan in physiologically concentrated DPBS. The solution was rapidly pipetted into 1 cm $\times$ 1 cm circular cylindrical

polytetrafluoroethylene molds and cooled to room temperature to induce gelation. Five gels were cast and tested at each concentration.

B. Testing protocols

The static elastic modulus of the material was determined using quasistatic testing with both indentation and dynamic mechanical analysis (DMA). The results of these two methods were compared to determine the efficacy of the proposed indentation method. Time-dependent indentation data are also presented as a proof of concept, demonstrating that the feedback loop may be used effectively to examine the viscoelastic behavior of various samples. Determination of the viscoelastic or nonlinear material properties of the sample is beyond the scope of the present manuscript. Various methods for determining these properties are available in the literature (e.g., see Refs. 16 and 38). These methods may be applied directly to the time-force-displacement data generated with this instrument.

FIG. 8. Comparison of the elastic modulus found using DMA (x-axis) with the elastic modulus found using the described indentation device (y-axis). (a) Moduli found using a simple linear fit of loading force-displacement data. The agreement found using this method indicates the accuracy of the force measurements. (b) Moduli found using a second-order polynomial fit of all force-displacement data and then applying the asymptotic approach. The agreement found using the asymptotic method indicates the efficacy of the contact detection method proposed. Comparison of the asymptotic unloading method applied to indentation data compared with (c) linear and (d) asymptotic loading methods applied to DMA data. Note that the asymptotic moduli are always larger than those found using the linear fitting method due to the strain-softening behavior of the gellan gels.

1. Quasistatic indentation

Measurement of linear elastic modulus E is straightforward, as one need only measure the force F and displacement u . The elastic modulus E of a linear elastic material may be found from the indentation of a semi-infinite half space as

$$E = \frac{1 - \nu^2}{2R} \left(\frac{F}{u} \right), \quad (8)$$

where ν is the material Poisson ratio and R is the probe radius.² Since $(1 - \nu^2)/2R$ is a constant, the measurement of the material stiffness (i.e., the slope of the force-displacement data, F/u) is sufficient to determine the elastic modulus. In practice, after contact was achieved, a triangular displacement waveform was specified. The amplitude was 100 μm with a frequency of 0.05 Hz, corresponding to a period of 20 s and a probe velocity of 10 $\mu\text{m/s}$. This frequency was selected after analyzing several higher frequencies to ensure that a quasistatic assumption was appropriate (that is, the measured force was independent of the frequency). Because the indentation depth is only 0.1% of the gel depth, there is no reasonable expectation of substrate

effects on the measured mechanical properties, which are generally observed when indentation depth approaches 10% of the sample depth.⁹ Thus, the semi-infinite assumptions of Sneddon's² indentation analysis should hold.

2. Time-dependent indentation

The measurement of viscoelastic material parameters may be achieved by measuring force and displacement. However, unlike purely elastic materials, the time-dependent behavior of the sample response must be considered. Accordingly, the control software allowed pure creep, pure relaxation, and sinusoidal loadings in addition to triangular waveforms for ramping force or displacement. Creep testing was performed by specifying a constant force (i.e., F_s was a Heaviside step function) and by measuring the change in displacement with time. Relaxation testing was performed by specifying a constant displacement (i.e., u_s was a Heaviside step function) and by measuring the change in force with time. Dynamic testing requires the application of an oscillating force or displacement and measuring the amplitude and phase of the other variable. Either the force or displacement

FIG. 9. Sample force and displacement as functions of time for a creep test. The creep test applied a constant load and monitored the displacement response with time. Note that a finite amount of time was required to achieve the prescribed constant force, but this time was minimized by utilizing a feedback loop with a properly tuned controller.

waveform may be specified, although displacement was generally controlled. The waveform for displacement was

$$u(t) = \frac{u_0}{2} [1 - \cos(\omega t)], \quad (9)$$

where u_0 is the amplitude of the applied displacement, ω is the radian frequency, and t is the time. The expected form of the resulting force was then

$$F(t) = \frac{F_0}{2} [1 - \cos(\omega t + \delta)], \quad (10)$$

where F_0 is the amplitude of the force waveform and δ is the phase angle by which the force waveform leads the displacement waveform.

It should be noted that these dynamic protocols only give stress or strain that is nominally constant for flat-tipped probes where the contact area is constant with indentation depth.¹² In addition, the circular cylindrical probe was selected because it gives a linear relationship between load and displacement for a perfectly linear elastic material.² This greatly simplifies the control procedure and subsequent analyses.

3. Dynamic mechanical analysis

To validate the findings from indentation experiments, cylinders of the same batches of material used in indentation testing were characterized with DMA using the Perkin-Elmer DMA 7e dynamic mechanical analyzer (Perkin-Elmer Corp., Norwalk, CT). The gellan cylinders were tested in quasistatic compression between parallel plates at a strain rate of $2.5 \times 10^{-4} \text{ s}^{-1}$ to a maximum load of 25 mN. The elastic modulus of a linearly elastic cylinder for small strains is given by

$$E = \frac{\ell_0}{\pi R_0^2} \left(\frac{F}{u} \right), \quad (11)$$

which is again dependent only on the material stiffness F/u .

C. Data analysis methods

Three methods were used to determine the elastic modulus for the samples. First, a simple linear fit was applied to the indentation loading curve. The slope of this line was the material stiffness F/u , which was then substituted into Eq. (8) to calculate the modulus. This essentially represented the average modulus because it averaged the stiffness over the entire range of displacements. The same method was applied to the DMA data to obtain the stiffness and modulus using Eq. (11).

The second method was an asymptotic approach used to estimate the elastic modulus in the small displacement limit for loading data and at the onset of unloading. Conceptually, this method is simply taking the stiffness F/u to be the slope of the tangent line in the limit as $u \rightarrow 0$ for loading data. A power series polynomial of the form

$$F(u) = \sum_{i=1}^p c_i u^i \quad (12)$$

was used, where c_i are the fitting coefficients and p is the polynomial degree of the fit. The slope to this curve as $u \rightarrow 0$ is found by evaluating its derivative,

$$\frac{\partial F}{\partial u} = \lim_{u \rightarrow 0} \sum_{i=1}^p i c_i u^{i-1}. \quad (13)$$

For this material, the loading data were well fitted for $p=2$.

The third method, which is commonly employed in instrumented indentation of elastoplastic materials,^{12,19} was to take the initial slope of the indentation unloading curve as the material stiffness. This method is generally thought to be more reproducible than measurements using the loading curve since the unloading behavior is assumed to be dominated by elastic recovery only.^{12,19,46,47} This method was not applicable to DMA data. Therefore, the moduli from indentation found using this method were compared to moduli from DMA derived using both linear and asymptotic methods described above. The asymptotic slope was found by fitting the unloading data with Eq. (12) and evaluating Eq. (13) at $u = u_{\max}$. For this material, the unloading data were fitted well with $p=3$.

Analyses for both indentation and DMA compression were performed using the linear and asymptotic methods. The gellan material described herein was selected for its non-linear behavior at low strains, likely a result of its cross-linking via hydrogen bonding. It therefore allowed a good check for the contact detection method. If the contact method was inaccurate, the asymptotic moduli found from indentation would differ significantly from those measured using DMA. Results from both methods for determining the modulus are presented and compared with simple linear fitting results. An example of these methods is given in Fig. 7 for clarity. Quantification of the nonlinear constitutive behavior of these materials is beyond the scope of this manuscript.

FIG. 10. Sample displacement and force data for a relaxation test. The relaxation test applied a constant displacement and monitored the force response with time. Note that a finite amount of time was required to achieve the prescribed displacement and some overshoot occurs, but this time and overshoot were minimized by utilizing a feedback loop with a properly tuned controller. To minimize errors in analysis due to these control issues, critical damping may be achieved by proper manipulation of controller gain parameters. Significant noise was present in the force data for short times after the application of the displacement. This is due to the cantilever beam's inertia, which causes it to oscillate rapidly after high-velocity maneuvers. This implies that such force measurements are not appropriate for measuring very rapid relaxation processes. Also of note is the nonmonotonic force behavior. For most polymeric systems, the force monotonically decreases throughout the duration of a relaxation experiment. However, due to the dynamic nature of the structure in these gels, the force increased after long times. This is likely a result of the gellan restructuring in response to applied stress.

IV. RESULTS

A. Quasistatic measurements

A comparison of the gellan elastic moduli determined using the new indentation device and DMA was good using both the linear fitting method [Fig. 8(a)] and the asymptotic fitting method [Fig. 8(b)]. The elastic moduli determined using the asymptotic technique were higher in all cases than the moduli determined using the linear technique.

B. Time-dependent measurements

Creep, relaxation, and dynamic experiments were successfully performed. Constant force (Fig. 9) or displacement (Fig. 10) was rapidly achieved using the feedback system described above. Note that a finite amount of time was required to achieve the prescribed force or displacement and some overshoot occurred, but the rise time and overshoot were minimized by utilizing a feedback loop with a properly tuned controller. To minimize errors in analysis due to these control issues, critical damping may be achieved by proper manipulation of controller gain parameters, although the parameters will require adjustment for each material tested since the material's dynamic response is important in determining the feedback error. Short rise times give confidence when modeling the data with pure creep, relaxation, or dynamic relations. Accurate dynamic waveforms were also generated using this system (Fig. 11).

FIG. 11. Sample displacement and force data for an oscillating dynamic test. Note that the data have been downsampled (i.e., for each data point shown, 50 others were collected) to allow visualization of the theoretical waveforms.

V. DISCUSSION

The good agreement between elastic moduli determined using DMA and indentation (Fig. 8) indicate that the combination of real-time contact detection, force measurement, and displacement measurement provide the capability for dynamic testing for determining viscoelastic parameters. The agreement of the line fitting method [Fig. 8(a)] is indicative of the accuracy of the force measurement technique used. The agreement of the asymptotic method [Fig. 8(b)] demonstrates the efficacy of the proposed contact method since the asymptotic behavior would differ significantly if contact were determined incorrectly due to the nonlinear behavior of the higher concentration gels. The asymptotic moduli were significantly higher, with the discrepancy increasing with sample stiffness. This was a result of the strain-softening character of the more concentrated gels. Since the apparent stiffness decreased with increasing displacement, the linear fit (which is essentially the average stiffness across all displacements) was always lower than the stiffness at the zero displacement limit. The disparity was slightly larger for the asymptotic method since a slight change in initial loading yields dramatically disparate results due to the quadratic character of the force-displacement data.

From the results presented in Figs. 7(c) and 7(d), it is clear that the unloading curve method is inappropriate for this material system. This is likely due to the gellan's "lossy" behavior, which is not generally a concern for indentation testing of stiff polymer, metal, or ceramic systems.¹² This method was both inconsistent and inaccurate, indicating the breakdown of the assumption that elastic recovery dominates during unloading. This method is generally used to quantify elastic behavior in elastoplastic systems rather than in viscous systems. We therefore speculate that the observed hysteresis was due to viscous dissipative losses rather than to plastic deformation. It is therefore suggested that loading curves and very slow loading rates must be used to quantify the elastic behavior of such systems. Hence it is suggested that either of the other two fitting methods should be selected

depending on how the data will be used. If nonlinear constitutive relations are required for a particular application, then an inverse finite element method and observation of the material's true displacement field is necessary to achieve good results.^{16,38}

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