Nonlinear Behavior in Dynamic Nanoindentation

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1 Introduction

Studying the mechanical response of materials at the nanoscale has received much attention in recent years [1–5]. These studies have been motivated by the development of new nanostructured materials, a continued miniaturization within engineering, electronic component applications, thin film technology, and a growing interest in the characterization of biomaterials [1, 6]. Three different techniques and instruments have been developed by researchers for these studies: 1) the atomic force microscope (AFM) ; 2) the surface force apparatus (SFA); and 3) a depth sensing nanoindentation technique [7]. The capabilities of these instruments cover a wide range of contact area - from several [μm^2] for SFA down to a few [nm^2] in AFM.

The SFA is primarily suited for direct measurements of intermolecular and surface forces, but not mechanical material behavior. A second instrument of interest is the AFM, which has become a popular instrument for imaging surface topography; this instrument has also been used to investigate the elastic and plastic properties of materials at the nanoscale [8]. However, several difficulties exist due to an unknown contact area (i.e. complications from a small tip size and unknown tip shape) and the reliance upon an inferred cantilever spring constant. Further complications are encountered in dynamic AFM modes, such as in tapping modes [9], which can render mechanical property estimates to be only qualitative.

The accurate determination of both contact area and displacement has been improved by recent developments in depth sensing nanoindentation [5]. The underlying goal is to couple force modulation with depth sensing to obtain quantitative measures of mechanical properties (e.g. modulus, hardness, viscoelasticity, and creep).

The basic idea of an indentation process is simple; the method requires touching a material of interest, whose material properties are unknown, with another material whose properties are known. The establishment of Brinell, Knoop, Vickers, and Rockwell tests all follow from a refinement of indenting one material with another [10]. Nanoindentation, which is sometimes called depth-sensing indentation, is simply an indentation test where the length scale of penetration is in nanometers (see Fig. 1). Dynamic nanoindentation refers to the process of imposing a known excitation force on the indenter and measuring the nanoscale response of the indenter-specimen system to identify the surface mechanical properties. While one of the original applications for this instrument was in the characterization of thin films, the number of applications for nanoindentation has exploded over the past half-decade in the areas of life science, materials science, and physical science [5].

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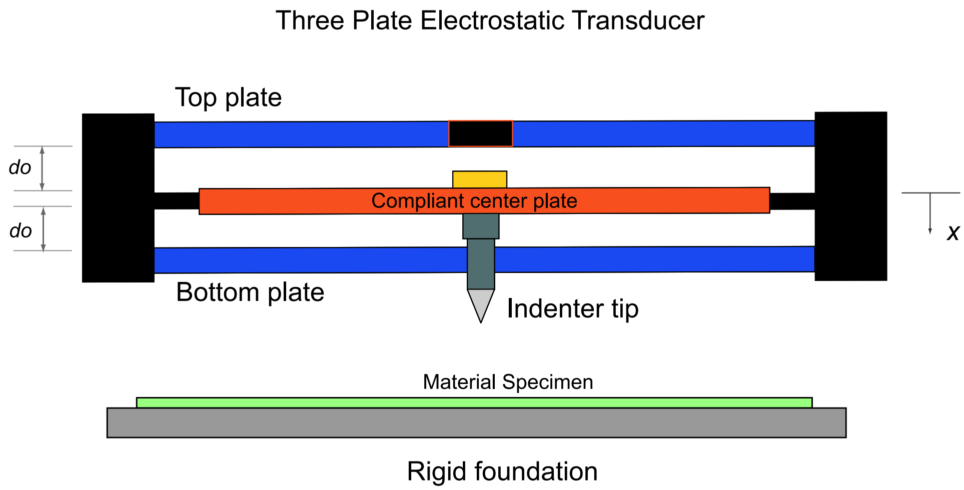

Figure 1: *Schematic of the dynamic nanoindentation process illustrating: 1) an example application of thin film material testing; and 2) the specific nanoindentation test instrument of interest that utilizes an electrostatic transducer to perform nanomechanical material test.*

To study the frequency dependent properties of materials, such as combined studies of the material storage modulus (stiffness) and the material loss modulus (viscoelasticity) in polymers, it is necessary to perform dynamic nanoindentation tests and examine the coupled indenter-specimen response at the nanometer scale. The common approach for identifying the specimen material properties requires monitoring the amplitude and phase relationships of the combined system. However, the current understanding of dynamic nanoindentation considers only linear system models in the presences of nonlinear contact forces [1, 10–12], nonlinearities in transducer actuation, van der Waals forces, adhesion, and nonlinear material behavior. Therefore, a current void in the literature is a comprehensive understanding of how these nonlinearities influence estimates of the mechanical material behavior in dynamic nanoindentation. The goal of the present study is to compare the results of applying linear identification methods to a nonlinear identification approach for characterization of mechanical material behavior in dynamic nanoindentation.

2 Modeling

The parameters for the present study are taken from a Hysitron Inc. nanoindenter, which consists of a load-displacement transducer with an electrostatic actuation force and displacement sensing electronics. The load-displacement transducer, shown in Fig. 1, utilizes two rigid outer plates and a compliant center plate to displace the indenter tip via electrostatic forces that are generated by the introduction of a voltage diffential; the center plate displacement is sensed by monitoring changes in capacitance. The first mode of this system can be modeled as follows:

$$m\ddot{x} + c\dot{x} + kx + k_3x^3 + h(x, \dot{x}) = \frac{\varepsilon_o A}{2}\left[\frac{V_B^2}{(d_o - x)^2} - \frac{V_T^2}{(x + d_o)^2}\right]. \quad (1)$$

where $m = 259.95 \times 10^{-6}$ [Kg] is the indenter modal mass, $c = 0.047$ [Ns/m] is the indenter damping, $k = 160.56$ [N/m] and $k_3 = 0.1k$ [N/m^3] are the linear and nonlinear restoring forces, $\varepsilon_o = 8.854 \times 10^{-12}$ [C^2/Nm^2] is a permeability constant, $A = 5.15 \times 10^{-9}$ [m^2] is the transducer plate area, $d_o = 0.87 \times 10^{-6}$ [m] is the transducer plate spacing, and $V_{T,B}$ are the top and bottom plate transducer voltages. During dynamic nanoindentation the top transducer plate is commonly turned off and the bottom voltage consist of a static and dynamic portion $V_B = V_{DC} + V_{AC}\cos\Omega t$, where Ω is the excitation frequency in [rad/s]. For the purposes of this paper, the interaction or contact forces between the assumed flat material surface and spherical indenter are considered to be a function of the material effective elastic modulus, $E = 1 \times 10^8$ [N/m^2], with the following form $h(x, \dot{x}) = \frac{4}{3}E\sqrt{R}x^{3/2}$ for a spherical indenter of radius $R = 2 \times 10^{-6}$ [m] (see Asif *et al.* [1]). The linear response of the combined indenter and material specimen was also given by Asif *et al.* [1] as

$$X = \frac{F_o}{\sqrt{\left(k + k_s - m\Omega^2\right)^2 + \left(c\Omega\right)^2}}, \tag{2}$$

where X is the response amplitude of the combined system due to an harmonic input force of magnitude $F_o = \varepsilon_o A V_{DC} V_{AC}/d_o^2$. Since the material behavior is assumed to be perfectly elastic, the identified specimen stiffness, k_s, is the only unknown parameter. For a viscoelastic material, Eq. (2) is commonly combined with the phase shift (ϕ), which is given by $\phi = \tan^{-1}\left(\frac{c\Omega}{k+k_s-m\Omega^2}\right)$, where c would now represent the total damping in the system, to obtain the material damping and stiffness from the measured input-output response. These values can be converted into the commonly reported material storage and loss moduli with well-known formulas when the viscoelastic material response is represented as a complex stiffness [7, 10, 12].

3 Summary and Conclusions

Numerical studies of Eq. (1), performed for several excitation voltage levels, show that the combination of transducer mechanical and electrostatic nonlinearity as well as the interaction force nonlinearity provide a significant source of error in dynamic nanoindentation. To provide a graphical example, the response amplitude and phase results from a $V_{DC} = 15$ [V] and $V_{AC} = 13$ [V] excitation are shown in Fig. 2. This figure shows that the response at the first harmonic, a result of nonlinearity in the system, is sometimes larger than response amplitude at the excitation frequency; therefore, significant errors can occur when identifying the material behavior under the assumption of a linear system model.

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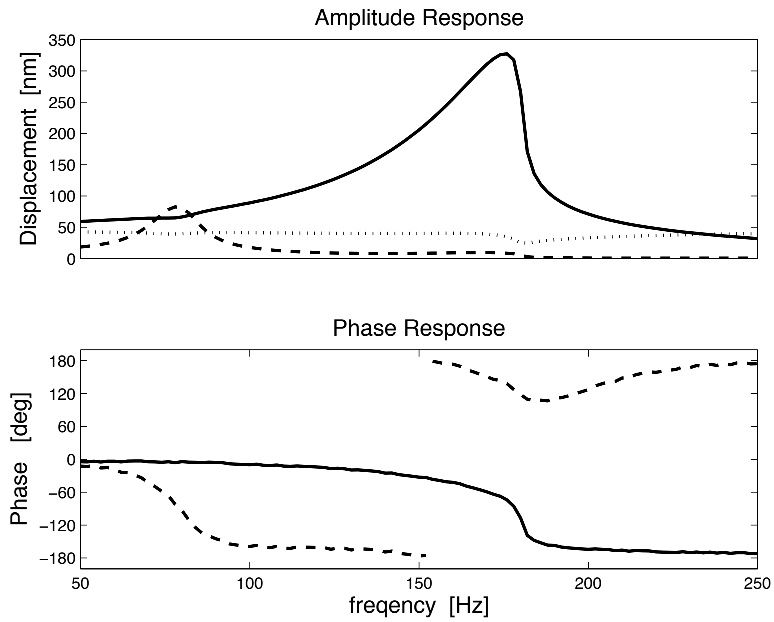

Figure 2: *Predicted response amplitude and phase of combined system. Curves are for the transducer static displacement (dotted line) and the response amplitude/phase at: 1) the excitation frequency (solid line); and 2) the first harmonic (dashed line).*

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