Reliable characterization of materials and nanostructured systems <<50nm using coherent EUV beams

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ABSTRACT

Coherent extreme ultraviolet beams from tabletop high harmonic generation offer revolutionary capabilities for observing nanoscale systems on their intrinsic length and time scales. By launching and monitoring acoustic waves in such systems, we fully characterize the mechanical properties of sub-50nm films. We find that the Poisson's ratio of low-*k* dielectric materials does not stay constant as often assumed, but increases when bond coordination is below a critical value. Within the same measurement, by following the heat dissipation dynamics from nano-gratings of width 20-1000nm and different periodicities, we confirm the effects of a newly identified *collectively-diffusive* regime, where closely-spaced nanowires cool faster than widely-spaced ones.

Keywords: Ultrafast X-Rays, nanometrology, nano-mechanical properties, ultrathin films, nondiffusive thermal transport, mean free path spectroscopy, photoacoustic, photothermal

1. INTRODUCTION

Modern nanofabrication techniques can synthesize single atomic layer films and nanostructures with <10nm characteristic dimensions. At these length scales, accurate measurements of material properties such as the elastic tensor[1-4] become very challenging[5]. Additionally, bulk model predictions are no longer accurate – for example, the Fourier law of diffusion fails to describe nanoscale thermal transport[6-8]. To inform device design and validate new theoretical models, especially for complex novel materials, new and powerful nanometrology techniques are needed. In particular, nanometrology techniques that are also noncontact and nondestructive would be indispensable for in-situ measurements during industrial-scale production and rapid iteration of new patterning strategies.

The challenge for most existing noncontact, nondestructive, nanoscale measurements of the elastic tensor of nanostructured systems is the diffraction limit of visible light, which sets a limit of hundreds of nanometers on direct measurements. Picosecond ultrasonics, for example, uses visible wavelengths to probe the optical properties (surface profile) of a material in the presence of acoustic waves[9-14]. Brillouin scattering can be used to characterize ~20nm films in a noncontact manner by detecting scattering between photons and acoustic phonons[15]. However, this technique is challenging to interpret due to low scattered light intensity and the difficulty in identifying the phonon modes involved[16].

In the case of nanoscale thermal transport, the diffraction limit of visible light is once again a barrier to probing transport deep in the nanoregime. For many techniques, visible light serves as both the probe and as a heat source, where the sample is heated either by as a single focused laser spot[17,18] or as a transient grating produced by the interference of two crossed laser beams[19]. In both cases, the characteristic length scale of the nanoscale heated region is diffraction-limited to hundreds of nanometers. It is possible to circumvent this limit using nanostructures to absorb the pump light and serve as nanoscale

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heaters[8,20,21]. However, the signal from visible wavelength probes is challenging to interpret because the sample response (e.g. reflectivity) changes with hot electron excitation, material density, and surface deformation, producing a very complicated signal.

To overcome these limitations, our technique probes nanoscale systems with <<100nm characteristic dimensions using coherent extreme ultraviolet (EUV) light produced via tabletop high harmonic generation (HHG). This coherent EUV light is an ideal probe of nanosystem dynamics because its wavelength and pulse duration matches the inherent length- and time-scales of charge, spin and energy transport. Moreover, EUV light can be extremely sensitive to specific material properties: for example in acoustic or thermal metrology, the measurement depends only on the surface deformation (profile), and is insensitive to contributions due to hot electrons. Here we show that EUV nanometrology can be used to extract the full elastic tensor of isotropic thin films of thickness <50nm thick, while also characterizing non-diffusive thermal transport from heaters with dimensions ≈ 20nm, all in a single measurement. Using this technique, we observed a divergence in the Poisson's ratio of low-k dielectric films below a critical value of bond coordination, and confirmed the effects of periodicity on nanoscale thermal transport in the collectively-diffusive regime, where closely-spaced nanoheaters can cool more efficiently than widely-spaced ones[7].

2. COHERENT EUV NANOMETROLOGY

To characterize the elastic properties of ultrathin films, as well as the heat dissipation away from nanoscale heat sources into various substrate materials, we employ a visible pump and EUV probe to directly observe the nanoscale dynamics (Figure 1a). Our samples consist of periodic arrays of nickel nanowires fabricated on the surface of dielectric or semiconductor substrates, in some cases with the addition of a low-*k* dielectric ultrathin film on top of the substrate. An 800nm infrared femtosecond pump pulse is preferentially absorbed by the metallic grating, causing rapid heating of the arrays. The impulsive thermal expansion of the nanostructures launches longitudinal acoustic waves (LAWs) within the nanostructures and into the material below and transverse surface acoustic waves (SAWs) in the film and substrate, where the SAW wavelength and penetration depth is set by the period of the nanostructured grating.

To directly probe the transport and mechanical properties of nanostructured materials with characteristic dimension (CD) << 100nm, we probe our sample using a novel coherent EUV light source based on high harmonic generation (HHG). HHG is a highly nonlinear quantum process in which laser light (UV, visible, infrared (IR)) is up-converted to coherent EUV/soft x-ray light with wavelengths ranging from 1-50nm and with pulse durations from attoseconds to femtoseconds[22-26]. For the work described here, we use 30nm EUV probe beams that diffract from the dynamically changing sample surface, and then collect the scattered EUV light on a CCD camera. Because of the short wavelength of the EUV light, the change in diffraction efficiency is large even for small deformations, and therefore we are sensitive to picometer-scale surface deformations. This signal is recorded as a function of the delay time between the IR pump and EUV probe pulses, allowing for direct observation of dynamics on multiple time-scales: from the nanosecond-scale thermal decay, to the hundreds of picoseconds-scale SAW oscillations, to the tens of picoseconds-scale LAW reflections from the film-substrate boundary, and the few picosecond-scale longitudinal resonances of the nanostructures (Figure 1b).

To process our data, we use finite element analysis (FEA) to analyze and extract the material characteristics of interest. The full elastic tensor of isotropic ultrathin films can be characterized by extracting the LAW and SAW velocities from the experimental signal. We account for the modification of the elastic properties due to the mass loading from the nanogratings by calculating the eigenmodes of the system and iteratively tuning Poisson's ration and Young's modulus until the FEA simulated SAW frequencies match the measured values. We also model the thermal relaxation in FEA simulations to quantify the deviation of the transport dynamics from the diffusive prediction. By combining the intrinsic thermal boundary resistivity (r_{TBR}) and the non-diffusive effects (r_{corr}) into an effective thermal boundary resistivity ($r_{eff} \equiv$

r_{TBR} + r_{corr}), we can find a best fit r_{eff} for our experimental signals that quantifies the deviation of the thermal transport from the diffusive prediction.

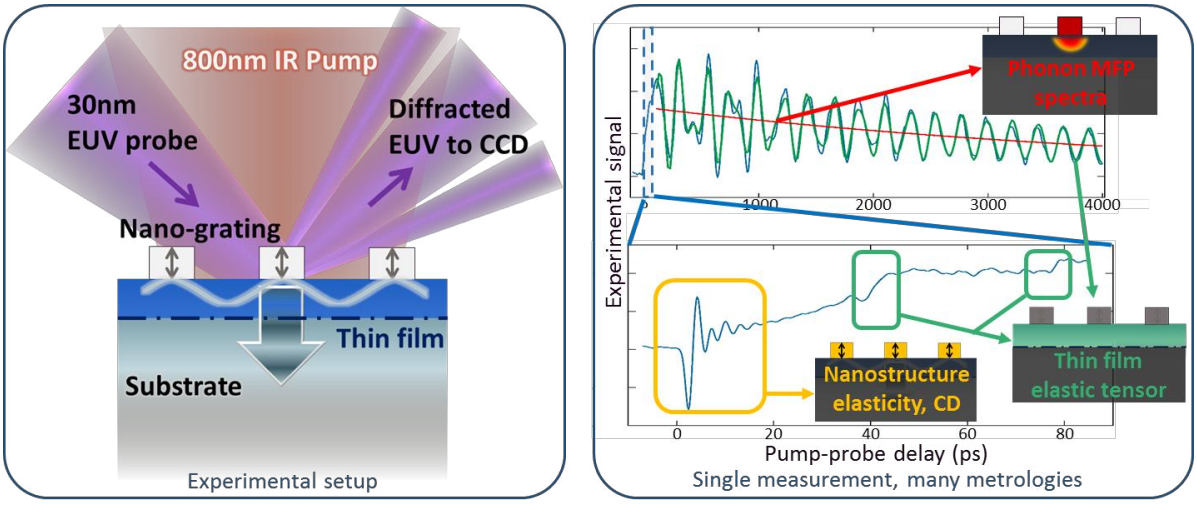

Figure 1. a) EUV photoacoustic nanometrology uses an IR pump laser pulse to excite a metallic nanograting, and EUV light to monitor changes in the surface profile caused by thermal and acoustic dynamics. b) The diffraction efficiency signal of the EUV probe beam monitors the full dynamics of a nanostructured system: the long-timescale, near-exponential thermal decay as the metallic structures dissipate heat into the substrate (red), the surface and longitudinal acoustic waves in the substrate (green), and the longitudinal resonant mode of the metallic nanostructures themselves (yellow). By directly observing the dynamics in nanostructured systems, we extract the full elastic tensor of sub-50nm films and uncover previously unobserved trends in the Poisson's ratio of low-*k* dielectrics. In addition, we probe and confirm the collectively-diffusive regime of nanoscale thermal transport.

3. CHARACTERIZATION OF THE FULL ELASTIC TENSOR OF ULTRA-THIN FILMS

By monitoring the oscillation frequencies of waves of known wavelength, we measure the acoustic wave velocities in a material, and thus probe its elasticity. In the past, we demonstrated the use of SAW excitations via nanowire gratings to realize a complete, nondestructive characterization of isotropic films down to 100nm in thickness without contribution from the substrate underneath[1,27]. More recently, by exciting LAW resonances across bilayer structures we were able to measure the density and elastic properties of sub-10nm layers of material[28].

Here we utilize our technique to study the elastic properties of a series of low-*k* dielectric sub-100nm thin films with varying nominal Young's modulus (5-150GPa). In order to confine SAW excitations to the films, a series of nickel nanowire gratings are fabricated on top with constant height of 10nm, varying linewidth from 15-1000nm and constant 33% duty cycle. The short periodicities allow us to excite acoustic waves of wavelength down to 45nm that are fully confined in films <20nm and very sensitive to elastic properties of ultra-thin films down to 5nm. The long periodicities, on the other hand, allow us to measure the elastic properties of the supporting substrates. The exquisite sensitivity of our coherent EUV probe to surface displacements allows us to extract the frequencies of the excited SAWs. Using FEA continuum mechanics models, we can simultaneously extract the Young's modulus and Poisson ratio of the thin films.

The extracted values of Young's modulus plotted in Figure 2a show good agreement with the expected nominal values, validating the technique on a very wide range of Young's modulus. However, currently only EUV nanometrology allows for the simultaneous extraction of the Poisson's ratio of such thin layers. While Poisson's ratio is generally assumed to be constant, our results have revealed an increase in Poisson's ratio with decreasing Young's modulus below a critical value around 40GPa, as shown in Figure 2b. This trend is potentially related to the bond coordination <*r*> of the film material

discussed by King, et al.[29] as all the wafers within this trend have values of $\langle r \rangle \leq 2.4$. This is a critical value for other material properties of a-SiC:H films, including thermal diffusivity, Young's modulus and hardness[29].

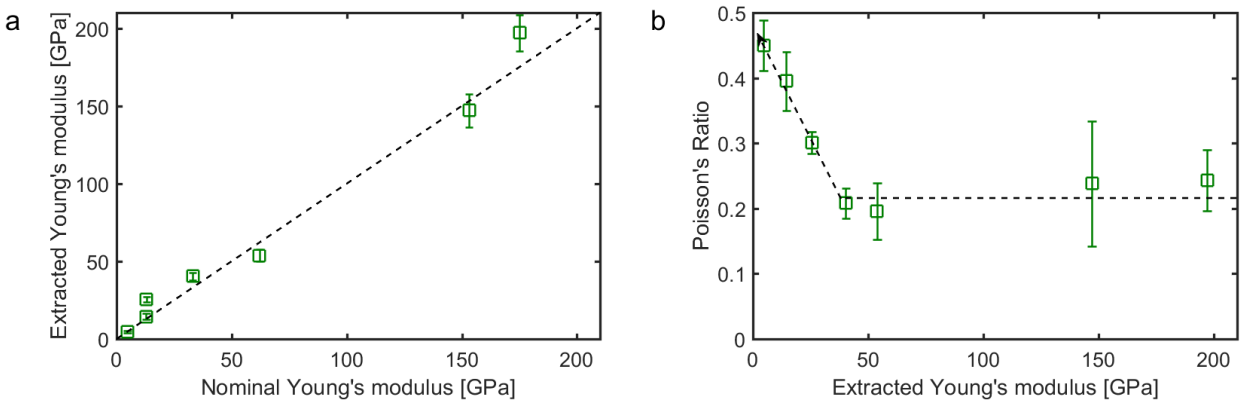

Figure 2: a) Extracted Young's modulus accounting for mass-loading versus nominal Young's modulus. This demonstrates good agreement between the experimental data and nominal values. b) Extracted Poisson's ratio versus Young's modulus, exhibiting a deviation from the expected constant value of Poission's ratio below a critical bond coordination value.

In addition to this series of low-*k* dielectric films, we also successfully excited a 45nm wavelength SAW and observed sub-picosecond echoes from LAWs in a 5nm nominal thickness film. Our analysis determined that this sample could not be a single 5nm thin film, and upon further inspection of the sample, it was confirmed that it consisted of two identical films of unknown thickness with a nitrogen-containing layer in between.

All of these results confirm that coherent EUV nanometrology is a reliable, non-destructive, accurate and robust metrology tool for the elastic properties of thin films, and is highly promising as a way of probing more complex nano-materials. Using the same technique, we are also able to study the thermal properties of nanostructured systems.

4. NANOSCALE THERMAL TRANSPORT

Recent work has shown that the rate of heat dissipation away from an isolated heat source is reduced significantly below the macroscopic Fourier's law prediction when the characteristic dimensions of the source is smaller than the mean free path (MFP) of the dominant heat carriers (phonons in dielectric/semiconductor materials)[7,8,17-19]. More recently, it has also been shown that the period in heat source arrays also plays an important role in determining the heat dissipation efficiency, in a new regime that emerges when the spacing between heat sources is comparable to the dominant carriers' MFP[7,20,21]. These effects are due to a breakdown of a fundamental assumption of heat diffusion: that the length scale over which heat is transported is larger than the MFP of the energy carriers, allowing for the establishment of local temperature averages and a continuous temperature gradient. At length scales that are comparable to or smaller than the phonon MFP, this assumption is no longer valid, and the concept of a continuous temperature gradient leads to unphysically fast heat diffusion predictions. In addition, a realistic model needs to account for the wide distribution of phonon MFPs in materials, going from a few nanometers to hundreds of microns. For a given heat source size, phonons with MFPs shorter than the heat source dimension remain fully diffusive and contribute to efficient heat dissipation and a low effective thermal resistivity (or equivalently, a high effective thermal conductivity). In contrast, phonons with long MFPs travel far from the heat source before scattering, with an effective thermal resistivity larger than the diffusive prediction. Phonons with

intermediate MFPs fall in between; heat transport is quasi-ballistic with varying degrees of reduced contributions to the conduction of heat away from the nanoscale source.

The thermal decay of our samples, as shown in Figure 1b, serves as a direct measurement of the heat dissipation efficiency away from metallic nanowires on different substrates. In past work, we uncovered a previously unobserved regime of nanoscale thermal transport that occurs when the separation between nickel nanowires on sapphire and silicon substrates is smaller than the substrate's phonon MFPs[7]. Instead of the expected steady rise in effective thermal boundary resistivity, r_{eff}, exclusively as a function of the decreasing heat source size, we observed a dependence on the spacing between the heat sources that counteracts the quasi-ballistic reduction in heat dissipation from isolated heat sources. This *collectively-diffusive* regime makes a surprising prediction with important consequences in thermal management: nanoscale heat sources that are spaced closer together will cool down faster than if spaced farther apart.

This prediction can be understood through a suppression function approach, as illustrated in Figure 3b. The linewidth *L* and period *P* of the heat sources define the location and width of an effective filter that suppresses the contributions to the total thermal conductivity of those phonon modes with MFP falling between *L* and *P*. Thus, when only *L* decreases, an increasing number of phonon modes are suppressed relative to the diffusive prediction, resulting in the monotonic rise in effective boundary resistivity[7,8]. However, when *P* is also decreased, phonons originating from neighboring heat sources can interact as though they had originated from one large heat source and the contribution to the thermal conductivity of long MFP phonons is reintroduced. This notch-filter suppression function predicts that fewer phonon modes will have their contribution to the thermal conductivity reduced if nanoscale heat sources are closer together.

This period dependence of nanoscale thermal transport was later reported in similar systems with other experimental techniques[20,21], but with a slightly different interpretation. Instead of a notch-filter suppression function, a monotonic suppression function with an amplitude depending only on the ratio *L/P* is imposed. This difference in interpretation motivates us to increase our experimental database with new experimental geometries so as to test the qualitative predictions of the different models.

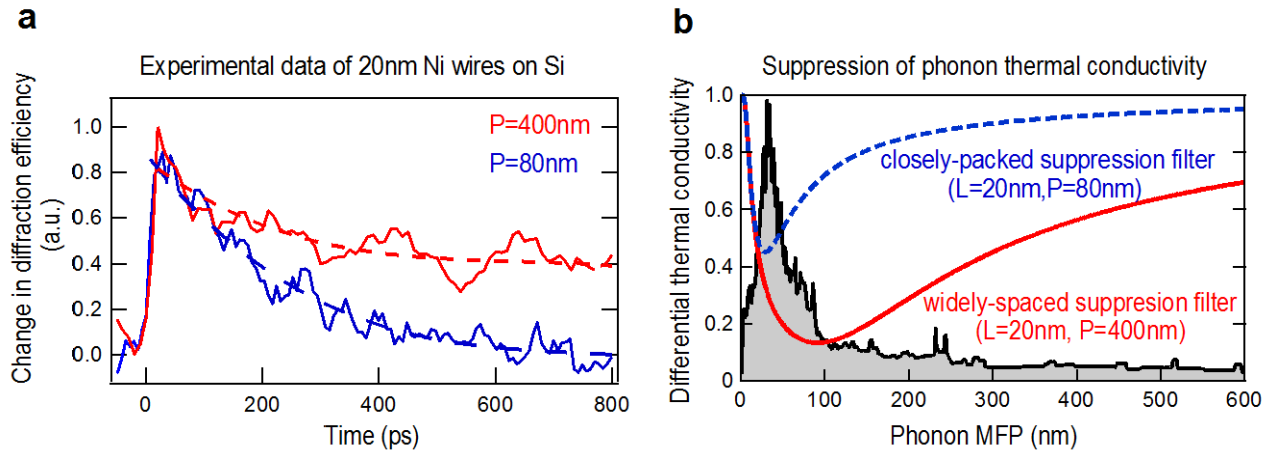

Figure 3: a) A direct comparison of the thermal decay of small linewidth gratings for two different periods validates the prediction of our model, i.e. 20nm linewidth nanowires spaced far apart (red) exhibit a slower initial thermal decay than when closely spaced (blue). The dashed lines provide a guide to the eye through the center of the acoustic oscillations. b) The trends in the heat dissipation efficiency can be understood by the interplay of the differential thermal conductivity spectrum of phonon MFPs in silicon (grey spectrum) and the effective suppression filter introduced by the excitation geometry. The widely-spaced heat sources' suppression filter (red solid line) will suppress the contributions to thermal conductivity of a wider distribution of phonons than its closely-spaced counterpart (blue dashed line).

Thus, we probe the heat dissipation away from nickel nanowire arrays of differing duty cycles on top of a silicon substrate with linewidths varying from 20 to 100nm. Two series of arrays are studied: one where the spacing between the nanowires is kept constant at 400nm and one where the spacing is kept at four times the linewidth (25% duty cycle). Raw data traces shown in Figure 3a of 20nm linewidth nanowires present clear evidence that the heat sources that are widely-spaced (P=400nm) thermally relax slower than the ones that are closely-spaced (P=80nm).

The confirmation of the *collectively-diffusive* predictions using a suppression function gives us a predictive tool to model the thermal behavior of nanostructured systems that can help mitigate thermal management challenges that arise as nanotechnology moves towards the 5nm node. However, these results also point out limitations of using an empirical model that is unable to make quantitative predictions without fitting parameters, and highlights the need for using more fundamental approaches that solve the Boltzmann transport equation in complex geometries.

5. CONCLUSIONS

Coherent EUV nanometrology has reached levels of sensitivity and reliability that allow us to access material properties of nanostructured systems that were otherwise unavailable. It is exquisitly sensitive to picometer-scale surface displacements, which allows us to follow acoustic dynamics with unprecedented small wavelengths, giving us access to the complete elastic tensor of isotropic sub-50nm low-*k* dielectric thin films. We revealed a previously unobserved trend in the Poisson's ratio of these films correlated with bond coordination and other elastic properties. Furthermore, by taking advantage of our technique's sensitivity to small size nanostructures, we probe the heat dissipation away from arrays of nanoscale heat sources with varying periodicities. We experimentally verify the predictions of the recently discovered *collectively-diffusive* regime by directly observing that closely-spaced nanostructures cool down faster than their isolated counterparts. With nanoelectronics components rapidly approaching the 5nm node, metrology tools like coherent EUV nanometrology are key to discovering new, unobserved behaviors and the physics that dominate the nanoworld, and to monitor and control the processes used to fabricate nanostructured devices. Finally, advances in bright high harmonic sources at even shorter wavelengths, and tabletop coherent diffractive imaging, are opening up the capability of even higher spatial resolution nanometrologies[24,25,30,31].

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