# Thursday Afternoon, November 7, 2024

### Spectroscopic Ellipsometry Room 116 - Session EL1-ThA

### **Fundamental Ellipsometry Applications**

Moderators: Tino Hofmann, University of North Carolina at Charlotte, Megan Stokey, Milwaukee School of Engineering

#### 2:15pm EL1-ThA-1 Mueller Matrix Ellipsometry for Optical Metasurfaces, Morten Kildemo, Department of Physics; NTNU;Norway INVITED

We review our recent works using spectroscopic Mueller Matrix Ellipsometry together with full wave modeling that has allowed on one hand to reveal new physics of optical plasmonic or dielectric metasurfaces [1-4], and on the other hand achieve high control of the manufactured metasurfaces. Furthermore, as metasurfaces are at the brink of a commercial breakthrough, we aim at showing that it is likely that this transition will be facilitated through the appropriate use of spectroscopic ellipsometry.

In part one of this talk, we discuss the physics of the full wave models of plasmonic metasurfaces realized from Mueller Matrix Ellipsometry, and in particular address the MME metrology of arrays of Au patches supporting both Gap Surface Plasmons and Surface Plasmon Polaritons[2,3]. A plane wave expansion of the field in the insulator shows that the fundamental localized resonances are composed of oppositely propagating modes, and we produce evidence that the sharp dispersive resonances observed in p-polarization, excited near the opening of diffracted orders, are grating coupled SPPs.

In part two, we discuss our recent work on design, manufacturing and a complete characterization of a class of optical metasurfaces: polarization beam splitting metasurfaces [4]. We describe our recently developed methodology using diffractive mode Mueller matrix spectroscopic ellipsometry (MMSE). Hence, we study both experimentally (and thereby with more conviction also numerically), how well these metasurfaces work in practice. In particular, we show that through appropriate control of the optical properties using MMSE, and feedback to both the nanomanufacture and design step, that we reach accurate and reproducible meta-surfaces. It is recalled that meta-surfaces are based on well-designed nano-resonators (in our case a-Si:H) that are manufactured on a plane interface.

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[2] P. M. Walmsness, N. Hale and M. Kildemo (2021). JOSA B 38 (2021): 2551-2561.

[3] P. M. Walmsness, N. Hale and M. Kildemo Opt. Lett. (2021) (in press).

[4] V. M. Bjelland, N. Hale, N. Schwarz, D. Vala, J. Høvik, and M. Kildemo, Opt. Express 32, 703-721 (2024).

3:00pm EL1-ThA-4 In-Situ Optical Investigation of Electrochemically Controlled Surfaces and Thin Films, *Christoph Cobet*, L. Rosillo Orozco, Johannes Kepler University, Austria; S. Vazquez-Miranda, ELI Beamlines Facility, Czechia; K. Hingerl, Johannes Kepler University, Austria

The applied electrical potential between an electrolyte and a solid electrode, whether it is a metal, semiconductor, polymer or a biomembrane, could initiate versatile surface or film modifications. First of all, the potential simply redistribute charges. But in the new thermodynamic equilibrium adsorbates or even the conformational appearance could change and thus determine the catalytic efficiency of an electrode material, for example. From an experimental point of view, the interfacial electric potential is, on the other hand, a very precise and powerful tool to manipulate thermodynamic equilibrium conditions. It can be modified over a huge range of several eV. Similar effects are otherwise only possible with extreme e.g. temperatures or pressures. However, the fundamental knowledge about the atomic structure and the related processes is still relatively limited compared to classical surface science in vacuum. The reasons are theoretical challenges in the description but primarily experimental limitations as electron based methods like XPS are not applicable at solid-liquid interfaces. Motivated by the increasing interests in this topic, we have started to use optical polarization methods such as spectroscopic ellipsometry (SE) and reflection anisotropy spectroscopy (RAS) to obtain new and complementary in-situ information. From experiments in vacuum or gas phase environment it is known that these methods could provide an exceptional surface sensitivity. This sensitivity Thursday Afternoon, November 7, 2024

allows us to observe the formation of surface quantum well states at a metal-electrolyte interface or an in-situ determination of the electronic band banding at semiconductor surfaces like the polar ZnO [0001] and [000-1] surface.

3:15pm EL1-ThA-5 Thz Electron Paramagnetic Resonance Generalized Spectroscopic Ellipsometry, Bloch Equations and Superconvergence Rules in the Frequency-Dependent Magnetic Susceptibility, *Mathias Schubert*, University of Nebraska-Lincoln, USA; V. Rindert, V. Darakchieva, Lund University, Sweden

A new optical technique is presented to detect the signatures of electron paramagnetic resonances in materials at terahertz frequencies and high magnetic fields using generalized spectroscopic ellipsometry.[1] Measurements dispense with the need for modulation techniques and resonance cavities.[1] The elements of the normalized Mueller matrix are determined, which contain hitherto undetected information about the polarization, frequency, and field response of unpaired electron spin moments including nuclear magnetic coupling.[1,2] Approaches to model analysis of the frequency dependent magnetic susceptibility tensor are discussed, Bloch equations are revisited, and an analogue to the Lyddane-Sachs-Teller relationship is shown from theory and experiment.[3,4] Examples include quantification of the defect properties of Fe and Cr in Ga2O<sub>3</sub>, N in 4H-SiC, and Fe in GaN.

[1] Terahertz electron paramagnetic resonance generalized spectroscopic ellipsometry: The magnetic response of the nitrogen defect in 4H-SiC, M. Schubert, S. Knight, S. Richter, P. Kuehne, V. Stanishev, A. Ruder, M. Stokey, R. Korlacki, K. Irmscher, P. Neugebauer, and V. Darakchieva, Appl. Phys. Lett. 120, 102101 (2022)

[2] Editors Highlights, High-field/high-frequency electron spin resonances of Fe-doped  $\beta$ -Ga2O3 by terahertz generalized ellipsometry: Monoclinic symmetry effects, Steffen Richter, Sean Knight, Oscar Bulancea-Lindvall, Sai Mu, Philipp Kühne, Megan Stokey, Alexander Ruder, Viktor Rindert, Viktor Ivády, Igor A. Abrikosov, Chris G. Van de Walle, Mathias Schubert, and Vanya Darakchieva, Phys. Rev. B. accepted (2024)

[3] The paramagnetic Lyddane-Sachs-Teller relation, Viktor Rindert, Vanya Darakchieva, Tapati Sarkar, Mathias Schubert, arXiv:2405.15382 [cond-mat.mtrl-sci]

[4] Bloch equations in Terahertz magnetic-resonance ellipsometry, Viktor Rindert, Steffen Richter, Philipp Kühne, Alexander Ruder, Vanya Darakchieva, Mathias Schubert, arXiv:2404.12805 [cond-mat.mtrl-sci]

### Spectroscopic Ellipsometry Room 116 - Session EL2-ThA

# Evolving Methodology and Analytical Methods of Ellipsometry

Moderators: Nikolas Podraza, University of Toledo, Mathias Schubert, University of Nebraska - Lincoln

### 3:30pm EL2-ThA-6 Dirty Ellipsometry: Finding Success with Nonideal Samples and Nonideal Data in a Nonideal World, Maxwell Junda, A. Green, X. Li, Covalent Metrology INVITED

Ellipsometry has tremendous utility in commercial pursuits of science and engineering across a range of industries. One of the missions of Covalent Metrology is to make ellipsometry available to as broad a range of applications and users as possible. While much of the industrial scale ellipsometry used in production process monitoring involves repeated measurements of similar samples, Covalent's ellipsometry projects typically have an R&D flavor and are often one-time experiments meant to answer a specific question. Consequently, unavoidable constraints can sometimes lead to data or results that are hampered by nonidealities, and the overall project falls short of the clean results showcased in much of published literature (or at least the vision in our minds upon conceiving the experiments). Several common nonidealities can be understood strictly from a technical perspective: samples are not flat or too rough, films are too thin or inhomogeneous, or a multilayer structure has too many unknown layers. However, there are multiple other less obvious or less visible challenges to be navigated in providing primarily industry-focused ellipsometry measurement services. One example is that it's common to have incomplete information about test samples while planning an experiment, either due to intellectual property-related secrecy, or sometimes a simple lack of knowledge. Budget constraints are another typical nonideality where practicality dictates finding ways to obtain the

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best possible results to challenging, sometimes open-ended questions within a limited amount of time.

Covalent Metrology has worked on numerous projects in recent years wherein spectroscopic ellipsometry has produced useful results, despite "ugly" optical models, largely unknown samples, or rushed timelines. Specific examples will be described including: (1) optical models for highly inhomogeneous films that have quantitatively poor fits to noisy data yet can still provide key results, (2) use of spectroscopic ellipsometry to monitor the mechanical lapping of layered samples to thin the surface layer to submicron thickness and (3) pairing ellipsometry with other metrologies (such as TEM or XPS) to reverse engineer multi-layer optical filter stacks, the details of which were completely unknown at the project start. Lessons in evaluating the feasibility of hypothetical experiments will also be discussed, focusing on key descriptive factors of sample and experiment that gate project success, and on the value of ellipsometric simulations to both test measurement viability and tune experiment parameters prior to sample measurement.

### 4:00pm EL2-ThA-8 Numerical Ellipsometry: AI for Real-Time, in situ Process Control for Absorbing Films Growing on Unknown Transparent Substrates, Frank Urban, D. Barton, Florida International University

Ellipsometry is an optical analytical method in which desired reflecting surface parameters are related to measurements by mathematical models. Recent work has shown that using AI methods can result in predicting reflecting surface parameters faster and more easily than by using iterative methods. This prior AI work used artificial neural networks applied to a growing absorbing film on a known substrate. Each different substrate required a set of separately trained networks across the wavelength spectrum thus necessitating training a new set of networks for each new substrate. The work presented here does not require substrate optical property data. Thus one set of spectroscopic networks can serve a large number of different substrates. This becomes possible by increasing the number of measurements per wavelength from two to three. For now we consider transparent substrates for which  $k_2 = 0$  or near zero. As before the non-iterative, stable, and fast performance lends itself to real-time, in situ monitoring of thin film growth. Examples for such growth of an absorbing metal film, chromium, will be given using two different substrates as proof of concept. The multilayer perceptron configuration consists of 6 input and 6 output neurons with two hidden layers of 80 neurons each. Solutions are performed at each wavelength independently and do not rely on fitting functions for optical properties.

# 4:15pm EL2-ThA-9 Gaining Insight Into InAs Plasma Treatments and Passivation via *in situ* Spectroscopic Ellipsometry, John Murphy, G. Jeringan, J. Nolde, Naval Research Laboratory

Indium Arsenide (InAs) is a crucial material for infrared photodiode fabrication. However, its performance is severely hindered by the spontaneous formation of a complex native oxide on its surface, which leads to a high surface state density. This density pins the Fermi level within the conduction band, promoting the formation of shunt paths and increasing the surface recombination velocity. Consequently, these effects contribute to increased dark current and degraded detector performance in InAs-based devices.

To improve the performance of InAs-based devices, a suitable passivation of these dangling bonds must be developed. Atomic layer processing techniques, including remote plasma treatments and self-cleaning processes involving metalorganic precursors, have been explored as possible solutions. These methods aim to control and improve the InAs surface by removing the native oxide and subsequently passivating it with wide-gap oxides using plasma-enhanced or thermal atomic layer deposition (PE-ALD).

When utilized in conjunction with ALD, in situ spectroscopic ellipsometry has proven invaluable to study ALD oxide growth; however, it can also be utilized to assess the quality of the InAs surface during plasma treatments. In this study, we employ *in situ* critical point analysis of pseudo-dielectric functions to evaluate the efficacy of remote plasma treatments in cleaning InAs (100) surfaces. We will correlate changes in the pseudo-dielectric function with alterations in surface morphology, as measured by atomic force microscopy, and surface chemistry, assessed via x-ray photoelectron spectroscopy. An air-free transfer apparatus will be used to prevent reoxidation of the surface. Finally, we will characterize the surface state density of plasma-treated and Al2O3-passivated InAs surfaces using capacitance-voltage measurements.

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### Spectroscopic Ellipsometry Room Central Hall - Session EL-ThP

### Spectroscopic Ellipsometry Poster Session

EL-ThP-1 Training Neural Networks with Simulated Spectroscopic Ellipsometry Data for Cadmium Telluride Photovoltaic Applications, *Alexander Bordovalos, B. Ramanujam, A. Shan, N. Podraza*, University of Toledo

Ellipsometric spectra can be simulated if the thicknesses and complex index of refraction (N = n + ik) spectra are known for the substrate and each layer of the film stack, such as, for example cadmium telluride (CdTe) based thin film solar cells. The ability to generate large data sets of simulated ellipsometric spectra enables the application of machine learning algorithms to spectroscopic ellipsometry data analysis tasks. Based on information learned from the analysis of ellipsometric spectra collected from real CdTe thin films and solar cells, ellipsometric spectra are simulated to assess characterization challenges that could be notably enhanced by machine learning. A promising machine learning task is compositional mapping. A bilayer structure of soda-lime glass / cadmium selenide telluride (CdSe(1-x)Te(x)) where x is one of nine values between 0.037 -0.81 / cadmium telluride (CdTe) is selected as a proof of concept structure to simulate ellipsometric spectra for training a neural network. Large numbers of data sets are generated for each alloy composition and the thicknesses of the CdSe(1-x)Te(x) and CdTe layers are randomly selected from a common range for photovoltaic applications. A neural network is trained with these data sets to determine which of the nine possible CdSe(1-x)Te(x) compositions was used to simulate the ellipsometry data as well as predict the thickness for the CdSe(1-x)Te(x) and CdTe in the bilayer stack. The neural network determined the correct CdSe(1-x)Te(x) composition 99% of the time on the test set, and 95% of the layer thickness predictions for the CdSe(1-x)Te(x) are within 10% of the ground truth values while 95% of the layer thickness predictions for the CdTe are within 1.2% of the ground truth values. The development of rapid compositional mapping from spectroscopic ellipsometry data could be highly impactful for determining compositional uniformity for thin-film technologies. This approach can be developed further to examine compositional gradient profiles within the film stacks.

### EL-ThP-2 Numerical Ellipsometry: Artificial Intelligence for rapid analysis of Indium Tin Oxide films on Silicon, Frank Urban, D. Barton, Florida International University

Ellipsometry is a well-known material analytical method widely used to measure thickness and optical properties of thin films and surfaces across a wide range of industrial and research applications including critical dimensions in chipmaking. The method employs the fact that light undergoes a change in polarization state upon reflection from or transmission through a material. The desired properties of the surface structure are related to measurements by the electromagnetic models expressed by Maxwells equations as well as models of material properties. The work here demonstrates the use of Artificial Intelligence (AI) in the form of a multilayer perceptron artificial neural network to apply the electromagnetic model. The reflecting surface examined here is composed of indium tin oxide films (ITO) approximately 400 nm in thickness deposited on silicon substrates. Solutions are provided by 299 artificial neural networks, one per wavelength from 210 nm to 1700 nm across which ITO exhibits transparent as well as absorbing characteristics. Thus, it serves as a proxy for a wide range of other materials. To train the network, simulated measurements are computed at two thicknesses which differ randomly by 1 to 6 nm and at three different incidence angles, 55°, 65°, and 75°. Following training, results are obtained in less than one second on a conventional desktop computer.

### **EL-ThP-3 Accurate Determination of Critical-Point Parameters in Spectra**, *L. Le*, Vietnam Academy of Science and Technology, Viet Nam; Y. Kim, Kyung Hee University, Republic of Korea; *David Aspnes*, North Carolina State University

The determination of the locations of critical points of overlapping features in spectra, optical and otherwise, continues to be a challenge even in the absence of complications due to baseline effects and noise.Recent progress in the use of nonlinear (maximum-entropy) methods to eliminate the noise and apodization artifacts characteristic of linear filters, and to detect weak singularities that otherwise would be overlooked, has highlighted the importance of working directly with low-order Fourier coefficients, where information is separated from baseline effects and noise.Here, we report the results of a systematic investigation of critical-point determination in both direct and reciprocal space, with emphasis not only on accuracy but also on uncertainty, using multiple averages of spectra with added noise.Reciprocal-space analysis can be viewed as the logical limit of classic derivative methods of extracting these parameters, which draw conclusions from a relatively narrow range of information-containing coefficients.

# EL-ThP-4 Optical Analysis of Ferroelectric PLZT Films Using Spectroscopic Ellipsometry, *S. Kotru, Sneha Kothapally,* The University of Alabama; *J. Hilfiker,* J. A. Woollam Co., Inc.

Spectroscopic ellipsometry was utilized to study the optical properties of ferroelectric lead lanthanum zirconate titanate (PLZT) films. These films were deposited on platinized silicon [Si(100)/ SiO<sub>2</sub>/TiO<sub>2</sub>/Pt(111)] substrates using the chemical solution deposition method. Films were annealed at two different temperatures (650 and 750 °C) using rapid thermal annealing. Shimadzu UV-1800 UV-VIS spectrophotometer with a resolution of 1 nm was used to measure the reflectance data in the spectral range of 300-1000 nm with a step size of 1 nm. The bandgap values were determined from the reflectance spectra using appropriate equations. A J.A. Woollam RC2 small spot spectroscopic ellipsometer was used to obtain the change in amplitude ( $\Psi$ ) and phase ( $\Delta$ ) of polarized light upon reflection from the film surface. The spectra were recorded in the wavelength range of 210-1500 nm at an incident angle of 65°. Refractive index (n) and extinction coefficient (k) were obtained by fitting the spectra ( $\Psi$ ,  $\Delta$ ) with the appropriate models. No significant changes were observed in the optical constants of PLZT films annealed at 650 and 750 °C. The optical transparency and the strong absorption in the ultraviolet (UV) region of PLZT films make them an attractive material for optoelectronic and UV sensing applications.

EL-ThP-6 Spectrally Resolved Absorption Based Kuhn's Dissymmetry Factor from Mueller Matrix Polarimetry, Ufuk Kilic, University of Nebraska-Lincoln; A. Ruder, M. Hilfiker, Onto Innovation; S. Wimer, University of Nebraska Lincoln; E. Schubert, M. Schubert, University of Nebraska-Lincoln Chirality or handedness is one of the most intriguing material properties of an object which cannot be made superimposable on its mirror image [1]. Within the last few decades, this symmetry breaking phenomenon has attracted great attention due to its application in various subdisciplines of physics, chemistry and biology [2-4]. The optical manifestation of chirality known as circular dichroism (CD) which is the difference between the absorbance (A) of left circularly of polarized (LCP) light from the right circularly polarized (RCP) light. Recently, Kuhn's dissymmetry factor, socalled g-factor (given as g-factor=2CD/(A<sub>LCP</sub>+A<sub>RCP</sub>)) is employed as a metric to quantify and compare the chiroptical ability of various systems: nanostructures, molecules, and thin films independent from their fabrication and characterization methods [5]. In this study, we provide a route to reach out the Kuhn's dissymmetry factor in terms of Mueller matrix elements for accurate evaluation and investigation of the chiroptical response of nanostructured thin film samples fabricated via glancing angle deposition technique [6-7]. Therefore, due to the semi-transparent nature of such fabricated structures, it is necessary to reach out both reflectivity and transmissivity properties. Hence, in this study, we present and discuss full road map of obtaining the spectral evolution of Kuhn's dissymmetry factor that utilizes a combinatorial reflection and transmission mode generalized spectroscopic ellipsometry in Mueller matrix configuration. Our method offers the differentiation of circular dichroism information from the other optical anisotropy types which paves the way through a universal and robust method for direct extraction of chirality.

#### References

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2. M. W. Glasscott, A. D. Pendergast, S. Goines, A. R. Bishop, A. T. Hoang, C. Renault, and J. E. Dick, Nature communications 10, 1 (2019). 8

3. J. P. Mazaleyrat and D. J. Cram, Journal of the American Chemical Society 103, 4585 (1981).

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EL-ThP-7 The Optical Constants of Calcium Fluoride from 0.03 - 9 eV, Jaden R. Love, C. Armenta, New Mexico State University; M. Stokey, M. Schubert, University of Nebraska - Lincoln; S. Zollner, New Mexico State University

In this presentation we describe the optical properties of calcium fluoride (CaF<sub>2</sub>), an insulating material for which most optical studies were conducted in the 1960's and are discussed in [1]. Our goal is to reexamine the optical constants of CaF<sub>2</sub> (111) and (100) MTI manufactured substrates in the range 0 - 9 eV using modern ellipsometry equipment and analysis techniques. From these early studies CaF2 is known to have an ultrawide bandgap of 12 eV, a large exciton binding energy of 1 eV, and a wide transparency range from 125 meV - 10 eV. The large range of transparency makes CaF<sub>2</sub> a suitable material for use in optical components such as those found in infrared detectors and telescopes.

CaF<sub>2</sub> crystalizes in the FCC fluorite structure within the space group Fm-3m and has a lattice constant of 5.4626 Å. Ca 2<sup>+</sup> atoms are in the Wyckoff (4a) position at the origin. F atoms are at the (8c) positions (14, 14, 14) and (14, 14,  $3_{4}$ ). CaF<sub>2</sub> has a three-fold degenerate Raman-active T<sub>2g</sub> mode and a threefold degenerate infrared active  $T_{2u}$  mode. The  $T_{2u}$  mode splits into a transverse optical (TO) doublet and a longitudinal optical (LO) singlet that were observed using infrared ellipsometry and modeled with a Lorentzian. The TO and LO energies are 261 and 477 cm<sup>-1</sup> , respectively, with an amplitude A = 4.1, a broadening of 4  $cm^{-1}$ , and a high-frequency dielectric constant of 1.98(1). There is a dip in the reststrahlen band formed by two phonon absorption that was modeled using an anharmonically broadened Lorentzian.

Data for the visible and near ultraviolet was obtained using an RC2 ellipsometer and VUV-SE at the University of Nebraska - Lincoln. The data sets were merged to form a continuous spectrum over the range from 0 – 9 eV. In this region the data shows normal dispersion that is modeled using a Tauc-Lorentz oscillator at 7.24 eV and a pole at 9.24 eV. The pseudodielectric function  $\langle \epsilon_2 \rangle$  is negative above 3 eV indicating the presence of a surface layer with larger refractive index than that of  $CaF_2$ . In the region between 7 – 9 eV there is a small peak in  $\langle \epsilon_2 \rangle$  modeled by a Gaussian at 7.6 eV with an amplitude A = 0.05. A Cauchy layer was added above the bulk CaF<sub>2</sub> substrate to improve the fit. We determined that the surface layer present on the samples has a thickness of 15 Å.

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#### EL-ThP-8 Metrology of Ultrathin Iron Catalyst Films by Spectroscopic Ellipsometry, Nicholas Allen, R. Vanfleet, M. Linford, D. Allred, R. Davis, Brigham Young University

Vertically aligned carbon nanotube (VACNT) forest growth is a chemical vapor deposition process that uses a thin-film iron catalyst on an alumina support. Iron catalyst thickness for VACNT growth is typically from ~1-10 nm and thickness strongly affects forest morphology. Consequently, characterization of iron thin films could improve process control of VACNT growth. In this study, we explored the use of spectroscopic ellipsometry (SE) to measure these iron thin films because the technique is rapid, highly sensitive, and non-destructive. SE does have challenges, however, as it is difficult to break the correlation in the analysis between the optical constants and thickness of ultra-thin films. The partial oxidization and the absorptive nature of the iron/iron oxide films adds further difficulty. We performed a multi-sample analysis of thermally evaporated iron films with target thicknesses of 1, 2, 4, 7, and 14 nm. To improve sensitivity, we used contrast enhancement by incorporating a 350 nm silicon oxide layer under the iron film and alumina support. We used a consecutive layers approach, collecting SE data and fitting the model for each film before depositing the next. We found high sensitivity to iron thickness with SE, with reproducible thickness results. We modeled the iron/iron oxide film as a composite with an effective medium approximation layer. Our SE model fits the data well with a relatively low mean squared error. We explored the sensitivity of iron/iron oxide thickness results to errors in the alumina thickness while using the consecutive layers approach, which explained most of the variance we found in measured iron thickness.

### EL-ThP-9 IR Ellipsometry on Thermally Oxidized Germanium (100), D. Ortega, Danissa Ortega, A. Moses, C. Armenta, J. Love, S. Yadav, S. Zollner, New Mexico State University

The use of germanium as a semiconductor has been heavily studied but further details about its thermal oxidation, more specifically: its growth methods and process parameters, can show its promise for widespread optoelectronic applications. By performing a Deal-Grove thermal oxidation on bulk Ge, its infrared (combined with ultra-violet) dielectric function can

be determined by utilizing J.A. Woollam Vertical Angle Spectroscopic Ellipsometers (VASE) through ellipsometric angles  $\psi$  and  $\Delta$ .

The cleaved 5x10 mm samples were first ultrasonically cleaned in deionized water at room temperature and dried with nitrogen to achieve a native oxide layer of ~1 nm. The samples were then rapidly thermally annealed in ultrapure oxygen at temperatures ranging from 525-575°C in 40 psi pressure for up to 2 hours, reaching thermal oxide layers as high as 35 nm. Spectroscopic ellipsometry measurements were taken using the IR-VASE and UV-VASE to achieve its dielectric function of  $\psi$  and  $\Delta$  in the photon energy range of 0.05-6 eV at room temperature. IR-VASE and UV-VASE measurements were taken at incidence angles from 50-80° which revealed excitations at photon energies ranging 0.09-0.14 eV. The dielectric function was characterized using a two-layer model: the Ge substrate, then the GeO2 oxide. In the UV range, a Tauc-Lorentz model was utilized, which characterized the fit parameters in the IR range. In the IR range, a Gaussian model fit with peak energy at 0.015 presented a lower MSE than if it were fit with a Tauc-Lorentz or two Gaussians. The modelling will continue to be improved with further growth of the thermal oxide layer at longer annealing times.

# Friday Morning, November 8, 2024

### Spectroscopic Ellipsometry Room 116 - Session EL-FrM

### **Emerging Applications and Workforce Development**

**Moderators: Ufuk Kilic**, University of Nebraska - Lincoln, **Stefan Zollner**, New Mexico State University

8:15am EL-FrM-1 Singular Propagation States of Electromagnetic Waves in Anisotropic Media, Chris Sturm, University Leipzig, Germany INVITED In optically anisotropic media, there are generally two eigenmodes for a given propagation direction, which propagate without changing their polarization. However, for certain directions, these two eigenmodes can completely degenerate, in their eigenstate and eigenvalue. In this case, only one well-defined state can propagate without changing its polarization and this direction corresponds to an exceptional point (EP) in the momentum space. In recent years, a growing interest in the fundamental physical properties of exceptional points (EP) and their use in applications has led to a significant increase in research activity in this area. The existence of such points was first reported by W. Voigt in 1902 for orthorhombic materials [1], who realized that in these materials at certain propagation directions, the propagation properties (complex refractive index) and the polarization of the two eigenmodes are simultaneously degenerated. Only a wave, either left or right circularly polarized, can propagate along such a direction without changing its state. It took almost 100 years, that the general case was discussed by Berry and Dennis in 2003 [2].

Here we present on overview on the EP in optically anisotropic materials and show that these points occur naturally in these systems [3]. In the presence of interfaces, the properties of the EP can be tuned, which is of particular of interest for technical applications [4]. Due to of the singular eigenstate at the EP, the typical used approach to describe the propagation as well as the reflection and transmission properties of an arbitrarily polarized wave at an interface by a superposition of the two eigenmodes is no longer applicable. In this case, an extension of the solution of the wave equation by a spatially dependent amplitude must be considered [3]. The results are illustrated by using the optical properties of real materials, which are used in current research, e.g., ZnO, KTP and  $\beta$ -Ga2O3.

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[2]M. V. Berry and M. R. Dennis, Proc. R. Soc. London, Ser. A. **459**, 1261 (2003).

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[4]S. Richter et al., Phys. Rev. Lett. 123(22) 227401 (2019).

8:45am EL-FrM-3 Infrared-Active Phonon Modes in Variably Alloyed Bulk β-(AlXGa1-X)2O3 Determined by Mueller-Matrix Spectroscopic Ellipsometry, *Preston Sorensen*, *I. Green*, University of Nebraska - Lincoln; *M. Stokey*, Milwaukee School of Engineering; *A. Mauze*, *Y. Zhang*, University of California Santa Barbara; *J. Speck*, University of California at Santa Barbara; *V. Stanishev*, *V. Darakchieva*, Lund University, Sweden; *Z. Galazka*, ikz berlin, Germany; *M. Schubert*, University of Nebraska - Lincoln

The monoclinic beta phase of gallium oxide is an ultra-wide bandgap semiconductor that has been widely studied for potential use in high power switching applications. As crystal growth methods have improved, we are able to investigate high quality (Al<sub>x</sub>Ga<sub>1-x</sub>)<sub>2</sub>O<sub>3</sub> films and bulk substrates, which have been desired due to the increase in band gap with increasing Al content. Here, we study the near and mid infrared-active phonon modes of highly alloyed (x = 0.05, 0.1, 0.15, 0.2, 0.25, 0.3, and 0.35) bulk (Al<sub>x</sub>Ga<sub>1-x</sub>)<sub>2</sub>O<sub>3</sub> with (100) surface orientation. We use generalized spectroscopic ellipsometry and implemented the previously described eigenpolarization model for phonon modes in monoclinic structure (Al<sub>x</sub>Ga<sub>1-x</sub>)<sub>2</sub>O<sub>3</sub>. We discuss the observed phonon mode parameter trends associated with alloying composition. We also investigate pseudomporhically-strained epitaxial films with x = 0.046, 0.097, and 0.163 grown on beta-phase (010)  $Ga_2O_3$ . We use our previously calculated phonon deformation potential parameter values and elastic coefficients and differentiate between strain and alloying induced phonon mode property variations as a function of Al content.

### 9:00am EL-FrM-4 Predicting Perovskite Photovoltaics Performance from Spectroscopic Ellipsometry, *Emily Amonette*, K. Dolia, Y. Yan, Z. Song, N. Podraza, University of Toledo

Complete wide band gap  $FA_{0.8}Cs_{0.2}Pb(I_{0.6}Br_{0.4})_3$  perovskite photovoltaic (PV) devices are measured by spectroscopic ellipsometry in the through-the-glass configuration and analyzed to determine complex optical property

spectra of the perovskite absorber properties as well as structural properties which are then used to simulate external quantum efficiency (EQE) spectra and to calculate PV device performance parameters such as short circuit current density, open circuit voltage, fill factor, and power conversion efficiency. Mapping spectroscopic ellipsometry measurements of an incomplete device are also collected from the perovskite film side to obtain layer thicknesses, perovskite band gap energies, and Urbach energies at each mapping point. These values are used to predict various PV device performance parameters such as short circuit current density, open circuit voltage, fill factor, and power conversion efficiency with the goal of increasing the accuracy of these predictions by comparing them to experimentally obtained parameters. The incomplete devices consist of glass superstrate / indium tin oxide front electrical contact / hole transport layer / perovskite absorber, while the complete devices consist of these components as well as an electron transport layer and silver back electrical contact. Simulations and calculations tend to overestimate PV device performance parameters, undermining the accuracy and usefulness of simulations. When these simulations are based on structural and optical properties obtained from spectroscopic ellipsometry measurements of incomplete perovskite films rather than complete PV devices, further inaccuracies arise as characteristics of that layer in the exposed perovskite film do not necessarily share the same of a complete, protected PV device. By comparing experimental PV performance parameters based on measured characteristics of the same devices under different assumptions in the modeling approach, the accuracy of simulated performance parameters are evaluated, and improvements in the models are implemented. The usefulness of this is apparent in situations where experimentally measuring PV device performance is unfeasible or overly tedious, as well as during intermediate steps during production.

9:15am EL-FrM-5 Vacuum and Extreme Ultraviolet Scatterometry for Critical Dimension Metrology, Thomas Germer, B. Barnes, S. Moffitt, S. Grantham, M. Sohn, D. Sunday, E. Shirley, National Institute of Standards and Technology (NIST) Scatterometry is often used to perform linewidth process monitoring in

Scatterometry is often used to perform linewidth process monitoring in semiconductor manufacturing. Dimensional parameters of a periodic structure are fit to the optical signature, such as that obtained by a spectroscopic ellipsometer. The method has been typically limited to wavelengths in the near-infrared, visible, or ultraviolet, where traditional refractive optics are available for focusing and polarization control. As features are being fabricated with smaller dimensions, there is a need to employ shorter wavelengths to achieve the accuracy and precision targets. In this work, we are exploring the use of scatterometry in the vacuum and extreme ultraviolet (VUV and EUV), motivated by our finding that differentiating optical signatures should extend well into this spectral region; that compact VUV/EUV sources, such as high harmonic generation (HHG), are becoming commercially available; and that optical elements, such as phase retarders and polarizers, can be constructed using reflective optics, albeit with non-optimal attributes. In this talk, we will discuss each of these three motivations, and describe the technical challenges and opportunities that they present.

9:45am EL-FrM-7 Immersion Ellipsometry of Ultrathin Films - Breaking the Correlation between Index of Refraction and Film Thickness, Samira Jafari, Brigham Young University; B. Johs, Film Sense; M. Linford, Brigham Young University

The optical constants and thicknesses of ultrathin (<5 - 10 nm) films are correlated in traditional ellipsometric measurements. Accordingly, most ellipsometric measurements of these films involves assuming an index of refraction for them. This work describes the use of immersion ellipsometry to break the correlation between optical constants and film thickness, allowing both to be measured in an experiment. In immersion ellipsometry, ellipsometric data is acquired in both air and liquid ambients, and the two data sets are combined in the analysis. The measurement under liquid adds information to the analysis that breaks the correlation between the film thickness and refractive index that exists for air-only ellipsometric measurements. We describe the use of multi-wavelength immersion ellipsometry (MWIE) to sequentially measure both the thicknesses and optical constants of two ultrathin thin films: native oxide on silicon and an alkyl monolayer of chloro(dimethyl)octadecylsilane (CDMOS) on that native oxide. The average thicknesses of the native oxide and CDMOS monolayer were 1.526 ± 0.027 nm and 1.968 ± 0.057 nm, and their average indices of refraction at 633 nm were  $1.519 \pm 0.005$  and  $1.471 \pm 0.004$ , respectively. The native oxide and CDMOS monolaver were also characterized with X-ray photoelectron spectroscopy (XPS) and contact angle goniometry. As expected, both the XPS C 1s peak and the water contact angle increase

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substantially after monolayer deposition. While immersion ellipsometry has been known for years, its use has been limited, probably because of a lack of awareness of the technique and/or the lack of readily available accessories for performing the experiment in many laboratories. As ultrathin films become more important, immersion ellipsometry should increase in importance as a means of characterizing them.

### 10:00am EL-FrM-8 Temperature Dependence of the Long-Wavelength Lattice Vibrations of NiO (111) Using Infrared Spectroscopic Ellipsometry from 25 K to 500 K, Yoshitha Hettige, J. Love, C. Armenta, A. Moses, J. Marquez, S. Zollner, New Mexico State University

NiO has a cubic rock salt structure, with two optical phonon modes called transverse (TO) and longitudinal (LO). The frequency of these TO and LO modes is related to the reduced mass and the strength of the bond between the Ni<sup>2+</sup> and O<sup>2-</sup> ions. These vibrations can be analyzed by measuring the ellipsometric angles  $\Psi$  and  $\Delta$  on an infrared ellipsometer.

A NiO (111) sample was mounted inside a Lakeshore Janis ST-400 cryostat. We measured the ellipsometric angles  $\Psi$  and  $\Delta$  of NiO at room temperature at a resolution of 8 cm<sup>-1</sup> from 250 to 8000 cm<sup>-1</sup> on J. A. Woollam IR-VASE Mark II ellipsometer at 70° angle of incidence. Then, we cooled the NiO using liquid He and measured the ellipsometric angles  $\Psi$  and  $\Delta$  between 25 and 500 K with a step size of 25 K under the same conditions. Low temperatures are achieved with a Lakeshore RCG4 recirculating helium cooler.

In our data analysis, we used one Lorentzian for the TO phonon absorption at ~400 cm<sup>-1</sup> and another one for the two-phonon absorption (TO+TA) at ~560 cm<sup>-1</sup>. The two edges of the reststrahlen band correspond to the TO energy (strong peak in the pseudodielectric function) and the LO energy (strong peak in the pseudoloss function). We found that the phonon energy has a redshift and increased broadening for increasing temperature due to the anharmonic decay of optical phonons. This temperature dependence of the phonon parameters are related to the self-energy of anharmonic decay. We fit the energy using the Bose-Einstein model which describes the data well. We found that the energy of the decay product is 64 meV, much larger than the TA(X) energy (16.5 meV). This unexpected result is probably because of the scattering of phonons by two magnons with an energy of about 190 meV.

### 10:30am EL-FrM-10 Far-Infrared Mueller Matrix Ellipsometry and Vortex Beam Spectroscopy Using Synchrotron Radiation, Andrei Sirenko, New Jersey Institute of Technology INVITED

Recent results for development of the new multi-user setup for lowtemperature Ellipsometry and transmission/reflection vortex beam polarimetry in high magnetic fields will be presented. The instrument has been installed at the synchrotron radiation source - the MET beamline of NSLS-II in Brookhaven National Laboratory. This instrument is able to acquire experimental data in all three major scenarios of rotating analyzer ellipsometry (RAE), rotating compensator ellipsometry (RCE), and full-Mueller matrix ellipsometry (MM-SE) in the spectral range between 5 cm<sup>-1</sup> and 10,000 cm<sup>-1</sup> with the synchrotron radiation as the light source. A wide range of angles of incidence AOI between 70° and 85° is enabled by the  $\theta$ - $2\theta$  goniometer. The ellipsometer has magnetic fields of up to  $\pm 7$  T with a capability to switch quickly between the exact Faraday and Voigt configurations for direction of the magnetic field with respect to the sample surface. Data analysis is based on the Berreman's 4 × 4 propagation matrix formalism to calculate the Mueller matrix parameters of anisotropic samples with magnetic permeability  $\mu$  different from 1. A nonlinear regression of the rotating analyzer ellipsometry and/or Mueller matrix spectra, which are usually acquired at variable angles of incidence and sample crystallographic orientations, allows extraction of dielectric constant and magnetic permeability tensors for bulk and thin-film samples.

In addition to the ellipsometric measurements, our setup is capable of producing the vortex beams of the synchrotron radiation with a distinct integer values of the orbital angular momentum (OAM). Recently we demonstrated that the vortex light with OAM can effectively couple to magnetism exhibiting dichroism in a magnetized medium. The vortex beams with various combinations of the OAM L=+/-1, +/-2, +/-3, and +/-4 and spin angular momentum S = +/-1, or conventional circular polarization, were used for studies of the magnon spectra in TbFe<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub>, h-Ni<sub>3</sub>TeO<sub>6</sub>, and h-Lu<sub>0.6</sub>Sc<sub>0.4</sub>FeO<sub>3</sub>single crystals. We observed strong vortex beam dichroism for the magnon doublets, which are split in an external magnetic field. The absorption conditions at the magnon resonances depend on the total angular momentum of light *J* that is determined by *J*=S+*L*. For the higher orders of *L*, the selection rules for AFM resonances dictated by *L* completely dominate over that for conventional circular polarization. A possibility to

expand the vortex beam spectroscopy to the abroad class of the electronic systems in quantum matters will be discussed.

Parts of this work were performed in collaboration with V. Martinez, P. Marsik, L. Bugnon, C. Bernhard, V. Kiryukhin, and S.-W. Cheong.

### 11:00am EL-FrM-12 Infrared Dielectric Function of Thiazolothiazole Embedded Polymer Films Determined by Spectroscopic Ellipsometry, *Nuren Shuchi*, *T. Adams*, *D. Louisos*, *G. Boreman*, *M. Walter*, *T. Hofmann*, University of North Carolina at Charlotte

Organic photochromic polymers, whose photo-chemical and optical properties can be altered through optical stimulation, are found in diverse applications ranging from tinted lenses and smart windows to memory devices, actuators, tunable filters, and holographic gratings [1-4]. Recently, extended viologens containing the thiazolo[5,4-d]thiazole (TTz) backbone are increasingly attracting interest due to their strong fluorescence, solution-processability and reversible photochromic transition. Especially, dipyridinium thiazolo[5,4-d]thiazole viologen exhibits high-contrast, fast and reversible photochromic changes. When exposed to radiation with an energy larger than 3.1 eV, it transitions from light yellow (TTz<sup>2+</sup>) to purple (TTz<sup>-+</sup>) to blue (TTz<sup>0</sup>) state due to two distinct, photo-induced single electron reductions [5]. The accurate knowledge of the complex dielectric function is essential for the design and fabrication of TTz-based optically tunable devices. The complex dielectric function of a non-photochromic TTz derivative and a photochromic TTz-embedded polymer has been determined previously in the visible and near-infrared spectral range [6,7] using spectroscopic ellipsometry.

In this presentation, we will discuss spectroscopic ellipsometry data obtained from bulk sample of photochromic thiazolo[5,4-d]thiazole embedded in polymer. The measurements were taken before and after irradiation with a 405 nm diode laser in the infrared spectral range from 500 cm<sup>-1</sup> to 1800 cm<sup>-1</sup>. The model dielectric functions of the thiazolothiazole embedded polymer film for its TTz<sup>2+</sup> (unirradiated) and TTz<sup>0</sup> (irradiated) states are composed of a series of Lorentz oscillators in the measured spectral range. A comparison of the obtained complex dielectric functions for the TTz<sup>2+</sup> and TTz<sup>0</sup> state shows that the oscillators located in the spectral ranges 500 cm<sup>-1</sup>- 700 cm<sup>-1</sup>, 1300 cm<sup>-1</sup>- 1400 cm<sup>-1</sup>, and 1500 cm<sup>-1</sup>- 1700 cm<sup>-1</sup> change in both amplitude and resonant frequency upon transition between the states. In addition, a resonance has been identified at approximately 1050 cm<sup>-1</sup>, for which, only a change of the oscillator amplitude was observed due to the photochromic transition.

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11:15am EL-FrM-13 Non-Destructive Measurement Limitations of Cavity Etched Si/SiGe Layer Superlattice Structures Using MMSE Based OCD Metrology and X-Ray Fluorescence, *Ezra Pasikatan*, SUNY Albany CNSE; A. Antonelli, ONTO Innovation; N. Keller, Onto Innovation; M. Kuhn, Rigaku; S. Murakami, Rigaku, Japan; A. Diebold, SUNY Albany CNSE

Next generation node 3D semiconductor device structures require nondestructive metrology in order to measure key geometries for process control in high volume manufacturing. A key challenge is understanding the limitation of metrology for measuring Si/Si(1-x)Ge(x) superlattice structures, which are used in the manufacture of gate all around (GAA) transistors and future 3D DRAM memory. Nanowire test structures (NWTS) are used to measure the critical cavity etch step, where the SiGe layers in the superlattice are selectively etched to leave silicon nanowires or nanosheets.

A set of four superlattice layer NWTS were measured using Mueller matrix spectroscopic ellipsometry (MMSE) based optical critical dimension (OCD) metrology and X-ray fluorescence (XRF). Measurements were done on samples at superlattice film, anisotropic column etch, and two levels of cavity etch processing steps. X-ray diffraction (XRD) was used to determine superlattice film sample layer information for optical modeling. Limitations of MMSE based OCD modeling were explored based on contributions to measurement and model uncertainty, as well as measurability indicators. Also, matching of the non-destructive OCD and XRF based cavity etch

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measurements was evaluated based on a set of destructive focused ion beam (FIB) prepared transmission electron microscope (TEM) samples.

11:30am EL-FrM-14 Elevated Temperature Spectroscopic Ellipsometry Analysis of Bulk Single-Crystal In<sub>2</sub>O<sub>3</sub>, *Sema Guvenc Kilic*, *U. Kilic*, University of Nebraska-Lincoln; *M. Hilfiker*, Onto Innovation; *Z. Galazka*, Leibniz-Institute für Kristallzüchtung, Germany; *M. Schubert*, University of Nebraska-Lincoln

Transparent conductive oxides (TCOs) are materials that have a wide band gap in the ultraviolet region [1] and have been utilized in the optoelectronic industry, including solar cells, transistors, window heaters, transparent electrodes, and flat panel displays [2,3]. Among these, indium oxide ( $In_2O_3$ ) is prominent due to its pronounced electron mobility and large band gap values.

In this study, we employed an in-situ spectroscopic ellipsometry (a singlerotating compensator ellipsometer, M-2000, J. A. Woollam Co., Inc.) instrument attached to a high vacuum chamber that employs a heater stage. While the angle of incidence on the sample within the chamber is at 75°, the pressure is measured to be under 6.5  $\times$  10<sup>-5</sup> Torr. Hence, we performed the investigation of the dielectric function properties of meltgrown bulk  $In_2O_3$  single crystal [4] at elevated temperatures (22°C  $\leq$  T  $\leq$ 600°C). For each temperature value, Cauchy dispersion analysis was applied across the transparent spectrum to determine the high-frequency index of refraction. In addition, critical point model dielectric function analysis was performed to obtain the complex dielectric function and critical point transitions for selected temperature values. Ellipsometry measurements were conducted covering the spectral range from the near-infrared to the ultraviolet (300 nm to 1200 nm). Additionally, we present and discuss the room temperature wide spectral range (near-IR to vacuum ultraviolet) complex dielectric function of In2O3. Results indicate a pronounced temperature dependence of both the real and imaginary parts of the dielectric function, attributed to possible alterations in the electronic band structure and carrier concentration with temperature. These findings provide crucial insights into the thermal behavior of In<sub>2</sub>O<sub>3</sub>, aiding in the design and optimization of temperature-resilient optoelectronic devices.

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11:45am EL-FrM-15 Modeling Many-body Effects in Ge Using Pumpprobed Femtosecond Ellipsometry, *Carlos Armenta*, New Mexico State University; *M. Zahradník*, ELI ERIC, Czechia; *C. Emminger*, Leipzig University, Austria; *S. Espinoza*, ELI ERIC, Czechia; *M. Rebarz*, ERIC ELI, Poland; *S. Vazquez*, ELI ERIC, Mexico; *J. Andreasson*, ELI ERIC, Czechia; *S. Zollner*, New Mexico State University

This study investigates the transient dielectric function of germanium at very high electron-hole pair densities using time-resolved spectroscopic ellipsometry. By employing a pump-probe technique, we explore the evolution of the critical points  $E_1$  and  $E_1+\Delta_1$  near the L-valley as a function of delay time. We primarily focus on phase-filling singularities and manybody effects in different undoped germanium samples. Our aim is to model the behavior of the material under different carrier concentrations, analyze the impact these processes have on the material's optical properties, occurring at the temporal resolution on the order of femtoseconds.

The analysis includes modeling the dielectric function of germanium as carrier densities evolve throughout time. It addresses additional effects during electron excitation and relaxation, such as excitonic screening and acoustic phonon oscillations from energy transfer to the lattice. Experiments are conducted on bulk germanium samples oriented along various crystallographic planes. By pumping the sample with a high-power laser with 800 nm wavelength, carrier densities on the order of  $10^{20}$  cm<sup>-3</sup> were achieved. Delay times range from -10 ps to 1 ns with a 500 fs resolution. Our findings aim to enhance the understanding of germanium's optical behavior under intense laser excitation, providing insights into rapid

charge carrier dynamics and their influence on the material's electronic structure.

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