

## Spectroscopic Ellipsometry Focus Topic

### Room 9 - Session EL+AS+EM+TF-MoM

#### Application of SE for the Characterization of Thin Films and Nanostructures

**Moderator:** Tino Hofmann, University of North Carolina at Charlotte

8:20am **EL+AS+EM+TF-MoM-1 Ultra-thin Plasmonic Metal Nitrides: Optical Properties and Applications, Alexandra Boltasseva, Purdue University**

**INVITED**

Transition metal nitrides (e.g. TiN, ZrN) have emerged as promising plasmonic materials due to their refractory properties and good metallic properties in the visible and near infrared regions. Due to their high melting point, they may be suitable for high temperature nanophotonic applications. We have performed comprehensive studies of the temperature induced deviations to the dielectric function in TiN thin films. The studies were conducted on 30 nm, 50 nm, and 200 nm TiN films on sapphire substrates at temperatures up to 900 °C in the wavelength range 350-2000 nm using a custom built in-situ high temperature ellipsometry setup. The results were fitted with a Drude-Lorentz model consisting of one Drude oscillator and 2 Lorentz oscillators. As the temperature is elevated, the real and imaginary parts both begin to degrade. However, the deviations to the optical properties of TiN are significantly smaller compared to its noble metal counterparts, with no structural degradation in the TiN films. In addition to high temperature applications, TiN could also be a potential material platform for investigating light-matter interactions at the nanoscale, since high quality, continuous films of TiN can be grown on substrates such as MgO and c-sapphire down to just a few monolayers. Ultrathin TiN films with thicknesses of 2, 4, 6, 8, and 10 nm were grown on MgO using DC reactive magnetron sputtering, resulting in high quality films with low roughness. The changes in the linear optical properties were investigated using variable angle spectroscopic ellipsometry at angles of 50° and 70° for wavelengths from 400 nm to 2000 nm. A Drude-Lorentz model consisting of one Drude oscillator and one Lorentz oscillator was used to fit the measurements. As the thickness decreased, an increase in the losses and a decrease in the plasma frequency was observed. However, the films remained highly metallic even at 2nm, demonstrating that they could be used for nanophotonic applications, including nonlinear optical devices and actively tunable plasmonic devices.

9:00am **EL+AS+EM+TF-MoM-3 Magnetron Sputtering of TiN Coatings: Optical Monitoring of the Growth Process by Means of Spectroscopic Ellipsometry, Jiri Bulir, J More Chevalier, L Fekete, J Remiasova, M Vondracek, M Novotny, J Lancok, Institute of Physics ASCR, Czech Republic**

The plasmonic applications requires search for novel materials with metal-like optical properties and low optical losses. Transition metal nitrides such as TiN, TaN, ZrN, HfN, NbN exhibit metallic properties depending on concentration of free-carrier of charge. Their plasmonic properties can be tuned by deposition parameters controlling the film structure and the stoichiometry.

In this work, we deal with study of growth process of TiN films. The films are grown by RF magnetron sputtering on fused silica, silicon and MgO substrates at substrate temperature ranging from 20°C to 600°C. The growth process is monitored using in-situ spectral ellipsometer in spectral range from 245 to 1690 nm. The ellipsometric data, which are obtained during the deposition process, are attentively analysed using mathematical models based on Drude-Lorentz oscillators.

The Lorentz oscillators are used for description of interband transition in ultraviolet and visible spectral range, whereas the Drude oscillator describes the free-electron behavior in the infrared spectral range. We show that the free-electron behavior is affected by thickness of the ultrathin coatings due to electron scattering effects at the interfaces. Number of physical parameters such as free-electron concentration, Drude relaxation time and electrical conductivity is estimated at each stage of the deposition process by analysis of dielectric functions using the mentioned model. The resulting evolution of the electrotransport properties during the TiN film growth is presented. Special attention is devoted to the initial nucleation stage when the free-electron behaviour is significantly influenced by the interface between the substrate and the TiN film. Based on evolution of electrotransport properties, we discuss differences between polycrystalline growth of TiN film on Si and fused silica substrates and epitaxial growth on MgO substrates.

The accomplished TiN coatings are analyzed using infrared ellipsometer operating in spectral range from 1.7µm to 30µm where the optical constants are influenced most importantly by free-electron behaviour. The obtained results are compared with those obtained by the in-situ ellipsometer. Special attention is focused on scattering of free electrons at grain boundaries and at the TiN layer interfaces. The estimated parameters are correlated with structure changes such as grain coarsening and surface morphology. The crystallinity is analysed by X-ray Diffractometry. The surface morphology of the completed coatings is studied using Atomic Force Microscopy and Scanning Electron Microscopy. The TiN film stoichiometry is estimated by X-ray Photoemission Spectroscopy.

9:20am **EL+AS+EM+TF-MoM-4 Variable Temperatures Spectroscopic Ellipsometry Study of the Optical Properties of InAlN/GaN Grown on Sapphire, Y Liang, Guangxi University, China; H Gu, Huazhong University of Science and Technology, China; J Xue, Xidian University, China; Chuanwei Zhang, Huazhong University of Science and Technology, China; Q Li, Guangxi University, China; Y Hao, Xidian University, China; S Liu, Huazhong University of Science and Technology, China; Q Yang, L Wan, Z Feng, Guangxi University, China**

Indium aluminum nitride (InAlN), a prospective material for lattice matched confinement layer, possesses the potential to improve the reliability and performance of high electron mobility transistors (HEMTs).<sup>[1]</sup> One of the important advantages of InAlN alloy is the possibility of growing in-plane lattice-matched to GaN for an indium content of around 17%. However, the bandgap we expected is hindered by the growth of high-quality InAlN films due to the phase separation and nonuniform composition distribution.<sup>[1-2]</sup>

In this work, InAlN/GaN heterostructures, grown by pulsed metal organic chemical vapor deposition (PMOCVD) on c-plane sapphire substrates, were investigated by a dual rotating-compensator Mueller matrix ellipsometer (ME-L ellipsometer, Wuhan Eoptics Technology Co. Ltd., China). The experimental data ( $\Psi$  and  $\Delta$ ), covering the wavelength ( $\lambda$ ) range from 193 nm up to 1700 nm at 1 nm step or energy (E) from 0.73 eV to 6.43 eV, were obtained by variable temperatures spectroscopic ellipsometric (VTSE) in three angles (50°, 55° and 60°). The Eoptics software was utilized to fit VTSE data using Tauc-Lorentz multiple oscillator modes. By analyzing the fitting results, the optical constants of the InAlN at variable temperatures (25°C-600°C) were obtained. The peak value of the refractive index increases from 269 nm to 284 nm with increasing temperature. The bandgaps are 4.57 eV and 4.35 eV at the temperature 25°C and 600°C, respectively. These results demonstrated that InAlN/GaN has a high thermal stability, scilicet no significant performance degradation in high temperature environment.

#### Reference

[1] Wenyuan Jiao, Wei Kong, Jincheng Li et al, Characterization of MBE-grown InAlN/GaN heterostructures valence band offsets with varying In composition, AIP ADVANCES 6, 035211 (2016).

[2] JunShuai Xue, JinCheng Zhang, Yue Hao, Investigation of TMIn pulse duration effect on the properties of InAlN/GaN heterostructures grown on sapphire by pulsed metal organic chemical vapor deposition, Journal of Crystal Growth 401, 661 (2014).

9:40am **EL+AS+EM+TF-MoM-5 Optical Properties of Cs<sub>2</sub>AgIn<sub>(1-x)</sub>Bi<sub>x</sub>Cl<sub>6</sub> Double Perovskite Studied by Spectroscopic Ellipsometry, Honggang Gu, S Li, B Song, J Tang, S Liu, Huazhong University of Science and Technology, China**

During the past several years, the organic-inorganic lead halide perovskites (APbX<sub>3</sub>, A = CH<sub>3</sub>NH<sub>3</sub> or NH<sub>2</sub>CHNH<sub>2</sub>, X = Cl, Br, or I) have been promising materials for photovoltaic, photoelectric -detecting and light-emitting devices due to their outstanding photoelectric properties, such as broad absorption range, high quantum efficiency, ultrafast charge generation, high charge carrier mobility and long charge carrier lifetime and diffusion length. However, there are two remaining challenges that need to be addressed in order to apply these materials to photoelectric productions, namely the compound stability and the presence of lead. Most recently, lead-free metal halide double perovskites, such as Cs<sub>2</sub>AgBiCl<sub>6</sub> and Cs<sub>2</sub>AgInCl<sub>6</sub>, have attracted extensive attention because of their nontoxicity and relative air-stability. In the study and application of these perovskite materials, the knowledge of their optical properties, such as the bandgap and the basic optical constants, is of great importance to predict the photoelectric characteristics and dig the potential of the materials.

Spectroscopic ellipsometry (SE) has been developed as a powerful tool to characterize the optical properties as well as structure parameters of novel materials, thin films and nanostructures. In this work, we study the optical properties of Cs<sub>2</sub>AgIn<sub>(1-x)</sub>Bi<sub>x</sub>Cl<sub>6</sub> perovskites by a spectroscopic ellipsometer

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(ME-L ellipsometer, Wuhan Eoptics Technology Co., Wuhan, China). The refractive index and the extinction coefficient of  $\text{Cs}_2\text{AgIn}_{(1-x)}\text{Bi}_x\text{Cl}_6$  with different composition coefficient  $x$  of bismuth are determined by the ellipsometer over the wavelength range of 250-1000nm. We find that the presence of bismuth introduces two critical points in the optical constant spectra of the perovskites, i.e., 315nm and 382nm in the refractive index spectra and 300nm and 375nm in the extinction coefficient spectra, respectively. Moreover, there is a red shift in the bandgaps and significant increase in both the refractive index and the extinction coefficient with the increase of composition coefficient  $x$  of bismuth.

10:00am **EL+AS+EM+TF-MoM-6 Charge Carrier Dynamics of Aluminum-doped Zinc Oxide Deposited by Spatial Atomic Layer Deposition**, *Daniel Fullager, G Boreman, T Hofmann*, University of North Carolina at Charlotte; *C Ellinger*, Eastman Kodak Company

Transparent conductors for displays, backplanes, touchscreens and other electronic devices are an area of active research and development; in this manner, aluminum-doped zinc oxide (AZO) has shown promise as an ITO replacement for some applications. Although there have been numerous reports on the optical properties and electrical conductivity of AZO, there has not yet been a Kramers-Kronig consistent dispersion model fully describing the charge carrier dynamics. In this presentation, we will report on the model dielectric function of AZO from the combination of UV-Vis and IR spectroscopic ellipsometry. A model dielectric function that describes the optical response over this wide spectral range will be presented and discussed. In particular, we will present a comparison between the commonly used extended Drude models and the dielectric function developed here in light of results obtained from density functional theory calculations.

The AZO films analyzed in this study were deposited using a spatial atomic layer deposition (SALD) process. While AZO can be deposited by several techniques, including sputtering, chemical vapor deposition (CVD), and atomic layer deposition (ALD), ALD does allow for the greatest ability to control the aluminum-doping level of AZO. However, the range of substrate sizes and form factors addressable by traditional chamber ALD are limited. Conversely, spatial ALD (SALD) is an atmospheric pressure, roll-compatible ALD process that enables the materials property control of ALD to be translated into a wider range of applications spaces. Furthermore, the use of selective area deposition in a "patterned-by-printing" approach enables the high-quality AZO deposited by SALD to be easily patterned, offering an integrated and facile path for manufacturing optical and electronic devices.

10:40am **EL+AS+EM+TF-MoM-8 Broad Range Ellipsometry Shining Light onto Multiphase Plasmonic Nanoparticles Synthesis, Properties and Functionality**, *Maria Losurdo*, CNR-NANOTEC, Italy **INVITED**

How rich are the physics, interface chemistry and optical properties associated with the surface plasmons of metal nanostructures and their potential for manipulating light at the nanoscale! For many technological applications nanoparticles (NPs) are supported on a substrate, and at the nanoscale, interaction and interfaces with the support become very important. We have demonstrated that the substrate/NPs interaction is the key to engineering not only the shape but also the crystalline phase of NPs.

This contribution will present and explore fundamental and applied aspects of multiphase core-shell plasmonic NPs supported on substrates of technological interest using various diagnostic tools, which comprise: (i) spectroscopic ellipsometry spanning the THz, IR, visible, and UV wavelength ranges, (ii) variable angle Muller Matrix ellipsometry to qualify size effects on anisotropy and depolarization of samples, (iii) *in-situ real-time* spectroscopic ellipsometry to understand growth and tailor particle size which ultimately controls the plasmon resonance, and (iv) various imaging and microscopy techniques to elucidate the interplay between the nanostructure of multiphase nanoparticle and their functionality.

The case studies involve liquid-shell/solid-core plasmonic NPs (Ga, Ga/Mg), plasmon-catalytic core/shell Ga/Pd and plasmon-magnetic Ni/Ga NPs supported on various substrates (glass, plastics, sapphire) that control their crystalline phases.

We will start with a description of the *real-time* ellipsometry capabilities in monitoring the growth of those multiphase core/shell NPs to detect the formation of the various phases in situ and to control the resulting plasmon resonance.

The discussion then will shift to a description of fundamental thermodynamics of substrate supported multiphase NPs and how their

growth dynamics is controlled by the interface energies, and how those new phenomena can be highlighted by real-time ellipsometry.

Ex-situ corroborating measurements of Mueller-matrix ellipsometry and hyperspectral cathodoluminescence spectroscopy and imaging will be presented to discuss phenomena of depolarization and of interaction of NPs resulting from the self-assembly.

Finally, since those NPs enable active plasmonics, we demonstrate the implications of the multi-phase nature of NPs, as well as solid-liquid phase coexistence on the plasmon resonance (LSPR) of supported NPs and on its exploitation to follow in real time phenomena in their application in catalysis (hydrogen storage and sensing) and optomagnetism and possible future directions.

*The contribution of the H2020 European programme under the project TWINFUSYON (GA692034) is acknowledged*

11:20am **EL+AS+EM+TF-MoM-10 Use of Evolutionary Algorithms for Ellipsometry Model Development and Validation using Eureka**, *Neil Murphy*, Air Force Research Laboratory; *L Sun*, General Dynamics Information Technology; *J Jones*, Air Force Research Laboratory; *J Grant*, Azimuth Corporation

Eureka, developed by Nutonian Inc., is a proprietary modeling engine based on automated evolutionary algorithms. In this study, we utilized Eureka to parameterize both the amplitude and phase difference data for reactively sputtered thin films. Specifically, evolutionary algorithms are used to develop and validate models for fitting raw ellipsometric data for a variety of optical materials including  $\text{SiO}_2$ ,  $\text{Ta}_2\text{O}_5$ , and Aluminum Zinc Oxide. These films, deposited using pulsed DC magnetron sputtering, were deposited on both silicon and fused quartz substrates, and measured using a J.A. Woollam VASE system. The resulting models are then compared to traditional models that are currently utilized to fit the candidate materials systems.

11:40am **EL+AS+EM+TF-MoM-11 Excitonic Effects on the Optical Properties of Thin ZnO Films on Different Substrates**, *Nuwanjula Samarasingha, Z Yoder, S Zollner*, New Mexico State University; *D Pal, A Mathur, A Singh, R Singh, S Chattopadhyay*, Indian Institute of Technology Indore, India

The presence of excitonic features in the optical constants of bulk semiconductors and insulators has been known for many years. In Si, Ge, and GaAs, the  $E_1$  critical points are strongly enhanced by two-dimensional excitons. Three-dimensional excitons have been seen in ellipsometry spectra for GaP and Ge. In addition to these semiconductors, wide band gap materials like ZnO exhibit strong excitonic features in the dielectric function ( $\epsilon$ ) which is directly related to the electronic band structure. The top valence band at the  $\Gamma$  point in the Brillouin zone is split into three bands by spin orbit and crystal field splitting. The corresponding free exciton transitions between the lowest conduction band and these three valence bands are denoted by A, C ( $\Gamma_7$  symmetry) and B ( $\Gamma_9$  symmetry). The transition from the B subband is forbidden for light polarized parallel to the optical axis (extraordinary dielectric function). ZnO is attractive for optoelectronic device applications due to its large excitonic binding energy of 60 meV at room temperature. The influence of this excitonic absorption on  $\epsilon$  was described by Tanguy [1].

Here we investigate the behavior of excitons in c-oriented ZnO thin films grown on Si (smaller band gap than ZnO) and  $\text{SiO}_2$  (larger band gap than ZnO) substrates using variable angle spectroscopic ellipsometry and FTIR ellipsometry. We also performed X-ray diffraction (XRD), X-ray reflectivity (XRR), and atomic force microscopy (AFM) to characterize the structural properties of our ZnO films.

In a thin epitaxial layer on a substrate with a different band gap, the wave functions of the electron and hole are strongly modified. In ZnO (band gap 3.37 eV) grown on a large-gap  $\text{SiO}_2$  substrate (type-I quantum well), both the electron and the hole are confined, which leads to an increase in the dipole overlap matrix element. Therefore, the real and imaginary part of  $\epsilon$  of thin ZnO layers on  $\text{SiO}_2$  are much larger than in the bulk and increase monotonically with decreasing thickness.

On the other hand, in a staggered type-II quantum well (ZnO on Si), either the electron is confined, or the hole, but not both. Therefore, the overlap dipole matrix element is strongly reduced. Therefore,  $\epsilon$  of thin ZnO layers on Si is much smaller than in the bulk and decreases monotonically with decreasing thickness. We will fit our ellipsometric spectra by describing the dielectric function of ZnO using the Tanguy model [1]. We will analyze the dependence of the excitonic Tanguy parameters on quantum well thickness and substrate material.

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Reference:

[1] C. Tanguy, Phys. Rev. Lett. **75**, 4090 (1995).

## Spectroscopic Ellipsometry Focus Topic

### Room 9 - Session EL+AS+EM-MoA

#### Spectroscopic Ellipsometry: Novel Applications and Theoretical Approaches

**Moderator:** Maria Losurdo, University Bari

##### 1:40pm EL+AS+EM-MoA-1 Temperature Dependent Mueller Matrix Measurements of Magnetised Ni near the Curie Temperature, *Farzin Abadizaman, S Zollner*, New Mexico State University

The temperature dependence of the optical constants of the magnetized bulk Ni demonstrates an anomaly near the Curie temperature. We investigate this anomaly by taking a measurement of the temperature dependent Mueller Matrix (MM). Using spectroscopic ellipsometry at an energy 1.96 eV, the MM measurement was taken from 350 K to 500 K with 10 K steps, and from 500 K to 670 K with 1 K steps, and from 670 K to 730 K with 10 K steps.

In order to distinguish the anisotropic properties of the magnetized Ni from the windows effects, three samples (Ge, Ni, and SiO<sub>2</sub> on Si) were measured inside the cryostat in the energy range from 1 eV to 5 eV with 0.1 eV steps. The results show that the anisotropic elements of the MM behave in the same fashion for all samples, except for element M<sub>24</sub>, which depends on the sample.

The MM data of magnetized Ni indicate very small changes in the anisotropic portion of the MM compared to the windows effect. The authors believe that these changes are due to the magneto-optical Kerr effect. However, since a rotating-analyzer ellipsometer was used, the last row of the MM is absent and a complete MM measurement needs to be performed to find the magneto-optical Kerr effect in the other anisotropic elements too.

However, very large decreases in the isotropic MM elements were found near the Curie temperature. This means, the changes in optical constants near T<sub>c</sub> are due to the on-diagonal Drude part of the dielectric tensor, which can be explained by s- to d-band electron-phonon scattering above and below T<sub>c</sub>. These changes are absent when decreasing the temperature and for unmagnetized Ni.

##### 2:00pm EL+AS+EM-MoA-2 Ellipsometry Based Observation of Material Ordering Process in Holography, *Hao Jiang, H Peng, Y Liao, S Liu*, Huazhong University of Science and Technology, China

Ordered materials with superior performance have been constantly pursued. Nevertheless, the direct, precise and non-destructive observation of the ordering process, which is especially critical for continuous manufacturing, remains a formidable challenge. Herein, we introduce Mueller matrix ellipsometry (MME) as a nondestructive method to quantitatively observe the material ordering process during holography. This non-destructive observation directly offers the exact width, refractive index, nanoparticle weight fraction and volume fraction in each constructive (bright) or destructive (dark) interference area, which is impossible to be implemented using other existing techniques. Meanwhile, the width of dark region is observed to decrease while the width of bright region increases with an augmentation of the holographic recording time, distinct from previous assumption. More importantly, an apparent diffusion coefficient of  $1.3 \times 10^{-15} \text{ m}^2 \text{ s}^{-1}$  is determined on the basis of recording time-dependent grating structure observation, which is 3 orders of magnitude lower than the Stokes-Einstein prediction. The distinct diffusion coefficient is regarded to be the result of competition between the forth diffusion driven by the chemical reaction and backward diffusion arising from nanoparticle concentration gradient. This novel protocol is envisioned to pave the way for precisely and non-destructively understanding the ordered structure formation in electronics, photonics, photovoltaics, biomaterials and other disciplines.

##### 2:20pm EL+AS+EM-MoA-3 Coherence in Polarimetry and Ellipsometry: Synthesizing Mueller Matrices in an Ellipsometer, *Oriol Arteaga*, Departament de Física Aplicada, Universitat de Barcelona, Spain **INVITED**

Interference phenomena are ubiquitous in optics and are the basis of the industry behind thin film optical coatings or thin film thickness measurements with ellipsometry. The interference of polarized beams was experimentally understood in 1819 when Arago and Fresnel derived the four laws governing the interference of polarized light. While spectroscopic ellipsometry provides information based on the position and number of

interference oscillations in thin films, typically one has no macroscopic control of the beams that coherently superpose.

In this work we present a polarimetric analysis of an analogue of Young's double slit experiment that allows merging beams in a well-controlled manner. The experiment is analyzed with a new formalism that is useful to describe optical coherence and polarization and that shows that the superposition of two macroscopically distinguishable beams can be an effective method to experimentally synthesize Mueller matrices with on-demand polarization properties. This offers the opportunity of working with "synthesized" optical elements that behave just like "real" ones. We will discuss how this method can have a practical application in the construction of ellipsometers or polarimeters.

##### 3:00pm EL+AS+EM-MoA-5 Femtosecond Spectroscopic Ellipsometry on Optoelectronic Materials and Photonic Structures, *Mateusz Rebarz, S Espinoza*, ELI Beamlines - Czech Academy of Science, Czech Republic; *S Richter, O Herrfurth, R Schmidt-Grund*, Universität Leipzig, Germany; *J Andreasson*, Chalmers University of Technology, Sweden; *S Zollner*, New Mexico State University

The ongoing progress in miniaturization and operational rates of electronic and optoelectronic devices obliges materials scientists to deeply understand the dynamics of the carriers upon external electromagnetic stimulus in very short time scale. Some phenomena such as scattering of electrons and phonons as well as recombination processes can be as short as tens of femtoseconds. All these processes affect the temporal and local dielectric constants and determine many operational parameters of the devices. In this work, we report on recent progress in developing a spectroscopic ellipsometer for characterization of ultrafast dynamic changes of dielectric properties in materials and photonic structures technologically relevant in optoelectronics. Our time-resolved ellipsometer is based on pump-probe technique and offers monitoring the time evolution of the dielectric properties in range 0-5 ns with time resolution ~100 fs in broadband spectral range (340-750 nm).

We present here the results of the measurements performed on Ge samples in comparison with theoretical predictions. Ultrafast phenomena such as excitation, relaxation and diffusion of charge carriers, band-gap renormalization and excitons screening will be discussed. In addition we report on the first data obtained from a ZnO-based planar microcavity especially on the temporal evolution of the microcavity modes. The time evolution of the ellipsometric parameters in the spectral range around the exciton-polariton mode will be discussed. We observed that such modes disappear upon the excitation, possibly due to screening of the excitons and re-appear after a few hundreds of femtoseconds as blueshifted modes. The investigation of the short-time dynamics of such modes can stimulate new theoretical approaches for the description of exciton-polariton systems.

##### 3:20pm EL+AS+EM-MoA-6 Temperature Dependence of the Dielectric Function and Interband Critical Points of Bulk Germanium, *Carola Emminger, N Samarasingha, F Abadizaman, N Fernando, S Zollner*, New Mexico State University

Exploration of the optical properties of bulk germanium (Ge) is necessary for the advancement of Ge technology. In fact, many of the Ge applications depend on the dielectric function ( $\epsilon$ ), which is directly related to the electronic band structure. Here we investigate the effect of temperature on the optical properties and interband critical points (CPs), primarily the E<sub>0</sub> and E<sub>0</sub>+ $\Delta_0$  critical points of bulk Ge in the temperature range from 10 to 738 K using spectroscopic ellipsometry at 70° angle of incidence. The data was taken in two parts, in the near IR region and in the UV region, which provides data from 0.5 to 6.2 eV. The low temperature environment was created in a UHV cryostat with liquid helium and nitrogen as cryogenes. To reduce the thickness of the native GeO<sub>2</sub> layer, the Ge sample was cleaned using ultra-pure water, isopropanol, an ultrasonic bath and ozone cleaning. It was possible to reduce the oxide thickness to about 11 Å at room temperature.

The authors used a two-phase model (GeO<sub>2</sub> layer/Ge substrate) and a parametric oscillator model with a set of adjustable parameters to extract the real and imaginary parts of the complex dielectric function of the bulk Ge for the whole temperature range. To investigate this temperature dependence of the CP parameters (threshold energy, broadening and phase angle) further, we also compared the second derivative  $d^2\epsilon/d^2\omega$  of the dielectric function with analytical line shapes.

The temperature has a significant influence on both the real and imaginary parts of the complex dielectric function of bulk Ge. This temperature dependent  $\epsilon$  can be explained by a Bose-Einstein occupation factor. Due to

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the electron-phonon interaction, we find a temperature dependent red shift (shift to lower energies) of the  $E_0$  and  $E_0+\Delta_0$  critical point energies. The temperature independent spin orbit splitting  $\Delta_0$  is found to be 286 meV. A similar effect has been seen in the  $E_1$ ,  $E_1+\Delta_1$ ,  $E_0'$ , and  $E_2$  CP energies. These CPs are broadened and shifted to the lower energies with increasing temperature.

4:00pm **EL+AS+EM-MoA-8 VUV Magneto-Optical Transient Ellipsometer, Shirly Espinoza, J Andreasson**, Institute of Physics ASCR, Czech Republic

This talk is about the ELIps instrument, an instrument that combines three advanced techniques of ellipsometry: VUV ellipsometry, Transient (Pump-probe) ellipsometry, and Magneto-optical ellipsometry [1].

The ELIps instrument allows users to work in a very wide range of energies from 1 eV to 40 eV. For measurements in the 1 eV to 6.5 eV range, it uses a transmission polarizer and analyzer combination. Meanwhile, for measurements in the 6.5–40 eV range, it uses a triple-reflection polarizer and analyzer combination. All the components are contained within a single UHV chamber designed with several additional ports to support future upgrades. For time resolved measurements in the VUV range the instrument will be used together with a high intensity High Harmonics Generation (HHG) source.

Time-resolved transient measurements of the optical properties of materials can be performed in the range of a few femtoseconds to nanoseconds. Initial experiments with transient absorption and transient ellipsometry in the NIR-UV range is being performed on proton-conducting materials for solid oxide fuel cell applications.

Additionally, ELIps is equipped with a Helmholtz coil, which allows the studies of the transverse magneto-optical Kerr effect on the sample, and with a cryostat for measurements at different temperatures.

Where is this instrument located? ELIps is in Prague, Czech Republic, at the European Extreme Light Infrastructure Beamlines (ELI Beamlines), a user facility project that will hold some of the most intense lasers in the world. It will open to the public on January 2018.

## References

[1] S. Espinoza, G. Neuber, C. D. Brooks, B. Besner, M. Hashemi, M. Rübhausen and J. Andreasson. 2017. User oriented end-station on VUV pump-probe magneto-optical ellipsometry at ELI beamlines. *Applied Surface Science*. Published online. doi:10.1016/j.apsusc.2017.02.005

## Acknowledgements

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4:20pm **EL+AS+EM-MoA-9 Infrared Ellipsometric Spectroscopy of  $Hg_{1-x}Cd_xTe$  Bulk Samples, Yanqing Gao**, Shanghai Institute of Technical Physics, Chinese Academy of Sciences, China

**The infrared spectroscopic ellipsometry of  $Hg_{1-x}Cd_xTe$  ( $x=0.195\sim 0.37$ ) bulk samples with different compositions were measured. The refractive index  $n$  and extinction coefficient  $k$  were obtained. An obvious refractive index enhancement effect was observed in the refractive index spectra for each composition. The energy position of the maximal refractive index value equals approximately that of the band gap. With the decrease of the component, the refractive index increased and the peak position shifted to the low energy direction, which consisted with the absorption edge. The refractive index  $n$  at  $E_g$  changed linearly with the composition  $x$ .**

4:40pm **EL+AS+EM-MoA-10 Infrared Ellipsometry Study of the Photo-generated Charge Carriers at the (001) and (110) Surfaces of  $SrTiO_3$  Crystals and the Interface of Corresponding  $LaAlO_3/SrTiO_3$  Heterostructures, Meghdad Yazdi-Rizi, P Marsik, B Mallett**, University of Fribourg, Switzerland; *K Sen, A Cerreta*, University of Fribourg; *A Dubroka*, Masaryk University; *M Scigaj, F Sánchez, G Herranz*, Institut de Ciència de Materials de Barcelona; *C Bernhard*, University of Fribourg, Switzerland

With infrared (IR) ellipsometry and DC resistance measurements we investigated the photo-doping at the (001) and (110) surfaces of  $SrTiO_3$  (STO) single crystals and at the corresponding interfaces of  $LaAlO_3/SrTiO_3$  (LAO/STO) heterostructures. In the bare STO crystals we find that the photo-generated charge carriers, which accumulate near the (001) surface, have a similar depth profile and sheet carrier concentration as the confined electrons that were previously observed in LAO/STO (001) heterostructures. A large fraction of these photo-generated charge carriers persist at low temperature at the STO (001) surface even after the UV light

has been switched off again. These persistent charge carriers seem to originate from oxygen vacancies that are trapped at the structural domain boundaries which develop below the so-called antiferrodistortive transition at  $T^* = 105$  K. This is most evident from a corresponding photo-doping study of the DC transport in STO (110) crystals for which the concentration of these domain boundaries can be modified by applying a weak uniaxial stress. The oxygen vacancies and their trapping by defects are also the source of the electrons that are confined to the interface of LAO/STO (110) heterostructures which likely do not have a polar discontinuity as in LAO/STO (001). In the former, the trapping and clustering of the oxygen vacancies also has a strong influence on the anisotropy of the charge carrier mobility. We show that this anisotropy can be readily varied and even inverted by various means, such as a gentle thermal treatment, UV irradiation, or even a weak uniaxial stress. Our experiments suggest that extended defects, which develop over long time periods (of weeks to months), can strongly influence the response of the confined charge carriers at the LAO/STO (110) interface.

# Tuesday Evening Poster Sessions, October 31, 2017

## Spectroscopic Ellipsometry Focus Topic

### Room Central Hall - Session EL-TuP

#### Spectroscopic Ellipsometry Poster Session

##### EL-TuP-1 Ultra High-speed Spectroscopic Ellipsometry and its Applications, *Gai Chin*, ULVAC, Japan

As a comprehensive manufacturer of metrology tools and deposition tools, ULVAC developed an innovative ultra high-speed spectroscopic ellipsometer for many deposition applications, such as PVD, CVD, ALD and others.

This novel spectroscopic ellipsometry can measure the thickness and optical constants of thin films at a dramatically fast speed. Its data acquisition time is as short as 10 ms. It does not require any active components for polarization-control, such as a rotating compensator or an electro-optical modulator.

It created great opportunities for new applications of the spectroscopic ellipsometry in which the compactness, the simplicity and the rapid response are extremely important. It can be integrated into the deposition tool and successfully measured thin films in-situ and ex-situ. Obviously, PVD, CVD and ALD are some promising applications for this novel spectroscopic ellipsometry.

This paper describes the principle, system configuration and our creative efforts on developing a series of ultra high-speed spectroscopic ellipsometers. Some of the novel applications will be also introduced, such as the PVD, CVD, ALD, EUV, OLED, MEMS and some measurement data of thin films from the semiconductor, flat panel display and other industries.

##### EL-TuP-2 Comparing and Evaluating the Calculation Results of Measurement Uncertainty for Various Types of Rotating-element Spectroscopic Ellipsometers, *Yongjai Cho*, *W Chegal*, *H Cho*, Korea Research Institute of Standards and Science, Republic of Korea

Various multi-channel rotating-element spectroscopic ellipsometers have excellent measurement abilities like real-time, high-precision, non-destructive, and contactless schemes, and as a result have been widely used in a semiconductor manufacturing process. With the development of semiconductor device process technologies, the thickness of the thin film used for these technologies is getting smaller and smaller and thus reaches a level of atomic layer and the shape of the nano pattern is changed from a two-dimensional structure into a three-dimensional structure and thus is becoming increasingly complicated. Therefore, for the rotating-element spectroscopic ellipsometers to be continuously used as a measurement tool for next-generation semiconductor industries, it is important to continuously enhance their measurement uncertainties. Recently, we developed the universal evaluations and expressions of measuring uncertainty for all types of rotating-element spectroscopic ellipsometers. We also introduced a general data-reduction process to represent the universal analytic functions of the combined standard uncertainties of the ellipsometric sample parameters. To solve the incompleteness of the analytic expressions, we formulated the estimated covariance for the Fourier coefficient means extracted from the radiant flux waveform using a new Fourier analysis. Our approach can be used for providing a method for calculating a theoretical model equation which may be applied to various kinds of multi-channel rotating-element spectroscopic ellipsometers and may determine a measurement confidence level thereof, that is, a theoretical equation on standard uncertainties of ellipsometric parameters determined on the basis of a series of observations for a sample. In this presentation, it will show that the calculation data of the combined standard uncertainty for the various types of the rotating-element spectroscopic ellipsometers are obtained using the universal expressions for the combined standard uncertainty. In particular, the calculation results for the dual-rotating-compensator spectroscopic ellipsometers will be compared and evaluated with the calculation results for the common single-rotating-element spectroscopic ellipsometers.

##### EL-TuP-3 Ellipsometry Analysis of a Germanium-on-insulator Wafer, *Rigo Carrasco*, *N Samarasingha Archichchege*, New Mexico State University; *B Nguyen*, Soitec, France; *S Zollner*, New Mexico State University

Germanium based photonic devices attract a lot of interest due to the fact that its band structure is easily influenced by strain and alloying with tin. A direct bandgap group IV semiconductor will show an improvement in efficiency in optoelectronic devices. Utilizing a germanium-on-insulator (GOI) substrate is a key feature for future silicon compatible germanium

based devices, allowing for easier integration by the microelectronics industry. [1]

Here, we analyzed the optical response of a GOI bonded wafer via spectroscopic ellipsometry. The ellipsometric angles,  $\psi$  and  $\Delta$ , and the depolarization were acquired from 0.5 to 6 eV in 0.01 eV increments using a J.A. Woollam variable angle spectroscopic ellipsometer (VASE) and from 0.1 to 0.8 eV using a Fourier transform infrared (FTIR) ellipsometer, allowing high precision measurements in the mid-infrared range. The measurements in both ranges were performed at angles of incidence from 60 to 75 degrees in 5 degree increments.

The optical response of the GOI wafer was modeled with four layers (Si, buried oxide, Ge, native oxide). As expected, strong interference oscillations were observed below the E1 critical point of Ge. For Ge, we used the parametric semiconductor oscillator model and compared it to our previously determined optical constants of bulk germanium. We are particularly interested in differences near the direct band gap. The infrared phonon vibrations of the buried oxide were also visible in the measurements. The same measurement procedure was performed on the GOI wafer before and after cleaning the sample to observe the optical effects of a native surface oxide. While the pseudo-dielectric function of the GOI sample appeared different from that of a bulk Ge sample due to interference effects, the optical constants of the germanium layer only showed small differences. We did not find any differences in the electronic structure of bulk Ge and a thin bonded Ge layer.

References:

[1] B.Y. Nguyen, M. Sadaka, G. Gaudin, W. Schwarzenbach, K. Boudele, C. Figuet, C. Maleville

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