Photoacoustic characterization of the mechanical properties of thin films

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Narrow band photoacoustics (laser ultrasounds) are used to characterize the properties of free-standing nanometer-sized thin films. Photoacoustic generation is achieved by use of a microchip laser which deposits pulsed laser energy in the form of a spatially periodic source on the structure. The resulting narrow band ultrasonic modes are monitored using a Michelson interferometer. By varying the geometry of the spatially periodic source, a wide range of acoustic wave numbers is probed. Results are presented for two-layer thin film aluminum/silicon-nitride (Al/Si3N4) membranes. For such thin films, only the two lowest order guided modes are generated and these in turn can be related to sheet and flexural modes in plates. The mechanical properties and residual stress in the thin films are evaluated from measured acoustic dispersion curves for these two lowest order modes. © 2002 American Institute of Physics. [DOI: 10.1063/1.1434303]

Microelectromechanical system (MEMS) devices such as pressure sensors, radio-frequency switches, and resonators often include free-standing thin film structures. The performance of these devices depends on their mechanical properties including the elastic moduli and residual stress.1 Currently bulge tests, nanoindentation techniques, and resonance methods are used to measure these properties.2–5 These tests require direct loading of the MEMS device, and detailed information about the dimensions and support conditions of the films is needed to accurately interpret the measurements.

Photoacoustic metrology uses pulsed laser irradiation to nondestructively induce high-frequency ultrasound in a test object via rapid thermal expansion.6 The resulting ultrasonic wave packets are also measured in a noncontact nondestructive manner using interferometric or diffractive optical probes. High-frequency photoacoustic interrogation techniques can provide information about the mechanical, thermal, and electronic properties of materials, and can also be used to detect surface and subsurface flaws in structures. Photoacoustic measurements are local and do not require knowledge of structural support conditions.

Photoacoustic techniques for thin film and coating characterization have been reported in the literature under widely varying terminology. Pump–probe techniques that use femtosecond lasers to generate ultrasounds in the GHz range have been used to monitor film/substrate reflections of very thin films.7 Guided or surface acoustic waves that propagate in the plane of the film have also been used for thin film characterization. These include broad band surface acoustic wave (SAW) techniques,8 the impulsive stimulated thermal scattering9 or laser-induced transient grating technique,10,11 and the phase-velocity scanning technique.12

In this letter, we describe a photoacoustic transient grating technique that generates narrow band guided acoustic waves and a balanced Michelson interferometer that monitors the guided modes in two-layer free-standing 500-nanometer-size aluminum/silicon nitride thin film structures. In ultrathin structures, only the two lowest order modes—the “sheet” mode and the flexural mode—exist well into the several hundred MHz frequency range. Typically, using optical diffractive techniques only the flexural mode has been detected in such structures.11 We demonstrate that our interferometric system has sufficient sensitivity to detect both modes, thereby enabling the determination of up to three mechanical properties (including residual stress) of the thin film structure.

The optical layout of the photoacoustic microscope is shown in Fig. 1. Narrow band acoustic waves are generated by the coherent interference of two crossed pulsed laser beams that are obtained from a microchip laser (480 ps pulse width, 13.2 μJ energy, and 1064 nm wavelength). The beams are focused onto the surface of the thin film to be tested, resulting in an intensity grating due to coherent interference. The rapid spatially nonuniform thermal heating that ensues in the thin film leads to the generation of acoustic waves with fixed wave number $k = 4\pi/\lambda_c \sin(\theta/2)$, where $\lambda_c$ is the gen-

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FIG. 1. Optical layout of the photoacoustic microscope. BS: beam splitter; PBS: polarizing beam splitter; pzt: piezo-electric transducer; $\lambda/2$: half-wave plate; $\lambda/4$: quarter-wave plate.
eration laser beam wavelength and \( \theta \) is the angle between the two crossed laser beams. The resulting ultrasonic displacements on the film are monitored using a balanced Michelson interferometer. It should be noted that interferometry provides higher detection sensitivity than optical diffractive probes at these frequencies.

Free-standing layered thin film structures of various thicknesses and in-plane extents were microfabricated. A photomask was designed to create rectangular openings in a (100) oriented silicon wafer. Silicon nitride was grown on both sides of the wafer using low pressure chemical vapor deposition (LPCVD). Protective coatings were deposited on both sides of the wafer and photoresist was spin coated on one side and exposed to obtain the desired pattern. The silicon nitride was then selectively removed from this side of the wafer and the silicon was etched to leave a free-standing film of silicon nitride with thicknesses ranging from 200 to 400 nm and lateral dimensions ranging from 100 to 2000 \( \mu m \). Aluminum films ranging from 300 to 500 nm in thickness were subsequently e-beam evaporated onto the silicon nitride. The structures were then photoacoustically tested without further processing.

The elastodynamic analysis of guided acoustic waves in layered solid media is well understood. Several acoustic modes can be sustained in layered materials. It is in general not possible to obtain simple analytical expressions for the phase velocities of the various modes in terms of the material properties and waveguide dimensions, so only numerical solutions are available. However, if we restrict the experiments to small values of \( kh \) (where \( k \) is the acoustic wave number and \( h \) is the overall thickness of the film), then only the two lowest order modes (called \( S_o \) and \( A_o \)) are possible. For the thin films under consideration, and for the experimental wave numbers used, this is indeed the case. The advantage of restricting the experiments to small \( kh \) values is that, asymptotically as \( kh \) approaches zero, these two modes essentially become sheet waves and flexural waves, respectively. In this case, analytical expressions can be obtained for the velocities in terms of the material properties, layer thicknesses, and residual stress in the film. The \( S_o \) mode phase velocity is given by

\[
\nu_{so} = \frac{\omega}{k} = \sqrt{\frac{C^*}{\rho^*}}
\]

where the composite stiffness and density are given by

\[
C^* = \sum_{k=1}^{N} \frac{E_k}{1-v_k^2} V_k; \quad \rho^* = \sum_{k=1}^{N} \rho_k V_k.
\]

Here \( E_k \) is the Young’s modulus, \( v_k \) is the Poisson ratio, and \( \rho_k \) is the density of the material in layer \( k \). The volume fraction is \( V_k = h_k/h \), where \( h \) is the total thickness of the plate and \( h_k \) is the layer thickness. For \( A_o \) mode, the phase velocity is given by

\[
\nu_{ao} = \frac{\omega}{k} = \sqrt{\frac{D^*}{\rho^*} (kh)^2 + \frac{\sigma_x}{\rho^*}}
\]

where \( \sigma_x \) is the composite (thickness-averaged) residual stress, and \( D^* \) is the normalized composite flexural rigidity that is explicitly (although not compactly) known in terms of the material properties and thicknesses of the layers. Note that (to the same order of approximation), the residual stresses in the thin film affect only the \( A_o \) mode and do not affect the \( S_o \) mode velocities. Furthermore, the \( S_o \) mode is nondispersive, whereas the \( A_o \) mode is dispersive.

Photoacoustic data were obtained for a two-layer 420 nm Al/236 nm Si\(_3\)N\(_4\) thin film sample. Dispersion curves of guided acoustic modes over a range of \( kh \) values were obtained by adjusting the angle between the two crossed generation beams. Figure 2(a) shows a representative time trace of the \( S_o \) mode obtained for a specific wave number (\( kh=0.104 \), corresponding to a grating spacing of \( \Lambda = 39.5 \mu m \)). Figure 3(a) shows \( A_o \) mode time domain data for the same specimen and wave number. As expected, the frequency content of the \( A_o \) mode [Fig. 3(b)] is significantly lower than that of the \( S_o \) mode [Fig. 2(b)]. The \( A_o \) mode signal has a peak frequency of 8.6 MHz, corresponding to phase velocity of 339 m/s. The \( S_o \) mode wave form, on the other hand, has a peak frequency of 176 MHz, corresponding to velocity of 6952 m/s.

Figure 4 shows the dispersion curves measured for \( S_o \) and \( A_o \) modes, respectively. As expected from small \( kh \) values the former is nondispersive and the latter is dispersive. Up to three thin film parameters can be extracted by minimizing the least-squares error between the experimentally measured dispersion data and the asymptotic expressions (shown by solid lines in Fig. 4). By assuming bulk values for the Poisson ratios of the two materials, and by independently measuring the layer thicknesses using ellipsometry and profilometry, it is possible to obtain the Young’s moduli of the two materials (\( E_{Al}, E_{SiN} \)) and the thickness-average residual stress, \( \sigma_x \). For the two-layer 420 nm Al/236 nm Si\(_3\)N\(_4\) thin
film sample, the measured values are $E_{\text{Al}} = 78$ GPa, $E_{\text{SiN}} = 200$ GPa, and $\sigma_s = 188$ MPa. On a different specimen, a two-layer 510nm Al/250nm Si$_3$N$_4$ film, the photoacoustic measurements yielded $E_{\text{Al}} = 56$ GPa, $E_{\text{SiN}} = 250$ GPa, and $\sigma_s = 214$ MPa. The modulus for aluminum measured is within the range of 50–90 GPa obtained by Lim et al.[16] on evaporated films. The modulus of the silicon nitride measured is also within the range of 250 GPa that is cited for LPCVD grown films, and values of 210 and 290 GPa for plasma enhanced chemical vapor deposition films and low pressure chemical vapor deposition films, respectively.[17]

The values in the literature for the modulus of aluminum and silicon nitride on the nanometer scale are known to vary greatly from batch to batch. The variations are typically attributed to changes in fabrication conditions. The photoacoustic measurements of the moduli of aluminum and silicon nitride are in the same range as published values. Photoacoustic techniques therefore provide a simple, local, noncontact, nondestructive means, by which to characterize materials of very thin layered films.

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