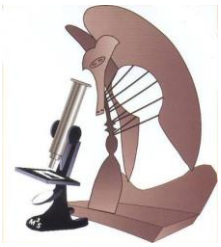
Announcements from MMMS Corporate Members 2023

Hosted by Baxter Healthcare Corporation



Presented by

Midwest Microscopy and Microanalysis Society (M^3S)

A local affiliate of the Microscopy Society of America and the Microanalysis Society

Friday, November 17th

Baxter Healthcare Headquarters

1 Baxter Parkway, Deerfield IL, 60015

Directions see page 5

Register by Friday, November 10th

Use the link below, this is required to reserve a lunch for this event

https://baxter.cventevents.com/M3SFall2023

Registration Fees & Program

Meeting Free for M^3S members, UnderGraduate & Graduate Students
Registration Fee $20.00 for non-Members

Registration includes M^3S membership for 2024 and is Payable on Site
Registration also includes Continental Breakfast, Refreshments & Lunch
Vendors are welcome to Exhibit, Table Fee is $125.00.

8:00 – 9:00AM **Registration -** Continental Breakfast

9:00 – 9:10AM **Welcome and Opening Remarks**

9:10 – 10:15AM **Eavesdropping on cardiac muscle cells using multi-scale light and electron microscopy**

Rengasayee Veeraraghavan, Associate Professor, The Ohio State University. **MSA Tour Speaker**

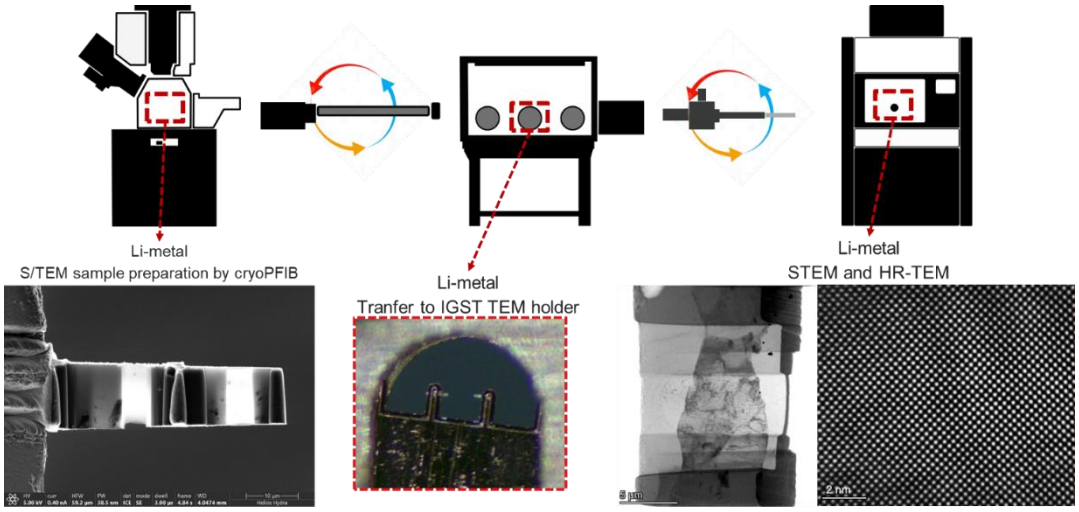
Cardiac conduction is the process by which electrical excitation spreads through the heart, triggering individual myocytes to contract in synchrony. Defects in conduction disrupt synchronous activation and are associated with life-threatening arrhythmias in many pathologies. For over a century, cardiac conduction has been viewed as occurring solely through the direct flow of ionic current from cell to cell via gap junctions. However, this view was challenged as early as 1960 based on studies in avian hearts, where conduction occurs in the near total absence of gap junctions. Since then, a growing body of phenomena that cannot be well explained by electrotonic coupling alone have led some to hypothesize a role for an alternate mechanism named ephaptic coupling in cardiac conduction. This non-canonical mechanism, which is known to occur in other tissues such as the brain and the retina, envisions cells communicating via electrochemical transients within restricted nanodomains at cell-cell contacts. In recent years, we and others have used multiscale light and electron microscopy, functional imaging and computational modeling approaches to provide strong evidence supporting a role for ephaptic coupling in the heart and identified ion channel-rich nanodomains within intercalated discs (cell-cell contact sites) as candidates for the cardiac ephapse. In addition to expanding our theoretical understanding of cardiac conduction and explaining seemingly paradoxical experimental and clinical findings, this work has yielded important insights into the mechanisms of cardiac arrhythmia and enabled us to develop novel antiarrhythmic therapies.

10:15 – 10:40AM **Unleashing the Power of CryoFIB in Materials Science Research**

Ken Wu, Materials and Structural Analysis, Thermo Fisher Scientific, 5350 NE Dawson Creek Drive. Hillsboro, Oregon

Electron microscopy and ion beam systems are essential tools in today's materials science applications, research, development, and discovery. The DualBeam FIB-SEM harnesses the power of both electrons and ions by combining a focused ion beam (FIB) and scanning electron microscope (SEM) in single instrument. This harmony of capabilities allows for site specific cross-section generation used in failure analysis, 3D characterization via Slice & View for detailed volumetric studies, preparation of ultra-thin lamella for transmission electron microscopy (TEM) analysis at atomic resolution, creation of high-quality tips for atom probe tomography (APT), and much more. However, ionbeam induced heating and beam/sample interactions can create difficulties in characterizing hydrated, low-Z, or soft materials such as polymer, battery components and biological samples. In step with advancements in cryogenic transmission electron microscopy, Thermo Fisher Scientific has also improved cryogenic DualBeam FIB-SEM (CryoFIB) techniques to make them more efficient, effective, and readily accessible to all users. The sample stage within DualBeam systems can now be equipped with a 360° rotatable cryogenic stage capable of reaching temperatures as low as -180°C within just 20 minutes. Moreover, the integration of a Cryo EasyLift nanomanipulator facilitates TEM lamella preparation while minimizing heat transfer and thus mitigating ion-beam induced sample heating as well as artifacts from start to finish. Additionally, there is a wide range of ion species to choose from depending on the targeted sample material, including Xe+ and Ar+, to produce TEM lamella without the detrimental effects of Ga+ implantation. While having capabilities such as these is an advantage, they are not really practical for achieving end goals (imaging, analysis, etc.) if viewed as completely independent components. Therefore, Thermo Fisher Scientific has focused on ensuring seamless workflows top to bottom, to achieve the goal of high-quality final images, results and data. In order to demonstrate a complete workflow from DualBeam to TEM for a challenging sample, a bulk piece of Li-metal was utilized as it is very sensitive air, moisture and temperature. Figure 1 outlines the schematic representation of this workflow. First, the DualBeam milling process was conducted at cryogenic temperatures to minimize

beam-induced damage to the sample, resulting in a very high-quality TEM. lamella. Next, the Thermo Scientific Inert Gas Sample Transfer



(IGST) system was used to protect the Li-metal and transfer the prepared lamella in an Ar atmosphere with the use of a CleanConnect module. Due to lithium's low melting point, the entire TEM lamella preparation process in the DualBeam was carried out under cryogenic temperatures. After the final thinning step, the lamella was then transferred to a glovebox via the CleanConnect and subsequently loaded into a Mel-Build Double Tilt LN2 Atmos Defend TEM holder. This holder allows movement of the lamella from the glovebox to the TEM while maintaining an Ar gas environment. Finally, with the holder/lamella in the TEM, the goal of artifact-free atomic resolution images and EELS data was achieved, demonstrating the workflow successfully protects the Li-metal lamella from oxidation during transfer between multiple instruments. In this presentation various CryoFIB techniques and workflows will be discussed including methods of needle attachment, sample lift out, and transfer. Additional examples from polymer and biological samples will also be shown.
Figure 1. Complete Workflow from DualBeam to TEM on Li-metal Sample

10:40 – 11:00AM Break – Visit with Vendor

11:00 - 11:20AM In situ Liquid Cell TEM Studies of Nucleation and Growth in Colloidal Multielement Alloy Nanoparticles in Solution

Azadeh Amiri[1], Vitaliy Yurkiv[2], Abhijit H. Phakatkar[3], Tolou Shokuhfar[3], Reza Shahbazian-Yassar[1]*
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The nucleation and growth of colloidal multielement nanoparticles are fundamental processes that determine the size, shape, and properties of resulting nanoparticles. However, the mechanisms behind the formation and growth of these colloidal multielement alloy nanoparticles are complex and not fully understood. Identifying the precursors leading to the formation of medium to high entropy nanoparticles is challenging due to the involvement of multiple elements with distinct properties. In this study, we investigated the in-situ colloidal synthesis of multielement alloys using liquid-cell transmission electron microscopy (TEM). The structure and composition of resulting nanoparticles were characterized using annular dark field scanning TEM (ADF-STEM) imaging and energy-dispersive spectroscopy (EDS) techniques. Two distinct pathways for nanoparticle formation were observed in a solution containing Au, Pt, Ir, Cu, and Ni elements, resulting in two sets of particles. One set of nanoparticles had a high content of Au and Cu, ranging in size from 10 to 30 nm. The other set consisted of multielement alloys composed of Pt, Cu, Ir, and Ni elements, with sizes less than 4 nm. The findings suggest that, in this synthesis approach, factors beyond the mixing of multiple elements come into play. The nature and characteristics of metal ion clusters, including their reduction rates and oxidation valence numbers, play a crucial role in determining the initial particle composition during the early stages of formation. Consequently, the variations in constituent elements and nanoparticle composition, along with their respective surface properties and activities, collectively contribute to the distinct growth behaviors observed for each nanoparticle set. This study provides insights into the mechanisms underlying the formation and growth of multi-element nanoparticles, highlighting the importance of understanding the interplay between composition, surface energies, and the miscibility of constituent elements.

11:20 - 11:45AM Machine-Vision Assisted In Situ Transmission Electron Microscopy

Gabriela Mendoza, Dylan Wood and Madeline Dressel Dukes, Protochips

The development of *in situ* and o*perando* techniques, which transform the transmission electron microscope (TEM) into a real-time nanoscale laboratory, has opened exciting windows into dynamic processes that control a material's growth, phase transformation and degradation behaviors. Experiments previously limited to bulk samples on the benchtop, or that required time-consuming, iterative sample preparation, such as temperature cycling, electrical analysis, catalysis, and electrochemistry, can now be performed directly within the TEM to provide a clearer understanding of the underlying nanoscale mechanisms and lead to new discoveries. These studies introduce new challenges in data collection, analysis, and reproducibility 1. *In situ* experiments require careful alignment of parameters from multiple sources as the changing sample conditions, applied stimuli and measured activity must be accurately aligned and indexed to the corresponding TEM images 2. The Protochips' family of *in situ* systems, Fusion-AX (temperature, electrical and electro-thermal), Poseidon-AX (liquid and electrochemistry), and Atmosphere-AX (gas environments and catalysis) combine unparalleled *in situ* TEM hardware with a machine-vision platform designed to streamline and enhance TEM operation and workflows, consolidate experimental

parameters and metadata, and employ machine-assisted algorithms to aid decision making. Here we will present case studies that apply *in situ* techniques to study relevant bulk metallurgical processes such as corrosion 3–5, and phase transformation during annealing6 and hardening processes7 at the nanoscale level. Images and metadata generated during an experiment performed using Protochips' *in situ* - AX system is collected and indexed through the machine vision software, AXON. User-triggered changes during the experiment (such as adjustments to beam conditions, detector acquisitions, the *in situ* environment, and applied stimuli) and measured sample responses are organized into an experimental timeline making it easy to sort, filter and analyze large datasets. The robust features and straightforward design of the *in situ* - AX systems coupled with a powerful machine-vision software enables users to take advantage of the latest innovations of *in situ* research and explore the dynamic nanoscale world around them.

(1) Boyes, E. D.; LaGrow, A. P.; Ward, M. R.; Martin, T. E.; Gai, P. L. Visualizing Single Atom Dynamics in Heterogeneous Catalysis Using Analytical in Situ Environmental Scanning Transmission Electron Microscopy. *Philosophical Transactions of the Royal Society A: Mathematical, Physical and Engineering Sciences* **2020**, *378* (2186), 20190605. https://doi.org/10.1098/rsta.2019.0605.
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(3) Kovalov, D.; Taylor, C. D.; Heinrich, H.; Kelly, R. G. Operando Electrochemical TEM, Ex-Situ SEM and Atomistic Modeling Studies of MnS Dissolution and Its Role in Triggering Pitting Corrosion in 304L Stainless Steel. *Corrosion Science* **2022**, *199*, 110184. https://doi.org/10.1016/j.corsci.2022.110184.
(4) Schilling, S.; Janssen, A.; Zaluzec, N. J.; Burke, M. G. Practical Aspects of Electrochemical Corrosion Measurements During *In Situ* Analytical Transmission Electron Microscopy Protochips (TEM) of Austenitic Stainless Steel in Aqueous Media. *Microsc Microanal* **2017**, *23* (4),741–750. https://doi.org/10.1017/S1431927617012314.
(5) Zhong, X. L.; Schilling, S.; Zaluzec, N. J.; Burke, M. G. Sample Preparation Methodologies for In Situ Liquid and Gaseous Cell Analytical Transmission Electron Microscopy of Electropolished Specimens. *Microscopy and Microanalysis* **2016**, *22* (6), 1350–1359. https://doi.org/10.1017/S1431927616011855.
(6) Shao, S.; Zhu, X.; Ten, V.; Kim, M. J.; Xia, X. Understanding the Impact of Wall Thickness on Thermal Stability of Silver–Gold Nanocages. *J. Phys. Chem. C* **2022**, *126*, 7337–7345. https://doi.org/10.1021/acs.jpcc.2c01433.
(7) Hillel, G.; Kalabukhov, S.; Frage, N.; Zaretsky, E.; Meshi, L. Direct Observation of Initial Stages of Precipitation Hardening Process in Commercial Al 6061 Alloy. *J Mater Sci* **2022**,*57* (22), 10395–10406. https://doi.org/10.1007/s10853-022-07341-2.

11:45AM – 1:15PM Lunch – Visit with Vendors

1:15 – 1:30PM MMMS Business Meeting

1:30 – 2:15PM Ultrafast Electron Microscopy: Exploring Nanoscale Dynamics

Thomas Gage, Assistant Scientist, Argonne National Laboratory **Invited Speaker**

Ultrafast Electron Microscopy (UEM) is a cutting-edge technique that revolutionizes scientific exploration, combining the exceptional spatial resolution of electron microscopes with the unmatched temporal precision of ultrafast lasers. This presentation will delve into the capabilities and contributions of the UEM facility at the Center for Nanoscale Materials, highlighting recent developments and scientific achievements. The discussion will primarily focus on two scientific directions that have gained substantial momentum in UEM research: phonon imaging and plasmonic field imaging. I will showcase the pivotal role of UEM in visualizing the dynamics of phonons and plasmonic fields at the nanoscale, enabling a deeper understanding of their interactions and behaviors in a wide range of materials. In addition to these specific applications, I will offer a broader perspective on the state of the UEM field, elucidating the current landscape and trends in ultrafast electron microscopy. Furthermore, I will outline our vision for the future, detailing how we plan to propel our microscopy center forward by embracing novel techniques and fostering collaborative scientific exploration.

2:15 – 2:40PM Multimodal STEM Experimentation and Data Analysis with Gatan DigitalMicrograph

Fernando C. Castro, Ph.D., Imaging & In-Situ Applications Scientist Gatan

Advancements in detector hardware and software control enable progressively more complex electron microscopy experiments, which can involve signals from multiple detectors, increased dataset dimensionality, and higher data rates and larger overall data size. This presentation will review Gatan's eaSI™ technology – a set of tools available to all DigitalMicrograph users – which enables multimodal STEM experiments and links and synchronizes the acquired data for efficient analysis. Furthermore, methods will be reviewed for high-speed 4D STEM acquisition with the Gatan ClearView camera, *in-situ* experiments using simultaneous EDS + EELS and 4D STEM+EDS acquisition, and multi-signal Spectrum Image analysis.

2:40 – 3:00PM Nanoscale Characterization of the Back Contact in CdTe Solar Cells

Noah Kamm[1], Ebin Bastola[2], Manoj Jamarkattel[2], Michael Heben[2], Walajabad S Sampath[3], James Sites[3], Robert F Klie[1]
[1]University of Illinois at Chicago, Chicago, IL, United States; [2]University of Toledo, Toledo, OH, United States; [3]Colorado State University, Fort Collins, CO, United States

CdTe has been a promising solar cell material candidate for decades, owing to its polycrystalline nature, causing it to have a relatively lower manufacturing cost than traditional Si cells. The best cells made have an efficiency of 22.1%, being close in competition with Si cells; however, this is still far away from the Shockley-Queisser limit. [1] One major problem facing CdTe cells is that the work function of the back contact layer is very high, making Ohmic contact formation challenging. The formation of a non-Ohmic contact leads to high effective resistance, greatly reducing the efficiency of the cell. One common way to partially circumvent this problem is through Cu doping in the CdTe layer; this technique is used in the current highest efficiency CdTe cells. [2] However, Cu doping in CdTe cells also leads to device degradation, leading to it becoming a subject of research interest. [3]

In this talk, we will investigate a CdTe cell that is Cu doped while also being passivated with Cl (another common technique to improve the performance of CdTe cells [4]) and having TeO_2 formation near the back contact. This TeO_2 formation may reduce electron-hole recombination at the back contact and thus provide an even better back contact interface. In particular the material of interest is a cell with a CdTe/Au back contact that has been treated with $CdCl_2$ and Cu_xAlO_y. The use of Au is due to its high work function [5]. The $CdCl_2$ and Cu_xAlO_y are used to passivate the cell grain boundaries with [**Cl**] and dope the cell with Cu respectively. Using an aberration-corrected scanning electron transmission microscope (STEM), energy-dispersive X-ray spectroscopy (EDS) and electron energy loss spectroscopy

(EELS) elemental characterization techniques will be performed, as well as annular dark-field (ADF) imaging, in order to determine the nanoscale structure at the back contact of this cell. [6]

[1]M. A. Scarpulla, B. McCandless, A. B. Phillips, Y. Yan, M. J. Heben, C. Wolden, G. Xiong, W. K. Metzger, D. Mao, D. Krasikov, I. Sankin, S. Grover, A. Munshi, W. Sampath, J. R. Sites, A. Bothwell, D. Albin, M. O. Reese, A. Romeo, M. Nardone, R. Klie, J. M. Walls, T. Fiducia, A. Abbas, and S. M.
Hayes, "CdTe-based thin film photovoltaics: Recent advances, current challenges and future prospects", Solar Energy Materials and Solar Cells **255**, 112289 (2023).
[2]R. S. Hall, D. Lamb, and S. J. C. Irvine, "Back contacts materials used in thin film CdTe solar cells—A review", en, Energy Science & Engineering **9**, eprint: https://onlinelibrary.wiley.com/doi/pdf/10.1002/ese3.843, 606–632 (2021).
[3]D. Krasikov, D. Guo, S. Demtsu, and I. Sankin, "Comparative study of As and Cu doping stability in CdSeTe absorbers", Solar Energy Materials and Solar Cells **224**, 111012 (2021).
[4]Y. Wang, G. Wang, Y. Zhou, Q. Xie, J. Chen, K. Zheng, L. Zheng, J. Pan, and R. Wang, "Research progress in doped absorber layer of CdTe solar cells", Renewable and Sustainable Energy Reviews **183**, 113427 (2023).
[5]W. M. H. Sachtler, G. J. H. Dorgelo, and A. A. Holscher, "The work function of gold", Surface Science **5**, 221–229 (1966).
[6]This material is based upon work supported by the U.S. Department of Energy's Office of Energy Efficiency and Renewable Energy (EERE) under the Solar Energy Technologies Office Award Number DE-EE0008974.

3:00 – 3:20PM Atomic structure and ordering of the 1D lepidocrocite-based TiO_2 nanofilaments (NFs)

Francisco Lagunas[1], Hussein O. Badr[2], David Bugallo[2], Yingjie Yang[1], Fatemeh Karimi[1], Robert F. Klie[1], Yong-Jie Hu[2], and Michel W. Barsoum[2]

[1] Department of Physics, University of Illinois at Chicago, Chicago, Illinois 60616, United States

[2] Department of Material Science &Engineering, Drexel University, Philadelphia, Pennsylvania, 19104, United States

1D titania-based nanofilaments (NFs) successfully synthesized by Badr et al. [1] using various Ti-containing, earth-abundant, and non-toxic powder precursors. This synthesis process is carried out by immersing the precursor powders in an aqueous solution of tetramethylammonium hydroxide (TMAH). Subsequently, the colloidal suspensions formed are subjected to vacuum filtration, resulting in the formation of free-standing Filtered Films. The resulting TiO_2-based 1D NFs have distinct physical and chemical characteristics that make them appropriate for use in a wide range of potential applications, including photocatalysis, photoluminescence, and paint pigments.[2] These NFs have the potential to stimulate innovations and improvements in a wide range of industries, thereby contributing to a greener and more technologically sophisticated future.[3]

In this contribution we present atomic resolution scanning transmission electron microscopy (STEM) analysis to provide insights into the atomic structure and ordering of the TiO_2 NFs. We observed that the TiO_2 NFs were 20-30 nm long. They predominantly grow along the [200] direction and stack along the b-direction in the plane that the NFs self-assemble to either create bundles or larger 2D flakes. Measuring the domain size of the NFs along c-direction from STEM images gives an average width of 3.0±1.0 nm. Based on atomic-resolution STEM, simulated electron diffraction patterns, and density functional theory modeling, cross sectional views of aligned TiO_2 nanoribbons show Lepidocrocite-based TiO_2 structures with minimal cross sections of 5x7 $Å^2$.[4]

1. Hussein. O. Badr, Jacob. Cope, Takayuki. Kono, et al., "Titanium Oxide-based 1D Nanofilaments, 2D Sheets, and Mesoporous Particles - Synthesis, Characterization, and Ion Intercalation", Matter (2023).
2. Hussein O. Badr, Francisco Lagunas, Daniel E. Autrey, et al., "On the structure of one-dimensional TiO_2 lepidocrocite", Matter 6, 1–14, January 4, 2023.
3. Hussein O. Badr, Tarek El-Melegy, Michael Carey, et al. "Bottom-up, scalable synthesis of two-dimensional titanium carbo-oxide-based conductive flakes", Mat. Today 54 (2022) 8-17.
4. This work is supported by a grant from the National Science Foundation (DMR-2309396)

3:20 – 3:45PM A new FIB/SEM for TEM sample preparation workflow

Patrick Phillips JEOL USA, Inc. Peabody, MA, USA

The newly released JIB-PS500i offers new hardware adaptations that simplify and improve the workflow to go from a bulk material sample to imaging in a TEM. In many fields of materials science, such as battery, semiconductor, thin film, quantum, etc., site-specific analysis is critical. Be it for chemical, structural, or electronic characterization of defects and interfaces, it is oftentimes necessary to identify a TEM sample region of interest on the nanometer scale. The JIB-PS500i aims to streamline lamella preparation workflow while simultaneously improving the quality of the lamellae.

The combination of an increased motion stage, and the transferable TEM sample cartridge, removes some of the cumbersome steps typically associated with preparing a high-quality lamella. By having the ability to place the lamella inline with the electron beam and a STEM detector, operators can screen their samples without venting. Not only do these adaptations allow for removal of processing steps, but also opens specialized sample preparation workflows such as battery materials and other oxygen sensitive samples.

3:45 – 4:05PM Guest molecule-mediated energy harvesting in a dynamic peptide–metal organic framework

Yu Chen, Northwestern University

The apparent piezoelectricity of biological materials is not yet fully understood at the molecular level. In particular, dynamic non-covalent interactions, such as host-guest binding, are not included in the classical piezoelectric model, which limits the rational design of eco-friendly piezoelectric supramolecular materials. Here, inspired by the conformation-dependent mechanoresponse of the Piezo channel proteins, we show that guest-host interactions can amplify the electromechanical response of a conformationally mobile peptide metal-organic framework (MOF) based on the endogenous carnosine dipeptide, demonstrating a new type of adaptive piezoelectric supramolecular material. Density functional theory (DFT) predictions validated by piezoresponse force microscopy (PFM) measurements show that directional alignment of the guest molecules in the host carnosine-zinc peptide MOF channel determines the macroscopic electromechanical properties. We produce stable, robust 1.4 V open-circuit voltage under applied force of 25 N with a frequency of 0.1 Hz. Our findings demonstrate that the regulation of host-guest interactions could serve as an efficient method for engineering sustainable peptide-based power generators.

4:05 – 4:15PM Closing Remarks

Directions to Baxter Corporate Headquarters: 1 Baxter Parkway, Deerfield Illinois, 60015

From South (O'Hare Airport): I-294 (Tri State Tollway) north to the merge with I-94 (west) towards Milwaukee. North on I-94 to Lake Cook Road exit. Turn left (west) to first light, Saunders Road. Turn right on Saunders to Baxter Parkway. Turn right on Baxter Parkway. Keep to the right. Follow the special event parking signs in the garage. See Deerfield Campus Map and proceed to "Cafeteria, Auditorium, Reception" building on ground level.
From South (Edens): North to the merge with I-94 (west) towards Milwaukee on Edens Spur. Exit on Deerfield Road. Turn left (west), then take left on Saunders Road. Turn left on Baxter Parkway. Keep to the right. Follow the special event parking signs in the garage. See Deerfield Campus Map and proceed to "Cafeteria, Auditorium, Reception" building on ground level.
From North (Milwaukee): From I-94 east, going south towards Chicago exit at Lake Cook Road exit. Turn right (west) to first light, Saunders Road. Turn right on Saunders to Baxter Parkway. Turn right on Baxter Parkway. Keep to the right. Follow the special event parking signs in the garage. See Deerfield Campus Map and proceed to "Cafeteria, Auditorium, Reception" building on ground level