Probing limits of acoustic nanometrology using coherent extreme ultraviolet light

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ABSTRACT

Photoacoustic nanometrology using coherent extreme ultraviolet (EUV) light detection is a unique and powerful tool for probing ultrathin films with a wide range of mechanical properties and thicknesses well under 100 nm. In this technique, short wavelength acoustic waves are generated through laser excitation of a nano-patterned metallic grating, and then probed by diffracting coherent EUV beams from the dynamic surface deformation. Both longitudinal and surface acoustic waves within thin films and metallic nanostructures can be observed using EUV light as a phase-sensitive probe. The use of nanostructured metal transducers enables the generation of particularly short wavelength surface acoustic waves, which truly confine the measurement within the ultrathin film layer of interest, to thicknesses < 50 nm for the first time. Simultaneous measurement of longitudinal and transverse surface wave velocities yields both the Young's modulus and Poisson's ratio of the film. In the future, this approach will make possible precise mechanical characterization of nanostructured systems at sub-10 nm length scales.

Keywords: metrology, photoacoustics, surface acoustic wave, longitudinal acoustic wave, thin film, low-k, extreme ultraviolet, high harmonic generation, Young's modulus, Poisson's ratio.

1. INTRODUCTION

Nanofabrication technologies now make it possible to deposit single-atomic-layer films and pattern nanometer scale structures. However, our ability to precisely characterize the mechanical properties of structured systems at sub-100nm dimensions remains limited. In addition to enabling the design, manufacture and process control of nanoscale devices, precise characterization of ultrathin films and nanostructures is necessary for understanding the physics which applies to such small-scale systems: how elastic properties change with scaling from bulk material to monolayers[1,2], for example, or how heat transport is modified by non-diffusive transport[3,4].

Currently, elastic characterization of thin films in the semiconductor industry relies mostly on nano-indentation measurements. While providing a measurement localized to the region directly compressed by the atomic force microscope tip, this is a destructive measurement which is greatly influenced by the substrate beneath the thin film of

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interest – particularly as the film thickness shrinks. Various models can be used to try to account for the substrate contribution, and thus extract the 'real' film characteristics[5,6]. However, these approaches still rely on fairly thick films (hundreds of nanometers) or knowledge of some of the material properties – such as the Poisson's ratio for the film and substrate.

Non-contact acoustic methods offer an important non-destructive approach for thin film metrology. In particular, picosecond ultrasonics has long been used for longitudinal acoustic wave (LAW) measurements of thin films. Here the strong absorption of a laser pump pulse in the film (or in a thin transducer layer) launches LAWs into the film, which can be observed by transient changes in the reflected intensity of a subsequent probe pulse. Measurements of the LAW velocity can then be used to calculate the Young's modulus of isotropic thin films, assuming a value of Poisson's ratio[7,8].

In more recent measurements, nanostructured transducers were introduced to gain access to transverse surface acoustic waves (SAWs), to avoid the need of assuming a value for Poisson's ratio[9-11]. However, the use of visible light probes inherently limits the sensitivity of such techniques, because thinner films require shorter-wavelength surface displacements, smaller than those easily accessible with the resolution of visible wavelengths. As a result, thus far visible picosecond ultrasonics has been used to characterize films as thin as 600 nm.

Brillouin light scattering is another non-contact option for thin film metrology, which does allow the simultaneous measurement of transverse and longitudinal acoustic velocities – and thus extracting both Young's modulus and Poisson's ratio. However, interpretation is more complex in this case, and the weak intensity of scattered light makes characterization strongly dependent on the experimental accuracy attained[12].

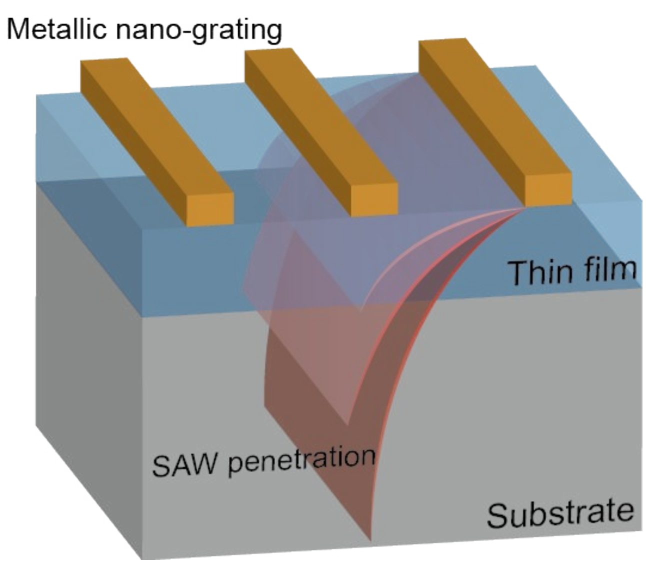

Figure 1. Surface acoustic waves penetrate into the material over a depth equal to a fraction of their wavelength. Thus, SAWs are an excellent layer-selective probe of elastic properties. The SAW wavelength can be set by the period of a metallic nano-grating, while the properties of the substrate material can be probed using a longer wavelength SAW (dark red) excited by a large-period grating. A short-wavelength SAW (light red) from a small-period grating will remain confined within the thin film layer, and probe its material properties without unwanted contribution from the substrate.

In this paper we make use of nanostructured, metallic grating transducers with periods as short as 90 nm to simultaneously excite SAWs with wavelengths set by the grating and LAWs in a series of 50-100-nm thin films with varying elastic properties. These represent the thinnest films directly probed to date using acoustic probes. The shortest SAW wavelength implies a small penetration depth fully confined within the thin film of interest, as shown schematically in Figure 1. This allows us to selectively probe the properties of the thin film material without unwanted contribution from the substrate underneath. This approach for mechanical nano-characterization will scale to sub-10 nm thin films, with the shortest SAW wavelength set by the smallest nanostructured transducers set by current nanofabrication limits[13]. This capability is important because simulations suggest a thickness dependence in the elastic properties of ultrathin film materials[1,2] – thus, experiments that allow direct testing of theoretical models are needed. Our technique also enables testing of new ultrathin film materials being developed in nano-electronics, as well as the development of a potential in-line characterization tool for process control.

2. EXPERIMENTS AND SAMPLES

In these experiments, we probe a series of 50-100-nm thin low-k dielectric SiC:H films deposited on silicon substrates, with a range of elastic properties (particularly Young's moduli nominally extending from 13 to 200 GPa) set by the levels of hydrogenation in the film material[14]. Time-resolved detection of the acoustic dynamics in these samples is obtained via pump and probe pulses derived from a Ti:sapphire amplifier system (3-3.5 kHz, 1.5-2 mJ, 25 fs). The 800 nm pump (heating) pulses and 30 nm EUV probe pulses are delayed with respect to each other by a mechanical delay stage[15,16.]

To launch nanoscale acoustic waves, we deposit periodic metallic nanostructures on the surface of the film to serve as the transducer of acoustic waves in the system. A schematic of the samples and technique is shown in Figure 2. These nanostructures absorb the ultrashort 800 nm pump pulse, resulting in an impulsive thermal expansion which launches the acoustic waves: LAWs which travel down into the thin film as in traditional picosecond ultrasonics and can reflect back from buried interfaces, and SAWs due to the transverse periodicity of excitation, which travel along the surface with a penetration depth of only a fraction of their wavelength. To ensure uniform heating of the nanostructures, the pump beam is kept relatively large, with a diameter of ≈ 400-500 μm and a fluence of ≈10 mJ/cm^2.

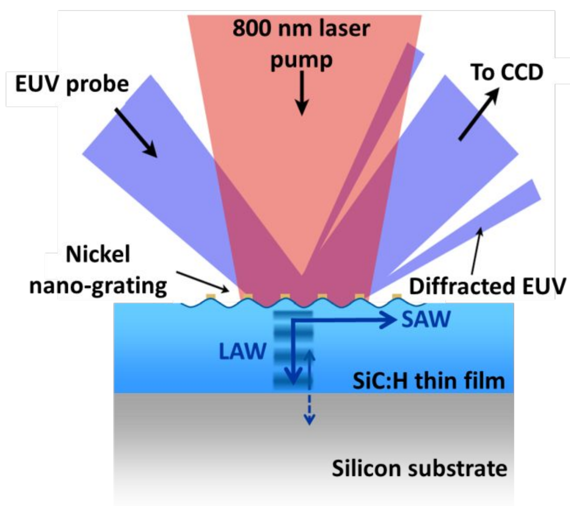

Figure 2. A 25-fs 800 nm laser pump pulse heats a nickel nano-grating whose subsequent impulsive expansion simultaneously launches SAWs at the surface and LAWs which travel down through the SiC:H thin film and reflect from the interface with the substrate. The time-resolved diffraction of a 30-nm extreme ultraviolet probe pulse from the periodic nano-grating structure reveals the acoustic dynamics, which can be used to determine the Young's modulus and Poisson's ratio of the dielectric film.

The SAW wavelength is set by the nanostructure period, which allows excitation of acoustic wavelengths much shorter than possible using current transient grating techniques[13]. True confinement of SAWs within the thin film layer requires a SAW wavelength Λ on the same order as the film thickness t, or approximately $\frac{\Lambda}{\pi} \lesssim t$. Using electron beam lithography and liftoff techniques, we manufacture nickel lines with thickness 10 nm and periods P ranging from 90 nm to 1500 nm, with constant filling fraction of 1/3. The strong absorption of 800 nm pump light and high coefficient of thermal expansion of nickel makes it an efficient photoacoustic transducer. The range of periods we use allows us to examine the transition from SAWs fully confined within the thin films to those propagating mostly in the silicon substrate. This allows us to unequivocally demonstrate a layer-selective measurement of both the substrate and thin film acoustic velocities.

The detection of the shortest-wavelength SAWs requires a comparably short-wavelength probe. We obtain this by focusing the laser probe beam into an argon-filled hollow-core waveguide for nonlinear upconversion to extreme ultraviolet wavelengths (centered around 30 nm) using the process of high harmonic generation[17]. This process yields the coherent short wavelength beams needed for high spatial resolution, while preserving (and even shortening) the femtosecond pulse duration and the full spatial coherence of the beam. The EUV probe beam is focused onto the sample by a glancing-incidence toroidal mirror to a spot size of approximately 100 μm; here it diffracts from the periodic nanostructures, and the time-dependent diffraction pattern is captured on an X-ray-sensitive CCD. This phase-sensitive measurement yields very high sensitivity to even picometer-scale surface displacements.

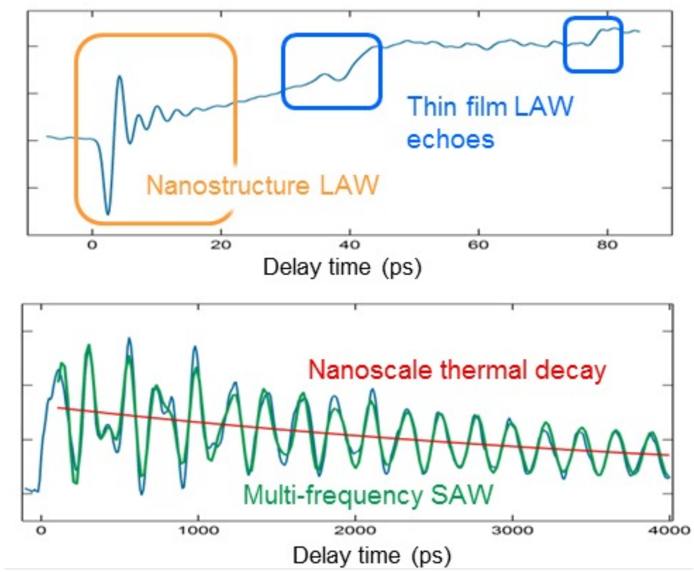

Figure 3. The acoustic dynamics in the thin-film/substrate system separates into two timescales. On the picosecond timescale (top), two sets of longitudinal acoustic waves are visible: in the first 20 ps, a fast oscillation represents the LAW in the metal nanostructures (orange box); later, two features arise which represent echoes on the surface from the LAW which travels through the thin film and reflects from the interface with the substrate (blue boxes). On the nanosecond timescale (bottom), there is a gradual, exponential-like thermal decay as heat dissipates from the nanostructures to the substrate (red). Finally, there is also a multi-frequency oscillation due to the SAWs propagating in the system with frequencies corresponding to harmonics of the nano-grating period (green).

3. DATA AND ANALYSIS

The acoustic dynamics naturally separates into two timescales, as shown in Figure 3. On the picosecond timescale, two LAWs are visible. First a high-frequency oscillation $f_{LAW,Ni}$ within the nickel nanostructures can be used in conjunction with the thickness of the deposited metal to calculate a longitudinal velocity, $v_{LAW,Ni} = 2t_{Ni}\, f_{LAW,Ni}$, which can be compared with the literature value, $v_{LAW,Ni}$ = 6040 m/s[18]. This is one way to verify the fidelity of the measurement. Second, echoes from the LAW traveling down through the thin film and reflecting back from the substrate interface are visible at time delays corresponding to its round-trip time, $\tau = 2t_{film}/v_{LAW,film}$. From knowledge of the film thickness t_{film} (known with good accuracy from ellipsometry measurements), this allows the calculation of the longitudinal acoustic velocity in the thin film material.

On the nanosecond timescale, two sets of dynamics are again present. There is a slow, near-exponential decay representing heat dissipation from the nanostructures into the substrate. Due to the small scale of the nano-wires, this thermal decay can be used to study non-diffusive thermal flow, as highlighted by Siemens et al.[3] Superimposed on this decay is the multi-frequency SAW oscillation excited by the periodic stress induced at the film surface by the initial nanostructure expansion. Fourier transform of this signal isolates the SAW frequencies f_{SAW}, and the SAW velocity can then be calculated using the wavelength Λ which is set by the nano-grating period: $v_{SAW} = \Lambda\, f_{SAW}$.

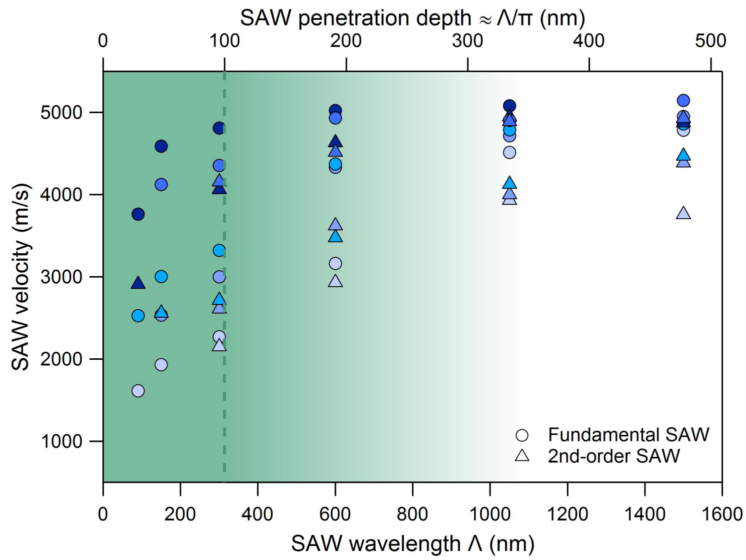

Figure 4. For long-wavelength, long penetration depth measurements, the observed SAW velocity corresponds well with that of the silicon substrate material (with $v_{SAW} \approx 5000$ m/s). In contrast, the short-wavelength, short penetration depth measurements separate the films by their elastic properties from the softest film with Young's modulus E = 13 GPa (light blue) to the stiffest with E = 200 GPa (dark blue). Full confinement of the SAW in the thin film occurs when the penetration depth is less than the film thickness (dotted line); however, the effect of the film material (green shading) on the measured velocity is visible even for somewhat longer penetration depths. In certain cases – e.g. for $\Lambda = 600$ nm – the fundamental SAW (circles) represents the silicon properties while the second-order SAW (triangles) is more confined within the film and displays a velocity more consistent with the thin film material.

Figure 4 shows that for large-period gratings, the measured SAW velocity is consistent with literature values for silicon alone (≈ 5000 m/s) because the penetration of the wave below the surface is such that it travels mostly in the substrate. In contrast, the short-period gratings display the slower velocities associated with all the different film materials because these waves are truly confined within the film layer, isolating the film properties from any substrate influence. Even different SAW orders launched by the same nano-grating can show this effect; in particular, the P = 600 nm grating excites a fundamental surface wave which mostly probes the substrate properties and a second-order surface wave with wavelength = 300 nm, which is much more surface-confined and representative of the thin film material properties.

The measured transverse and longitudinal acoustic velocities are used along with the thin film material density (from X-ray reflectivity measurements) to calculate a first approximation of the Young's modulus E and Poisson's ratio v for an isotropic film[19]. Figure 5 shows that our measurements of E for the series of thin films correspond well to the nominal values obtained from nano-indentation measurements. We are also able to measure the Poisson's ratio for these films without previous assumptions, a quantity that was not accessible before. It is also worth noting that these measurements are still possible (although with larger error) even when the elastic properties of the film are close to those of the substrate, when weaker reflection of the longitudinal wave is expected from the film/substrate interface due to a smaller acoustic mismatch.

This technique can also be used for mechanical characterization of pre-existing patterned chips not specifically designed for this particular approach. Any periodic nanostructured grating with an area at least comparable to the pump beam spot size can be used as acoustic transducer. A reasonably high quality nano-grating, as well as a grating thickness different from the $\lambda/2$ destructive interference condition for a given diffracting probe wavelength, are required to obtain an optimal signal-to-noise ratio to accurately extract the acoustic frequencies.

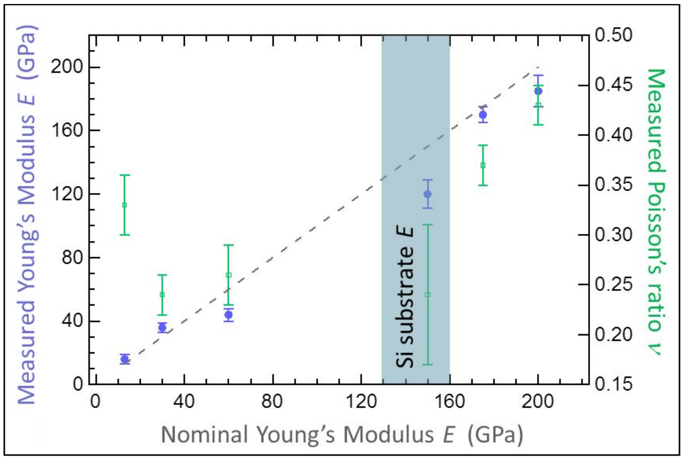

Figure 5. EUV photoacoustic measurements of the Young's moduli of various films (blue points) are consistent with the nominal values obtained from nano-indentation measurements (perfect agreement marked by the dotted line). In addition, from the same measurement we are able to extract the Poisson's ratios for the films (green points), a quantity which was inaccessible before. Even in the region where the substrate and thin film properties are quite similar (shaded in blue) and reflection of the LAW from the interface is diminished due to a lower acoustic mismatch, EUV acoustic metrology is still effective, although with larger error.

Of course, the presence of the deposited nanostructures does introduce loading on the film, which will modify the SAW velocities somewhat. To understand and account for this effect, we employ finite-element simulations to model the whole system. Adjusting the properties of the modeled thin film until the experimentally observed velocity dispersion is recovered yields a more precise determination of E and ν. However, the precision and high sensitivity of the SAW frequency measurements using coherent EUV light would easily allow the development of an in-line monitoring tool to immediately see any changes in the elastic properties of a material during manufacturing.

4. CONCLUSION

The layer-specificity makes coherent EUV-probed acoustic nanometrology attractive for characterizing the mechanical properties of ultrathin films. We have shown that this technique is effective for a wide range of low-k dielectric films with varying elastic properties, including Young's moduli from 13 to 200 GPa. Moreover, we have not yet reached the limits of this EUV nanometrology system. In particular, EUV-probed acoustic nanometrology is still effective when the thin film and substrate have rather similar characteristics – even though longitudinal acoustic reflection from the interface is diminished. This work also extends thin film nanometrology to films of 50-nm thickness – to our knowledge the thinnest film directly measured with a non-contact technique to date. Given the current capabilities of nanofabrication for periodic metallic gratings as well as advances in high harmonic sources at wavelengths down to 1 nm[20], this technique will enable the characterization of sub-10 nm films.

One great advantage of this approach for thin film measurement is the possibility for a wide variety of separate measurements using the same setup. EUV high harmonics are sensitive to magnetic[21] and thermal dynamics[3] and can also be used to directly image a sample via coherent diffractive imaging[22]. Therefore this technique offers the possibility for the future development of a unique flexible nanometrology tool capable of a wide variety of characterization modalities.

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