Spectroscopic Ellipsometry Focus Topic
Room: 112 - Session EL+EM+EN-ThM

Spectroscopic Ellipsometry: Novel Applications and Theoretical Approaches
Moderator: Tino Hofmann, University of Nebraska - Lincoln, Vimal Kamineni, Globalfoundries, Ny, Usa

8:00am **EL+EM+EN-ThM1 Multi-Spectral Polarimetric Imaging and Biomedical Applications**, ***Bernard Drevillon***, *A. Pierangelo*, LPICM-CNRS, Ecole Polytechnique, France **INVITED**

In the last years Polarimetric Imaging has received considerable attention in the literature thanks to its tremendous potential for the assessment of biological tissues in biomedical diagnostics. Light Polarization allows obtaining morphological information on tissues microscopic structure, potentially improving the diagnosis and treatment of several pathologies. Moreover, polarimetric imaging can be implemented using conventional light sources, like LED or halogen lamps, making it a cheap alternative to current standards. For several years the PICM Laboratory has designed and built innovative polarimetric imagers for biomedical applications. In particular, the development of the *eigenvalue calibration method* [1], led to the design of several polarimeters for macroscopic and microscopic analysis (in real and Fourier space) of *ex vivo* samples and for *in vivo* diagnoses. The development of such new instruments ranged from the simple measurement of polarization degree to the complete Mueller polarimetry. Several studies were devoted to the early detection and staging of uterine cervix cancer and to show that polarimetric imaging is effective for the visualisation and first grading of cervical dysplastic regions for patients with anomalous Pap smear [2]. Mueller matrix imaging polarimetry also provides enhanced contrast to differentiate types of cancer of colon and their stage of progress and penetration, which is currently detectable only by histological examination [3]. Moreover, this technique may also be useful to quickly verify the presence of residual cancer in the rectum after treatment with radiochemotherapy [4]. Finally, as a complementary development to experimental techniques, the set-up of Monte-Carlo detailed modelling of polarized light scattering in tissues has been carried out in the last few years and provides fundamental insight on the origin of observed polarimetric contrasts [5]. In conclusion the synergy of new experimental techniques based on polarimetry with the biomedical analysis and theoretical computer models, led to significant advances in the field of biological tissues characterization and diagnosis of related pathologies.

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8:40am **EL+EM+EN-ThM3 Anisotropic Optical Properties of Rhombohedral and Tetragonal $BiFeO_3$ Phases**, ***Daniel Schmidt***, National University of Singapore, *L. You,* Nanyang Technological University, Singapore, *X. Chi,* National University of Singapore, *J. Wang,* Nanyang Technological University, Singapore, *A. Rusydi*, National University of Singapore

Single crystalline bismuth ferrite ($BiFeO_3$) is a multiferroic perovskite structure and exhibits magnetic as well as strong ferroelectric behaviour at room-temperature. Since about a decade $BiFeO_3$ is of strong research interest due to its potential applicability in ferroelectric memory devices and spintronics, for example [1].

While the lattice system of bulk $BiFeO_3$ is rhombohedral, the crystal structure of thin films can be engineered by introducing epitaxial strain. Depending on the choice of single crystalline substrate materials the thin film $BiFeO_3$ crystal structure and associated physical properties can be modified.

Here, we present the anisotropic optical properties of high-quality multiferroic $BiFeO_3$ thin films determined with Mueller matrix ellipsometry at room-temperature within the spectral range of 0.6 and 6.5 eV. The full dielectric function tensors of tetragonal-like and rhombohedral-like $BiFeO_3$ phases epitaxially grown on $LaAlO_3$ and $SrTiO_3$ single crystal substrates, respectively, are discussed. Significant birefringence and dichroism are observed as well as strain-induced differences in critical point energies between both phases.

The importance of careful optical analysis of anisotropic Mueller matrix data will be discussed, which allows for characterization of subtle sub-band gap crystal field transitions and reveals indications of an indirect band gap. Such transitions have been observed before by means of other techniques but not by ellipsometry. Additionally, the analysis of Mueller matrix data revealed that an unintentional substrate miscut can introduce an overall polarization tilt of the ferroelectric thin films. This tilt was confirmed by extensive in- and out-of-plane piezoelectric force microscopy studies.

An accurate determination of the dielectric function tensor is of high importance to verify or, if necessary, improve and correct ab-inito calculations, which are crucial for understanding the driving physical principles in such complex materials. A comparison of the experimental results with state-of-the art first-principle calculations will be presented.

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9:00am **EL+EM+EN-ThM4 Temperature Dependent Structural and Optical Properties of SnO_2 Thin Film**, ***Junbo Gong***, *R.C. Dai, Z.P. Wang, Z.M. Zhang, Z.J. Ding*, University of Science and Technology of China

SnO_2, which is an n-type semiconducting material with a wide band gap 4 eV, is an interesting material due to its high electrical conductivity and optical transparency. SnO_2 film is attractive for many applications such as optoelectronic devices, gas sensors, thin film transistors, transparent electrodes, anti-reflecting coating, and as catalyst support.

In this work, the ellipsometric parameters of SnO_2 films on quartz glass are measured by spectra ellipsometer(J. A. Woollam M-2000U) in the wavelength range of 300 to 1000 nm at different temperature from room temperature to 600 ^oC. By using a semitransparent model, the precise thickness and optical constants of SnO_2 thin film depending on the temperature were obtained and the evolution process was studied. The film thickness significantly decreased with increased temperature from 100 ^oC to 300 ^oC and the absorption edge has an obvious blue shift which means an increased band gap. The result reveals that this process is not reversible. Combined with XRD measurement, we identified that the change of thickness and optical properties of SnO_2 film was due to a phase transition from rutile structure to columbite structure.

9:20am **EL+EM+EN-ThM5 Determining Curvature Radius of a Curved Surface by use of Mueller Matrix Ellipsometry**, ***Weiqi Li***, *H. Jiang, C.W. Zhang, X.G. Chen, S.Y. Liu*, Huazhong University of Science and Technology, China

Determining curvature radius of a curved surface by use of Mueller matrix ellipsometry

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Ellipsometry is a powerful metrology tool for the characterization of surfaces and thin films. Generally, the basic principle of conventional ellipsometry is based on the assumption that the studied film or structure is on a planar surface [1], in another word, the conventional ellipsometry works the best for a flat surface. When the studied surface is tilted or curved, the measurement accuracy of the conventional ellipsometry will be significantly degraded, or even be incorrect. It is thus important to develop a method to deal with the cases when the surface for characterization is tilted or curved. Comparing with the conventional ellipsometry, the Mueller matrix ellipsometry (MME) can provide all 16 elements of a 4 by 4 Mueller matrix, and consequently can acquire much more useful information about the curved surface and thereby shows great potential in the curved surface metrology.

In this work, we propose an optical model that is able to process curved surface based on our in-house developed dual rotating-compensator MME [2] to characterize the surface layer of a single crystal silicon sphere crown with a radius of about 51 mm for demonstration. Focus probe accessory is used in the MME to achieve sufficient small spot on the curved surface so that the detected area on the spherical surface can be approximately regarded as a tilted one. We found that some of the measured off-diagonal Mueller matrix elements are very sensitive to the offset between the actual detected spot and the surface vertex, which is proportional to the deviation angle α of the surface normal across the surface vertex. An optical model of the spherical layer is proposed by considering the curved surface of the silicon sphere crown and the offset. With the proposed model, the deviation angle α as well as the surface layer thickness can be extracted from the measured Mueller matrix spectrum, and then the curvature radius of the sphere crown can be achieved. Experiments are performed on the silicon sphere crown show that not only the accuracy of measurement can be improved but also the curvature radius of the sphere crown is capable to be measured using the proposed optical model.

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[2] S. Y. Liu, X. G. Chen, and C. W. Zhang, *Thin Solid Films* **584**, 176-185 (2015).

9:40am **EL+EM+EN-ThM6 Cavity-Enhanced Optical Hall Effect in AlInN/GaN-based HEMT Structures Detected at Terahertz Frequencies**, ***Sean Knight***, University of Nebraska-Lincoln, *S. Schöche,* J.A. Woollam Co. Inc., *V. Darakchieva, P. Kühne,* Linköping University, Sweden, *J.-F. Carlin, N. Grandjean,* Ecole Polytechnique Fédérale de Lausanne (EPFL), Switzerland, *C.M. Herzinger,* J.A. Woollam Co. Inc., *M. Schubert, T. Hofmann*, University of Nebraska-Lincoln

The terahertz optical Hall effect (THz-OHE) has been established as a non-contact and therefore valuable tool for the investigation of free charge carrier properties in semiconductor heterostructures [1-4]. In this work, we demonstrate that the THz-OHE signal for samples grown on THz transparent substrates can be controlled and enhanced by a tunable, externally coupled Fabry-Pérot cavity mode [5]. An AlInN/GaN-based high electron mobility transistor structure (HEMT) grown on a sapphire substrate is investigated as an example, while the cavity enhancement phenomenon discussed here is generally applicable to situations when a layered sample is deposited onto a THz transparent substrate. We show that in the vicinity of an externally coupled-cavity mode, a strong enhancement of the OHE signatures of up to one order of magnitude can be achieved by optimizing the cavity geometry, which is very useful for small magnetic field strengths. This signal enhancement allows the determination of free charge carrier effective mass, mobility, and density parameters using OHE measurements in low magnetic fields. Previously, high-field electromagnets needed to be employed for THz-OHE measurement for the determination of free charge carrier parameters in semiconductor heterostructures. Tuning the external cavity allows an enhancement of the THz-OHE signatures by as much as one order of magnitude. We propose to employ this enhancement effect to reliably and accurately determine free charge carrier properties in semiconductor structures at small magnetic fields dispensing with the need for expensive high magnetic fields. Cavity-enhanced THz-OHE may therefore enable the wide spread contactless measurement of free charge carrier properties at THz frequencies and which is indispensable for the development of the next generation of group-III nitride-based high frequency devices.

11:00am **EL+EM+EN-ThM10 Biosensor based on Imaging Ellipsometry and its Biomedical Applications**, *Y. Niu*, ***Gang Jin***, Institute of Mechanics, Chinese Academy of Sciences, China **INVITED**

The concept of biosensor based on imaging ellipsometry (BIE) was proposed in 1995 [1, 2]. With the development in recent 20 years, it has been formed an automatic analysis technique for detecting biomolecule detection interaction with merits of rapid, label-free, quantitative, high throughput and real-time. Its principle, methodology, biosensor system and biomedical applications are reviewed in this report.

A BIE system can be divided into four parts: the microfluidic array reactor, the imaging ellipsometer, the control system, and the biosensor database. The microfluidic array reactor serves to fabricate the protein microarray and accommodate biomolecular interactions. Using the microfluidic array reactor, various ligands are immobilized to different cells to form a sensing array, and each sensing surface can be prepared homogeneously under the flow condition. The imaging ellipsometer acts as a reader for data acquisition from the microarray. Since imaging ellipsometry is sensitive to slight variations of optical thickness, it can be used to visualize ultra-thin films and the change of molecular mass surface concentration. The control system combines the reactor with the imaging ellipsometer and functions to control the hardware's mechanical motion and obtain results in images, while the biosensor database is to aid BIE users in determining optimized experimental conditions and comparing previous test data.

The sensitivity and flexibility of the biosensor is very important for practical purpose, especially in biomedical fields. The sensitivity depends not only upon the resolution of imaging ellipsometry but also upon the bio-system of ligand-receptor on the microarray that is the bioactivity and its act related to the ligand screen, ligand immobilization, unspecific blocking and interaction conditions, etc. The flexibility mainly depends on the mechanical, electrical, informatics and biological control. So far, a serviceable engineering system of the biosensor and some bio-systems is installed available for more applications, especially for high throughput protein analysis, such as antibody screening [3], disease markers serological detection [4] and joint detection of tumor markers [5] as well as virus infection identification [6-7].

References

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11:40am **EL+EM+EN-ThM12 Screening Breast Cancer by Joint Detection of Tumor Marker Carbohydrate Antigen 15-3 and Carbohydrate Antigen 242 with Biosensor Based on Imaging Ellipsometry**, ***Yu Niu**, G. Jin*, Institute of Mechanics, Chinese Academy of Sciences, China

Breast cancer which develops from breast tissue is the leading type of cancer in women worldwide, accounting for more than 25% of all carcinogenesis [1]. Compared with other common cancers, the survival rate of breast cancer is remarkably positive and optimistic that between 80% and 90% of those in developed country could be alive for at least 5 years. Therefore, screening high risk population and further concluding a clinical diagnosis in the early stage act as a pivotal factor to cure breast cancer, because it can provide overwhelming contribution to carry out essential therapy in time. Carbohydrate Antigen 15-3 (CA 15-3) and Carbohydrate Antigen 242 (CA 242) are widely-used tumor markers for breast cancer in clinic and their concentrations in serum vary sensitively with breast cancer genesis. The biosensor based on imaging ellipsometry (BIE) for visualization of biomolecular interactions was reported in 1995 [2] and now it is composed of a 48 protein unit array and imaging ellipsometry reader with a field of view (20 x 30 mm) and good resolution for protein adsorption layer on a silicon substrate (lateral and vertical is 1 μm and 0.1 nm, respectively) [3]. In this investigation, joint detection of these two tumor markers simultaneously has been performed with BIE as a trial for screening breast cancer for clinical purpose.

To realize the joint detection, a series of design and optimization has been performed, including the ellipsometric setting, ligand immobilization strategy, ligand surface density, as well as the blocking and rinsing procedures. The test concentration range calibration and the detection limit for quantitative detection have been established by standard samples, which meet the standards of clinical test. By diluting sera to the detection range fitting to the calibration curves, joint quantitative detection of CA 15-3 and CA 242 can be achieved simultaneously.

149 serum samples composed of both the healthy and patients have been performed with BIE. Compared with the results obtained by standard approaches in clinic, the correlation analysis indicates the BIE are highly consistent with clinical methods. In order to estimate the BIE performance for tumor markers detection, ROC curve analysis has been introduced. Its result suggests that the single marker detection by BIE presents good capability to distinguish the normal from patients and the joint detection of CA 15-3 and CA 242 plays a positive role in the improvement of the diagnosis specificity and accuracy.

Reference

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12:00pm **EL+EM+EN-ThM13 Decomposition of Angle Resolved Spectroscopic Mueller Matrices from Scarabaeidae Beetles**, ***Roger Magnusson***, Linköping University, Sweden, *R. Ossikovski, E. Garcia-Caurel,* LPICM-CNRS, Ecole Polytechnique, France, *K. Järrendahl, H. Arwin*, Linköping University, Sweden

We use angle-dependent Mueller-matrix spectroscopic ellipsometry (MMSE) to determine Mueller matrices of Scarabaeidae beetles which show fascinating reflection properties due to structural phenomena in the exocuticle which are often depolarizing. It has been shown by Cloude [1] that a depolarizing matrix can be decomposed into a sum of up to four non-depolarizing matrices according to $\mathbf{M}= a\mathbf{M}_1+b\mathbf{M}_2+c\mathbf{M}_3+d\mathbf{M}_4$, where a, b, c and d are eigenvalues of the covariance matrix of **M**. Using the same eigenvalues the matrices $\mathbf{M}_i$ can be calculated. This method provides the full solution to the decomposition with both the non-depolarizing matrices and the weight of each of them in the sum.

An alternative to Cloude decomposition is *regression decomposition*. Here any Mueller matrix can be decomposed into a set of matrices $\mathbf{M}_i$ which are specified beforehand. Whereas in Cloude decomposition the only constraint on the matrices is that they are physically realizable non-depolarizing Mueller matrices, we can now limit the constraint and only use Mueller matrices representing pure optical devices having direct physical meaning, such as polarizers, retarders, etc. This leaves a, b, c, d as fit parameters to minimize the Frobenius norm $\mathbf{M}^{exp}$ -$\mathbf{M}^{reg}$ where $\mathbf{M}^{exp}$ is the experimentally determined Mueller matrix to be decomposed and $\mathbf{M}^{reg}$ is the sum of all $\mathbf{M}_i$. Depending on $\mathbf{M}^{exp}$ an appropriate choice of $\mathbf{M}^{reg}$ matrices has to be made and different values of a, b, c and d are obtained through regression analysis.

We have previously shown that regression decomposition can be used to show that the Mueller matrix of *Cetonia aurata* can be decomposed into a sum of a circular polarizer and a mirror [2]. Here we expand the analysis to include angle-resolved spectral Mueller matrices, and also include more species of Scarabaeidae beetles.

One effect of the decomposition is that when depolarization is caused by an inhomogeneous sample with regions of different optical properties the Mueller matrices of the different regions can be retrieved under certain conditions. Regression decomposition also has potential to be a classification tool for biological samples where a set of standard matrices are used in the decomposition and the parameters a, b, c, d are used to quantify the polarizing properties of the sample.

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Authors Index

Bold page numbers indicate the presenter