

## 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 an 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 interest for technical applications [4]. Due to 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transmission properties of an arbitrarily polarized wave at an interface by a superposition of the two eigenmodes is no longer applicable.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  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>.

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

[3]Adv. Photonics Res. **5**, 2300235 (2024).

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#### 8:45am EL-FrM-3 Infrared-Active Phonon Modes in Variably Alloyed Bulk $\beta$ -(Al<sub>x</sub>Ga<sub>1-x</sub>)<sub>2</sub>O<sub>3</sub> 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 pseudomorphically-strained epitaxial films with  $x = 0.046, 0.097,$  and  $0.163$  grown on beta-phase (010) Ga<sub>2</sub>O<sub>3</sub>. 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<sub>0.8</sub>CS<sub>0.2</sub>Pb(I<sub>0.6</sub>Br<sub>0.4</sub>)<sub>3</sub> 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) **INVITED**

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  $1.526 \pm 0.027$  nm and  $1.968 \pm 0.057$  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 monolayer 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

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  $\text{Ni}^{2+}$  and  $\text{O}^{2-}$  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  $8\text{ cm}^{-1}$  from  $250$  to  $8000\text{ cm}^{-1}$  on J. A. Woollam IR-VASE Mark II ellipsometer at  $70^\circ$  angle of incidence. Then, we cooled the NiO using liquid He and measured the ellipsometric angles  $\Psi$  and  $\Delta$  between  $25$  and  $500\text{ K}$  with a step size of  $25\text{ K}$  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  $\sim 400\text{ cm}^{-1}$  and another one for the two-phonon absorption (TO+TA) at  $\sim 560\text{ cm}^{-1}$ . 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  $64\text{ meV}$ , much larger than the TA(X) energy ( $16.5\text{ meV}$ ). This unexpected result is probably because of the scattering of phonons by two magnons with an energy of about  $190\text{ meV}$ .

**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 low-temperature 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 NLSL-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  $5\text{ cm}^{-1}$  and  $10,000\text{ cm}^{-1}$  with the synchrotron radiation as the light source. A wide range of angles of incidence AOI between  $70^\circ$  and  $85^\circ$  is enabled by the  $\theta$ - $2\theta$  goniometer. The ellipsometer has magnetic fields of up to  $\pm 7\text{ T}$  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  $4 \times 4$  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  $L = \pm 1, \pm 2, \pm 3,$  and  $\pm 4$  and spin angular momentum  $S = \pm 1$ , or conventional circular polarization, were used for studies of the magnon spectra in  $\text{TbFe}_3(\text{BO}_3)_4$ ,  $h\text{-Ni}_3\text{TeO}_6$ , and  $h\text{-Lu}_{0.6}\text{Sc}_{0.4}\text{FeO}_3$  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 broad 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, dipyrindinium thiazolo[5,4-d]thiazole viologen exhibits high-contrast, fast and reversible photochromic changes. When exposed to radiation with an energy larger than  $3.1\text{ eV}$ , it transitions from light yellow ( $\text{TTz}^{2+}$ ) to purple ( $\text{TTz}^{\bullet+}$ ) to blue ( $\text{TTz}^0$ ) 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  $405\text{ nm}$  diode laser in the infrared spectral range from  $500\text{ cm}^{-1}$  to  $1800\text{ cm}^{-1}$ . The model dielectric functions of the thiazolothiazole embedded polymer film for its  $\text{TTz}^{2+}$  (unirradiated) and  $\text{TTz}^0$  (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  $\text{TTz}^{2+}$  and  $\text{TTz}^0$  state shows that the oscillators located in the spectral ranges  $500\text{ cm}^{-1}$ - $700\text{ cm}^{-1}$ ,  $1300\text{ cm}^{-1}$ - $1400\text{ cm}^{-1}$ , and  $1500\text{ cm}^{-1}$ - $1700\text{ cm}^{-1}$  change in both amplitude and resonant frequency upon transition between the states. In addition, a resonance has been identified at approximately  $1050\text{ cm}^{-1}$ , for which, only a change of the oscillator amplitude was observed due to the photochromic transition.

## References

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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 non-destructive 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.

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

11:30am **EL-FrM-14 Elevated Temperature Spectroscopic Ellipsometry Analysis of Bulk Single-Crystal  $\text{In}_2\text{O}_3$** , *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 ( $\text{In}_2\text{O}_3$ ) is prominent due to its pronounced electron mobility and large band gap values.

In this study, we employed an in-situ spectroscopic ellipsometry (a single-rotating 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  $75^\circ$ , the pressure is measured to be under  $6.5 \times 10^{-5}$  Torr. Hence, we performed the investigation of the dielectric function properties of melt-grown bulk  $\text{In}_2\text{O}_3$  single crystal [4] at elevated temperatures ( $22^\circ\text{C} \leq T \leq 600^\circ\text{C}$ ). 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  $\text{In}_2\text{O}_3$ . 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  $\text{In}_2\text{O}_3$ , 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 Pump-probed 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 many-body 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  $10^{20} \text{ cm}^{-3}$  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

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