Arslan, Ilke – Argonne National Lab

Advanced Measurements on the Nanoscale Enabled by Technique Development in the Scanning Transmission Electron Microscope

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With recent advances in in-situ microscopy, a new era in microscopy has arrived that allows for the dynamic imaging of materials under reaction conditions in the (scanning) transmission electron microscope ((S)TEM). It is no longer sufficient to image materials under vacuum conditions, but to get closer to the conditions in which the material will be used, such as high temperature, liquid environments, gas environments, or a combination thereof. Combining an in-situ or ex-situ experiment with electron tomography is a very powerful method for materials characterization as this provides the 3-D morphology/chemistry of the materials in more relevant environments. Recent advances in both the fields of in-situ imaging and 3-D imaging are now making it possible to work towards 3-D in-situ imaging. This talk will focus on new developments such as in-situ heating in liquid, new reconstruction algorithms for electron tomography with significantly fewer images, and chemical composition mapping in 3-D. The benefits and limitations of these methods will be discussed, with an outlook on working towards combining them.

Batson, Philip – Rutgers University

Phonon Spectroscopy and Mapping in Nanostructures

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During the past 20 years, spatially resolved EELS has emerged as an important tool for the characterization of nanoscale structures designed to efficiently couple external photon fields into the bulk, using surface plasmon polariton excitations [1-3]. With recent instrumental improvements, phonon vibrational modes are now accessible down to a few tens of meV [4], opening up a new area for exploration – bulk and surface polariton phonons in nanoscale structures. This new area contains some surprises that we are currently trying to understand. These include screening of very low energy excitations by higher energy electronic transitions; a highly occupied ground state, obeying boson population statistics; and strong multipole scattering which swamps the dipole response observed using photon absorption and small angle dipole electron scattering.

Local screening is an issue because vibrational modes lie at very small energies – below about 200 meV. In microscopy, keV electrons produce a plasma of many highly excited electronic which can screen the swift electron potential, suppressing the electron's ability to interact at long range. Thus in these materials, the bulk phonon scattering contains high spatial resolution components that can be mapped.

Also for the very low energy vibrational modes, the occupied state population strongly distorts the experimental scattering at and below the 25-30meV room temperature energy, especially for the acoustic modes. Scattering intensities below this range become very strong, as much as 10% of the zero loss peak. While these increase the visibility of the vibrational modes near the zero loss, the distortion needs to be understood to derive a phonon density of states for comparison with other spectroscopies.

Simple optical modeling of electron scattering fails for several symmetries of phonon modes in typical materials. Transverse optical, and acoustic modes do not appear in the IR spectroscopies, for instance. Some of this has become clearer, particularly the dependence of spatial resolution on scattering conditions [6], and on short-ranged multipole scattering components [7-8].

However, it is still difficult to measure a Phonon Density of States which resembles accepted understanding in known situations. We think that this can be understood using a careful analysis of spatially resolved EELS in a diffraction limited electron optical system, using coherent summation over scattering into the EELS spectrometer collection aperture. This kind of analysis has been shown to work for determining local symmetry of an excited specimen state using deliberately designed electron channeling conditions [9]. The possibility represents interesting and exciting possibilities for future spatially resolved EELS measurements in modern, very high resolution spectroscopic systems.

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Bell, David – Harvard University

Identifying Topological Materials for Quantum Computing Applications with Advanced Electron Microscopy

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Depending on the composition, Quantum Materials may act as conductors, insulators, semiconductors or even as superconductors. Combinations of different quantum materials are of high interest to explore new phenomena and act as the foundation for future electronic devices at the nanometer scale. Our quantum materials research reaches from defect formation in graphene to the characterization of hybrid quantum materials. I will present our work utilizing Low-Voltage Monochromated EELS and Low-Voltage High-Resolution Electron Microscopy (LV HREM). Together, these often improve the contrast to damage ratio obtained on a large class of samples. The exploration and synthesis constitute only one aspect of the challenges in the development of new topological materials, another challenge is their characterization. Since the phenomena appear at very restricted and dedicated conditions, the characterization method must have very high sensitivity, resolution, localization and precision.

Transmission electron microscopy is a powerful technique to investigate structural, compositional or electromagnetic properties of topological materials. Especially, recent implementation of aberration correction in the transmission electron microscopy made chemical and structural characterization with very high spatial resolution (in the range of picometers) and sensitivity possible. This in turn allows detailed analysis of

superconductor and topological materials, where small compositional variations have large effects on the material properties.

For topological materials 2005 was an important milestone as a consequence the realization of the existence of a metallically conductive surface state in an insulator material. Within couples of years, the experimental evidences of the surface state followed the theory studies. A high spin orbit coupling creates edge states where quantum spin Hall Effect can exist in the absence of an external magnetic field. The discovery of the new phenomenon opened up intensive discussions in condensed matter, and even very well-known conventional material systems such as Bi2(Te,Se)3, BiSb alloys etc., became "exotic" and highly investigated materials again.

One idea is using the kagome lattice as a topological switch. The kagome lattice is a two-dimensional network of corner-sharing triangles known as a platform for exotic quantum magnetic states. Theoretical work has predicted that the kagome lattice may also host Dirac electronic states that could lead to topological and insulating phases, but these have evaded experimental detection to date. Fe3Sn2 is a rare metallic Kagome ferromagnet, which synthesis as a single crystal has not previously been reported. We study this single crystal as well as other topological insulators with the particular interest in the correlated behavior in topologically non-trivial materials.

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Cheng, Holland – University of California, Davis

Multimodal Imaging and Convolutional Neural Network for Nanomedicine

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Structural proteins of viruses have the capacity to function both in assembly and in disassembly, which is made possible through a built-in flexibility triggered by cellular events. Focusing on the highly selected viral capsid over their conformational domains between the metastable intermediates and the stable mature forms so far has provided us the essence of the needed stability, resisting extreme pH and digestive enzymes, to deliver medicals or agents to target tumors through both the circulation and the mucosal routes (1-3). Aided by multimodal imaging integrated with the deep learning via convolutional neural network to guide the tracking and the targeting of the payloads, the design principles of inserting heterologous epitopes to target the mucosal surfaces will be exemplified regarding in this presentation. Deep learning has gained enormous attention by the success of its convolutional neural networks in demonstrated machine learning tasks including high-content image classification. Crucial precision of cargo deliveries can be better realized through the AI deep-learning and the multiple modality of imaging domains to fully enable the targeting-engineered nanocapsids via various non-invasive mucosal routes. Further advancement of imaging technology like cryoEM and cellular tomography (see details in nobel.se; the Nobel Prize in Chemistry 2017) will be demonstrated in the unveiling of the associated molecular mechanisms essential to the platform vector design towards the success of constructing a non-invasive, mucosal targeting system (4-6).

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Crozier, Peter – Arizona State University

In Situ and *Operando* Electron Microscopy Imaging and Spectroscopy of Catalysts

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In-situ or *operando* environmental transmission electron microscopy (ETEM) is a powerful technique for the investigation of structure-reactivity relationships in high surface area catalysts under reaction conditions. With new instruments, atomic resolution imaging and spectroscopy can be carried out in the presence of gas, liquid, light and thermal stimuli [1-5]. The combination of mass spectrometry and electron energy-loss spectroscopy (EELS) allow catalytic products to be detected and quantified directly in the electron microscope. Through careful analysis of the ETEM reactor geometry, quantitative chemical reaction rates can be determine so that ***reaction kinetics*** may be correlated with changes in surface structure allowing true *operando* measurements to be performed where kinetics and structure can be related. For example, on CO oxidation over Ru, correlating reaction kinetics with structure modification allows us to show that thin layers of RuO_2 that partially cover the surface of the Ru nanoparticle under reaction conditions are ***not*** active sites but spectator species. This highlights the danger of simply assuming that structures observed under *in situ* conditions are related to functionality. *In situ* structures may or may not play an active role in functionality and rigorous *operando* measurements must be performed to establish more reliable structure-function relationships.

The oxygen exchange reaction is a fundamental process taking place on oxide surfaces involving the creation and annihilation of oxygen vacancies. We have investigated the structural rearrangements taking place on different surfaces of CeO_2 and TiO_2 nanoparticles. With negative C_s aberration corrected electron microscopy, oxygen columns may be directly visualized under favorable conditions especially when direct exposure detectors are employed. The creation of oxygen vacancies can also result in local cation relaxations, which are often easier to detect during *in situ* experiments. The interplay between oxygen vacancy formation energy, cation displacements and the oxygen exchange reaction will be explored and discussed.

Recent developments in monochromated EELS allow the electronic and vibrational structure of catalyst surfaces to be probed with focused electron beams [6]. Using the so-called “aloof beam” approach to EELS, radiation damage is controlled which should allow local electronic surface and defect states to be observed and correlated with catalytic properties [7,8]. Recent applications of these novel approaches will be illustrated to elucidate the structure and electronic properties of catalytic nanoparticles surfaces relevant to a variety of energy conversion processes such as water splitting, CO oxidation and reforming.

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Damiano, John – Protochips

Optimizing in situ TEM/STEM for EDS

Modern aberration-corrected electron microscopes offer unprecedented imaging and analytical capabilities, and in situ techniques allow scientists to study materials in realistic environments at high temperatures. However, the design of closed cell systems often limits the collection of EDS signals which are critical for materials characterization. This talk will describe the evolution of closed cell designs toward optimizing the collection of EDS spectra from samples in gas and liquid environments.

Dunin-Borkowski, Rafal – Institute for Microstructure Research

Towards three-dimensional characterization of magnetic moments inside individual nanocrystals in the transmission electron microscope

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Off-axis electron holography is a powerful technique for recording the phase shift of a high-energy electron wave that has passed through an electron-transparent specimen in the transmission electron microscope. The phase shift is, in turn, sensitive to the electrostatic potential and magnetic induction in the specimen, projected in the electron beam direction.

The determination of the local magnetization in a specimen from a phase image recorded using off-axis electron holography is a highly challenging ill-posed problem. We have developed a model-based iterative reconstruction technique, which can be used to retrieve the projected in-plane magnetization distribution from the magnetic contribution to a recorded phase image, or alternatively the three-dimensional magnetization distribution from two, optimally orthogonal, tilt series of magnetic phase images [1].

Our approach involves the optimized implementation of a forward model, which utilizes sparse-matrix multiplications for efficient projections and subsequent fast-Fourier-transform-based convolutions with pre-calculated convolution kernels based on analytical solutions for the magnetic phase images of simple geometrical objects. The ill-posed problem is tackled by first replacing the original problem by a least squares minimization, which is augmented by regularization to find a unique solution for the reconstructed magnetization distribution. Tikhonov regularization of first order is used to apply a smoothness constraint to the magnetization, which is justified by the minimization of exchange energy. A priori information about the positions and sizes of magnetic objects within the field of view can be incorporated in the form of a three-dimensional mask, which reduces the number of unknowns to be retrieved. The approach can account for arbitrary linear phase ramps and phase offsets, as well as for untrustworthy (low confidence) regions in phase

images. Diagnostic measures can be used to analyze the quality of the reconstruction results, while the chosen regularization strength can be optimized to obtain the best solution in the presence of noise.

Highly encouraging experimental results have been obtained from the reconstruction of the projected magnetization distributions of magnetic skyrmions examined in extended films and geometrically-confined structures, as well as from the reconstruction of three-dimensional magnetization distributions in sub-100-nm magnetite nanocrystals [2].

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Dwyer, Christian – Arizona State University

Prospects of vibrational mapping in the STEM

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Advances in instrumentation continue to make vibrational spectroscopy in the STEM an extremely exciting new field. Notwithstanding the obvious importance of energy resolution, of intrinsic relevance to many electron microscopists is the spatial resolution at which vibrational mapping can be achieved. In this talk, I will review the recent theoretical and experimental progress in the field, and highlight some potential future directions for achieving improved signal-to-noise and spatial resolution. I will describe current efforts to improve the spatial resolution using off-axial scattering signals.

Gao, Peng – Peking University

Electron Microscopy Laboratory at PKU

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In this talk, we briefly introduce Electron Microscopy Laboratory in Peking University. As a user facility in Peking University, our lab provided service for more than 200 research groups each year. Typically, more than 200 students were trained to independently operate the microscopes every year. There are 6 TEMs and 6 SEM/FIB/SIM in the lab now. In the past two years, the lab was equipped with two aberration corrected microscopes, FEI titan Themis and monochromatic Nion. The Nion machine delivered 6 meV energy resolution and 57 pm spatial resolutions at 60 keV.

Besides EELS, our current research also includes in situ TEM tracking of the solid-state phase transformations with high temporal resolution and quantitative measurements of local structure distortion with picometer-precision using annular bright field imaging. We focus on the surface, heterointerface, dislocations and grain

boundaries of crystal materials. The ongoing systems include the alkali-metal ions battery materials, ferroic materials, and low dimensional quantum materials. More details will be given in the talk.

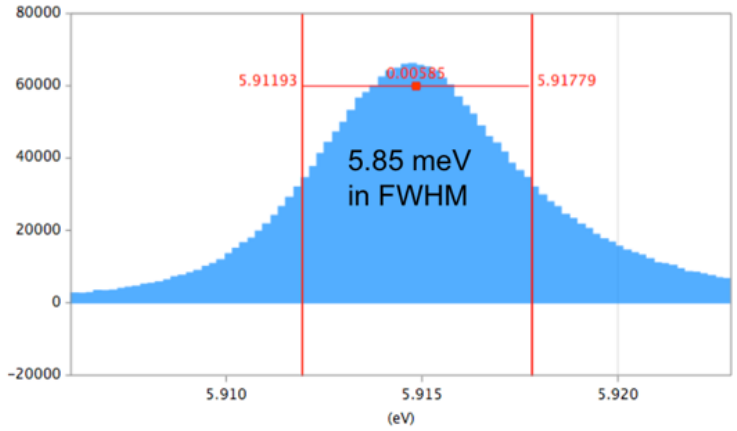

FIG. 1. Low loss EELS measurements.

Gonen, Shane – University of California, San Francisco

Harnessing the potential of Cryo-EM through protein design

Recent advances in electron cryo-microscopy (cryo-EM) have resulted in a proliferation of structures at high resolution without the need for growing crystals. While the limits of cryo-EM are being pushed further and further, a fundamental size limitation still persists when dealing with biological samples that are radiation sensitive. Concurrently, there have been exciting developments in the field of protein design. Computational power has increased exponentially, the cost of DNA is low and our understanding of protein behavior has resulted in many different avenues being influenced through design. In this talk I will describe the design and structural characterization of self-assembling nanomaterials and their applications for cryo-EM.

Gonen, Tamir – University of California, Los Angeles

MicroED: conception, practice and future opportunities

My laboratory studies the structures of membrane proteins that are important in maintaining homeostasis in the brain. Understanding structure (and hence function) requires scientists to build an atomic resolution map of every atom in the protein of interest, that is, an atomic structural model of the protein of interest captured in various functional states. In 2013 we unveiled the method MicroED, electron diffraction of microscopic crystals, and demonstrated that it is feasible to determine high-resolution protein structures by electron crystallography of three-dimensional crystals in an electron cryo-microscope (CryoEM). The CryoEM is used in diffraction mode for structural analysis of proteins of interest using vanishingly small crystals. The crystals are often a billion times smaller in volume than what is normally used for other structural biology methods like x-ray crystallography. In this seminar I will describe the basics of this method, from concept to data collection, analysis and structure determination, and illustrate how samples that were previously unattainable can now be studied by MicroED. I will conclude by highlighting how this new method is helping us understand major brain diseases like Parkinson's disease; helping us discover and design new drugs; shedding new light on chemical synthesis; and showing us unprecedented level of details with sub atomic resolutions.

Idrobo, Juan – Oak Ridge National Laboratory

Towards a new era in imaging and spectroscopy in the STEM: Probing Magnetism, Temperature & Anharmonicity at the Nanoscale

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Electron microscopist for decades have focused in increasing the spatial and energy resolution of the instruments. That focus has resulted in the invention of aberration correctors, monochromator and improved spectrometers. Here, I will argue that improving spatial resolution, which is reflected in smaller electron probes in STEM, is not always the best path to reveal the materials properties. In particular, based on theoretical grounds, in some cases it is even desirable to have a non fully aberration-corrected electron probe [1] .

In this talk, I will discuss how one can use aberrated electron beams to detect magnetic ordering by observing a dichroic signal in the fine structure of an L-edge in a transition metal element for the case of the antiferromagnetic LaMnAsO [2] and $BiFeO_3$.

I will also present our current efforts to study local lattice vibrations using electron energy-loss spectroscopy (EELS). The physical basis for EELS is that a high energy electron beam in a (S)TEM will interact with the electrons or phonons inside the sample, exciting them from lower to higher energy states, while losing a corresponding amount of energy. However, the opposite interaction is also possible: the fast electron can gain energy from a sample that is initially in a higher energy state, albeit with an exponentially smaller probability that depends on the temperature of the sample. Here, I will show that we can directly measure the local temperature of boron nitride flakes in the nano-environment, and probe their anharmonicity using monochromated electron beams [3,4].

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Jensen, Grant – California Institute of Technology

Structural biology in vivo through electron cryotomography

In the last ten years electron cryotomography has made it possible to visualize large macromolecular assemblies inside intact cells in a near-native, "frozen-hydrated" state in 3-D to a few nanometers resolution. Increasingly, atomic models of individual proteins and smaller complexes obtained by X-ray crystallography, NMR spectroscopy, or other methods can be fit into cryotomograms to reveal how the various pieces work together inside cells. A few good pictures is therefore sometimes all that is really needed to distinguish between competing models. To illustrate these points, I will briefly summarize the key technological advances that have made electron cryotomography possible and then present several examples of current results from our recent work in bacterial cell biology, likely including new images and mechanistic insights into secretion systems and the Type IV pilus. Finally, if time permits, I will describe prospects for further improvements, including advances in correlated super-resolution light microscopy and electron cryotomography (CLEM), cryo-FIB-milling, the speed of data collection, and software to segment and analyze tomograms.

Jinschek, Joerg – Ohio State University

Understand dynamic processes and structure-property relationships in nanomaterials using in-situ electron microscopy

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Our efforts in more efficient energy use and conversion, on more efficient transportation, and on environmental protecting technologies relies heavily on the advancement of functional nanomaterials. At any stage in this research and development, studies of these nanomaterials' structure, properties, and function are critical, including detailed atomic-scale insights.
Progress in technology and methodology has made scanning / transmission electron microscopes (S/TEM) powerful and indispensable tools for characterizing nanostructures. However, studies e.g. at room temperature and/or under standard high vacuum conditions might be inadequate to investigate the actual functional state of a material or system, whose properties depend on varying operating or environmental conditions.

Fortunately, in recent years the technology has been significantly advanced to enable in-situ studies while maintaining high-resolution imaging and analytical capabilities when applying in-situ stimuli to functional nanomaterials under (near) operational conditions [1,2], such as temperature, current, gas etc.

Implementation of differential pumping apertures in an aberration corrected TEM (ETEM) enables environmental studies, e.g. oxidation, reduction, or corrosion experiments [1].
Optimized in situ stages - based on MEMS technology – enable accurate realization of experimental in situ conditions, and the opportunity to detect the material's function simultaneously as well (= in operando). Sample preparation in conjunction with MEMS cartridges as sample supports have been adopted [5]. Fine temperature control enables quantitative studies at elevated temperatures [6]. Others have utilized EM methods to measure the actual temperature (gradient) of the specimen precisely [7,8]. Moreover, the integration of a heater into a gas-flow MEMS nanoreactor enables operando EM combining structural characterization of e.g. catalytic materials with simultaneous measurement of its activity for gaseous reactions [9].

These advancements open up for unprecedented experiments of dynamic phenomena in materials science to understand the structure-property-function relationship on the (sub)nanometer length scale.

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Klie, Robert – University of Illinois, Chicago

In-Situ Materials Characterization at High Spatial Resolution: 2D Materials Based Liquid-Cell Microscopy

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Two-dimensional materials, including graphene, BN and transition metal dichalcogenides (TMDs), exhibit great potential for a variety of applications, such as transistors, spintronics, photovoltaics, or even as window layers for in-situ microscopy holders. When 2-dim materials are used in electronic devices, the potential for miniaturization offers remarkable improvements in electrical performance. Yet, the effects of heat dissipation can be a problem in designing electronic devices. Therefore, the thermal properties of 2-dim materials are an important subject of current research and new methods are being developed for temperature measurements at the nanometer scale. Over the last few years, we developed a new approach that now enables us to measure the local temperature distribution and thermal expansion coefficient of 2-dim materials with nanometer resolution.[1] Our nanoscale thermometry measurements are based on the principle that the plasma peak position is sensitive to the density of charge carriers, which changes as a materials expands/contracts as a function of temperature. For 2-dim materials, we have shown that the thermal expansion coefficient is also highly dependent on the number of layers, with monolayer materials exhibiting the higher expansion coefficient.

For TEM in-situ liquid holders based on graphene as a window layer, it was demonstrated that graphene interacts strongly with the contained liquid, which can be beneficial in reducing electron beam induced damage.[2] However, the strong interaction between the reaction species with the graphene window layer can potentially alter the chemistry within the liquid cell and might yield effects are not observed in more conventional reactor setups. Therefore, we have developed new, more inert window layers for liquid cells based on 2-dim materials that will enable in-situ characterization of fluids at unprecedented spatial and energy resolution. The electron-beam induced change in the pH of liquids will be demonstrated using the dissolution and precipitation of calcium hydroxyapatite in a liquid cell based on 2-dim material. The benefits monochromated STEM on the characterization of liquids encapsulated by 2-dim materials will also be discussed. In particular, we will discuss the possibility of using such cells for vibrational EELS experiments.

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Lovejoy, Tracy - Nion

Ultra-High Energy and Spatial Resolution STEM-EELS

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EELS in the electron microscope has come a long way in the last 5 years. The best energy resolution reached prior to 2013 was about 40 meV in a 30 msec exposure (full-width at half-maximum of the zero loss peak (ZLP) at 200 kV) and the ZLP "tail" intensity was about 1/1000 of the ZLP maximum at an energy loss of 300 meV [1]. The introduction of the Nion ground-potential monochromator in 2013 improved the energy resolution to 16 meV in a 55 msec exposure (at 60 kV), a level that allowed vibrational (phonon) signals to be explored in the electron microscope [2]. The introduction of the Nion EEL spectrometer in 2017 improved the resolution to 6 meV in a 100 msec exposure at 60 keV, and the ZLP tail has been reduced to the 1/1000 level at 40 meV loss. The performance is still improving as we optimize the operation of the whole monochromated STEM system further.

This progress is largely due to focussing on stability, which comes naturally to a design team used to producing <1 Å STEM probes and keeping them stationary to <0.1 Å. Our design has:

a) Eliminated the deleterious effects of high tension (HT) instabilities, by putting the monochromator at ground potential — HT instability does not affect the energy resolution.

b) Eliminated the deleterious effects of instabilities in the prism current by connecting the monochromator prisms plus the spectrometer prism in series.
c) Improved the HT stability by using the MC's energy-selecting slit in an HT feedback loop.
d) Improved the stability of the EELS by a careful design of its multipoles, avoiding schemes that lead to substantial dipole-like instabilities that randomly displace the spectrum.
e) Decreased the sensitivity of the system to stray magnetic fields and random mechanical vibrations, by putting thorough mu-metal shielding around the monochromator and the EELS, and by strengthening the EELS with robust anti-vibration bracing.

Another key ingredient was designing the key constituents of the whole system – the electron gun, the monochromator, the aberration-corrected probe-forming optics, the spectrometer coupling optics, and the spectrometer – in a consistent way that emphasizes high-order aberration correction in addition to stability. The electron source is imaged in many different planes including the monochromator slit, the sample-level probe, and the EELS ZLP. At each of these places, higher performance (better energy or spatial resolution) becomes possible by minimizing the size of the image, and problems appearing in one place affect the rest of the system.

A world of experimental possibilities that has been opened up by this new performance level. Recent examples of applications include momentum-resolved vibrational spectroscopy that provides similar information to neutron scattering and inelastic X-ray spectroscopy, but from much smaller volumes, measuring the temperature of small sample areas by energy gain/energy loss spectroscopy, and vibrational spectroscopy of water that detected isotopic substitution. These and other examples will be presented in the talk.

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Ma, Xiuliang – University of Chinese Academy of Sciences

Atomic mapping of domains and interfacial structures in ferroelectric films

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In this presentation I will briefly introduce some representative studies on transmission electron microscopy of materials at IMR/Shenyang in the past three decades [1-3]. I will show more details on our recent studies of atomic mapping of domains and interfacial structures in ferroelectric thin films [3-12].

$PbTiO_3$ is a ferroelectric with a tetragonal structure. We have grown a number of $PbTiO_3$ and $BiFeO_3$ film samples on various substrates by pulsed laser deposition. We perform large-scale strain analysis by employing the combinations of geometrical phase analysis and aberration-corrected scanning transmission electron microscopic imaging, which makes the investigation of ferroelectric domain structures accurate and straightforward [4]. We observe the as-grown epitaxial films in which the electric dipoles at domain-walls and interfaces are characterized by means of aberration-corrected scanning transmission electron microscopy [3-11]. In addition, we identify a giant linear strain gradient with extremely low elastic energy in a perovskite $BiFeO_3$ nanostructures array grown on $LaAlO_3$ substrate via a high deposition flux [12]. The present strain gradient, resulting from the disclinations in the $BiFeO_3$ nanostructures array, induces a polarization of several microcoulomb per square centimetre. It leads to a large built-in electric field of several megavoltage per metre, and gives rise to a large enhancement of solar absorption.

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Miao, Jianwei – University of California, Los Angeles

Atomic Electron Tomography: Probing 3D Structure and Material Properties at the Single-Atom Level

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To understand material properties and functionality at the most fundamental level, one must know the 3D positions of atoms with high precision. For crystalline materials, crystallography has provided this information since the pioneering work of Max von Laue, William Henry Bragg, and William Lawrence Bragg over a century ago. However, perfect crystals are rare in nature. Real materials often contain crystal defects, surface reconstructions, nanoscale heterogeneities, and disorders, which strongly influence material properties and performance. Here, I present atomic electron tomography (AET) for 3D structure determination of crystal defects and disordered materials at the single-atom level (1). Using a Fourier based iterative algorithm, we first demonstrated electron tomography at 2.4-Å resolution without assumption of crystallinity in 2012 (2). We then applied AET to image the 3D structure of grain boundaries and stacking faults and the 3D core structure of edge and screw dislocations at atomic resolution (3). Furthermore, in combination of AET and atom tracing algorithms, we localized the coordinates of individual atoms and point defects in materials with a 3D precision of ~19 pm, allowing direct measurements of 3D atomic displacements and the full strain tensor (4). More recently, we determined the 3D coordinates of 6,569 Fe and 16,627 Pt atoms in an FePt nanoparticle, and correlated chemical order/disorder and crystal defects with material properties at the individual atomic level (5). We identified rich structural variety with unprecedented 3D detail including atomic composition, grain boundaries, anti-phase boundaries, anti-site point defects and swap defects. We showed that the experimentally measured coordinates and chemical species with 22 pm precision can be used as direct input for density functional theory calculations of material properties such as atomic spin and orbital magnetic moments and local magnetocrystalline anisotropy (5). Looking forward, AET will not only advance our ability in 3D atomic structure determination of crystal defects and disordered materials, but also transform our understanding of materials properties and functionality at the fundamental level.

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Minor, Andrew – Lawrence Berkeley National Laboratory

New modes of imaging for in situ TEM nanomechanical testing

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There is an ongoing revolution in the development of electron detector technology that has enabled modes of electron microscopy imaging that had only before been theorized. The age of electron microscopy as a tool for imaging is quickly giving way to a new frontier of multidimensional datasets to be mined. This talk will highlight recent advances with in situ Transmission Electron Microscopy (TEM) nanomechanical testing and imaging techniques that provide insight into small-scale plasticity and the evolution of defect structures in materials. In addition to measuring the strength of small-volumes, measuring the evolution of strain during plastic deformation is of great importance for correlating the defect structure with material properties. Here we demonstrate that strain mapping can be carried out during in-situ deformation in a TEM with the precision of a few nanometers without stopping the experiment. This talk will describe our recent results from in situ TEM nanomechanical testing that provide insight into multiscale metallurgical phenomena using these techniques, such as the role of short range ordering in Ti alloys and deformation phenomena in metallic glasses.

Pan, Xiaoqing – University of California, Irvine

Probing the Atomic Structure and Dynamic Behaviors of Materials by *In-situ* Transmission Electron Microscopy

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As advances in aberration-corrected transmission electron microscopy (TEM), the development of *in situ* techniques allows us to study the dynamic evolution of materials in response to applied fields and to changes in environments. In this talk, I will present our work on the development of *in-situ* TEM techniques for imaging electric polarization, probing the nucleation and growth of ferroelectric domains during electric polarization switching, and observing the dynamic reaction and structural evolution of catalysts under realistic conditions with atomic precision through a MEMS-based, electron-transparent closed cell with a heating stage.

Pennycook, Stephen – National University of Singapore

New insights into materials with the NUS ARM

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The National University of Singapore installed a JEOL ARM200F last year, incorporating a cold field emission gun, ASCOR aberration corrector, UHR pole piece, Gatan Quantum ER spectrometer, OneView camera and Oxford Aztec EDS system. It is installed within a JEOL environmentally controlled room and shows sub-Ångstrom information transfer even at 40 kV accelerating voltage (see Fig. 1). Examples will be shown of new edge structures in nanoporous MoS_2 with good catalytic properties [1], the atom-by-atom fabrication of monolayer Mo membranes via beam induced sputtering, and beam-induced healing of holes.

In piezoelectrics, precise mapping of atomic displacements reveals a hierarchical nanodomain structure as the origin of excellent properties [2,3]. Similarly, in thermoelectrics, a hierarchical structure ranging from point defects through nanoscale and microscale precipitates is observed in a high-performance material with lattice thermal conductivity approaching the theoretical minimum.

The ASCOR corrector can provide probe semi-angles up to 60 mrad which allows depth resolution at the nm-scale. In a TiO_2 film grown on $LaAlO_3$, La is seen to have preferentially diffused along stacking fault intersections originating at an interface step.

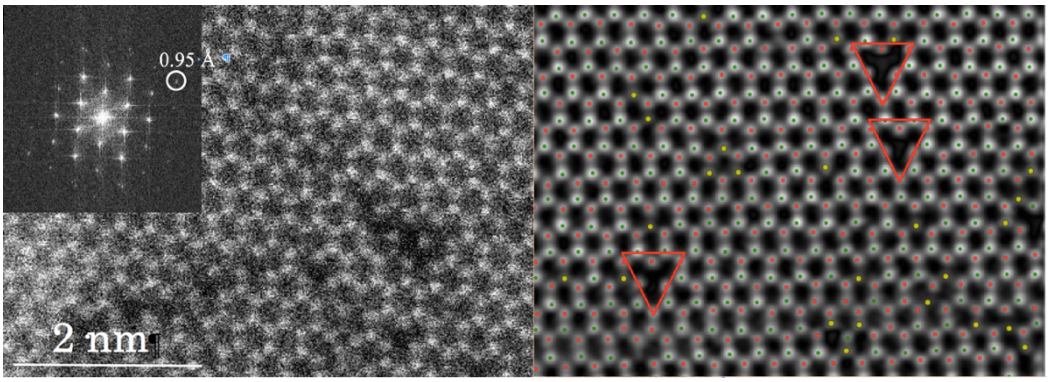

FIG. 1. Left: STEM image of $MoSe_2$ obtained at 40 kV showing information transfer to 0.95 Å (inset fast Fourier transform). Right: A filtered image (not the same sample) showing identification of Mo (red), Se_2 (green), Se (yellow, single vacancy), and Se divacancies (red triangles). Data taken on a JEOL ARM200F with ASCOR corrector at 40 kV, courtesy Xiaoxu Zhao and Jiadong Dan.

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Rodriguez, Jose – University of California, Los Angeles

Nanoscale mapping of lattice structure in amyloid nanocrystals by cryo electron diffraction

Imperfections in protein crystals are challenging to assess with approaches that rely on whole crystal measurements. Accordingly, our determination of structures from protein nanocrystals relies on estimates of crystallinity, including the size of domain blocks. My group has now shown that amyloid nanocrystals exhibit lattice deformations that are perceptible at the nanoscale and span large areas of single crystals. We measure 1.2Å resolution diffraction from as few as 2500 diffracting molecules and record patterns that are sensitive to crystal thickness and descriptive of three-dimensional lattice orientation. Real-space maps obtained by unbiased clustering of two-dimensional diffraction scans reveal regions of varying absorption, order/disorder, and three-dimensional lattice reorientation on a 10-20nm scale. The high plasticity observed in prion

nanocrystals mirrors deformations estimated in larger protein crystals and suggests an inhomogeneous lattice structure is present within peptide nanocrystals. Knowledge of these features could better our understanding of macromolecular self-assembly and improve the determination of macromolecular structures from nano and microcrystals by emerging crystallographic approaches.

Sawada, Hidetaka – JEOL

Enhancement of image resolution and analytical sensitivity by latest technology in STEM and TEM

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Latest technology in high-end atomic-resolution electron microscope has enhanced the resolution, analytical capability, and field observation in STEM and TEM with a small energy spread of electron source by monochromater, a large convergence angle, and high sensitivity EDS (Energy Dispersive X-ray Spectrometry) by large solid angle.

The developed microscope is equipped with an ultra-stable cold field emission gun (CFEG) and spherical aberration correctors for probe and/or image forming systems. The developed CFEG is evacuated with NEG (non-evaporative getters), a SIP (with noble pump) and two-intermediate SIPs, resulting in ultimate current stability of the CFEG. Observation at 200 kV and 300kV can image a sub-angstrom structure, and low-kV imaging of 30-80 kV imaging is useful for soft materials to reduce the specimen damage. The instruments have demonstrated sub-angstrom resolution, showing that ultra-high resolution of sub-50 pm information was detected in the STEM DF image at 300kV. We demonstrate experimental application results of sub-angstrom imaging using spherical aberration corrector, low kv imaging, and atomic resolution elemental mapping using EELS (Electron Energy-Loss Spectroscopy) and EDS. In addition to atomic resolution EELS, a super-high sensitivity EDS system with large solid angle has been developed by optimizing the shape of the objective lens pole piece and detector positions for a larger detection area of the detectors. Several application data of the high-resolution and EDS analysis performance with aberration corrected electron microscope are demonstrated. The system enhances acquisition throughput for the elemental and chemical information.

Scheu, Christina – Max Planck Institut fur Eisenforschung

Combining ultimate resolution: Cs corrected STEM and 3D atom probe tomography

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Correlating atom probe tomography (APT) with transmission electron microscopy (TEM) has been performed in literature, typically using conventional TEM micrographs for defect analysis. In that way, dislocations or grain boundaries have been identified within atom probe needles and the 3D reconstructed tomograms used to determine e.g. segregation at 1D or 2D defects. Only few examples exist where APT has been coupled with high resolution STEM work. In our work we were interested to determine whether a quantitative analysis of e.g. strain state and coherency of an interface can be directly obtained from APT needles using aberration-corrected STEM prior to the field evaporation. A specific region of interest was analyzed first in an Cs corrected STEM, the data analyzed in depth by e.g. nearest neighbor analysis or comparison with reference lattices and

subsequently investigated by 3D APT. Identical locations were studied which allowed to combine both – the superior spatial resolution of a STEM with the excellent chemical analysis of the APT. Compared to energy dispersive X-ray spectroscopy or electron energy loss spectroscopy (EELS), the time-of-flight detector within the APT allows to detect light elements like hydrogen and ppm segregation to defects which would be not detectable by the STEM based methods. On the other side, EELS gives the possibility to study e.g. bonding behavior or oxidation states of individual atoms which are not accessible by APT. In the presentation, several examples will be discussed including the interface structure and strain state between nanometer-sized carbides in an austenitic steel matrix [1] to grain boundaries in silicon [2].

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Schoenung, Julie – University of California, Irvine

Application of Advanced Microscopy Techniques to Reveal Characteristics of Ceramic/Metal Interfaces

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The microstructure and adhesion of heterophase interfaces is vital information in the design of structural composites, electroceramic devices, and environmental coatings, but has not yet been well understood due to the complexion in the heterophase interfaces, such as the presence of various types of atomic-scale elemental segregation layers and lattice distorted layers. When equipped with both high-speed cameras and holders with mechanical test units, transmission electron microscopy (TEM) has the capability to in-situ observe microstructure evolution over time under externally applied stress conditions and thus becomes a very promising microscopic tool to probe adhesion behavior of heterophase interfaces.

Here, we utilized advanced microscopy techniques to understand the interfacial behavior from two aspects. First, we studied the influence of length scale on the spatial distribution and characteristics of ceramic/metal interfaces in nanostructured composites. Specifically, the characteristics of the B_4C/Al interface, namely the local chemistry and interfacial structure, were studied in detail using TEM and atom-probe tomography. Results reveal significant differences in these characteristics as a function of particle length scale. Mechanisms related to length scale effects on the formation of the corresponding structures and chemistries in the interfaces are discussed. Second, we implemented an in-situ TEM based approach to isolate an individual ceramic/metal interface and to evaluate its adhesion behavior at the nanoscale. A Hysitron PI-95 picoindenter was used to carry out in-situ TEM adhesion tests, for which a push-to-pull (PTP) device was utilized to transform the compression loading into a tensile force. The goal was to understand not only effects of "intrinsic microstructural features," but also of "extrinsic sample size" on the nanomechanical response at a ceramic/metal interface. The results indicate that ceramic/metal interfaces at the nanoscale are inherently tough and ductile, even though the metallic phase may be polycrystalline, and irrespective of whether the interfaces are subject to local segregation and chemical variation.

Shibata, Naoya – University of Tokyo

Aberration-corrected DPC STEM for materials research

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Due to the rapid progresses in segmented/pixelated detector developments in scanning transmission electron microscopy (STEM), differential phase contrast (DPC) imaging is becoming very powerful method to directly visualize electromagnetic field structures within materials and devices. It has been demonstrated that, by using DPC STEM for atomic-resolution observation, we can directly visualize atomic electric field, the field between positively charged atomic nucleus and negatively charged surrounding electron clouds [1-3]. Figure 1 show simultaneous (A) annular dark field (ADF) image, (B) electric field vector color map and (C) electric field strength map of $SrTiO_3$ crystal observed along [001] direction constructed from DPC STEM [3]. The direction of rotating color contrast is the same in all the atomic columns irrespective of the atomic species, visualizing that the (projected) atomic electric field points outward from the center of the atomic columns. In this talk, the current status of aberration-corrected DPC STEM along with some applications in materials studies such as pn junction in semiconductor devices and magnetic skyrmion in helimagnets [4] will be reported. New developments for atomic-resolution DPC STEM under magnetic field free condition will be also reported.

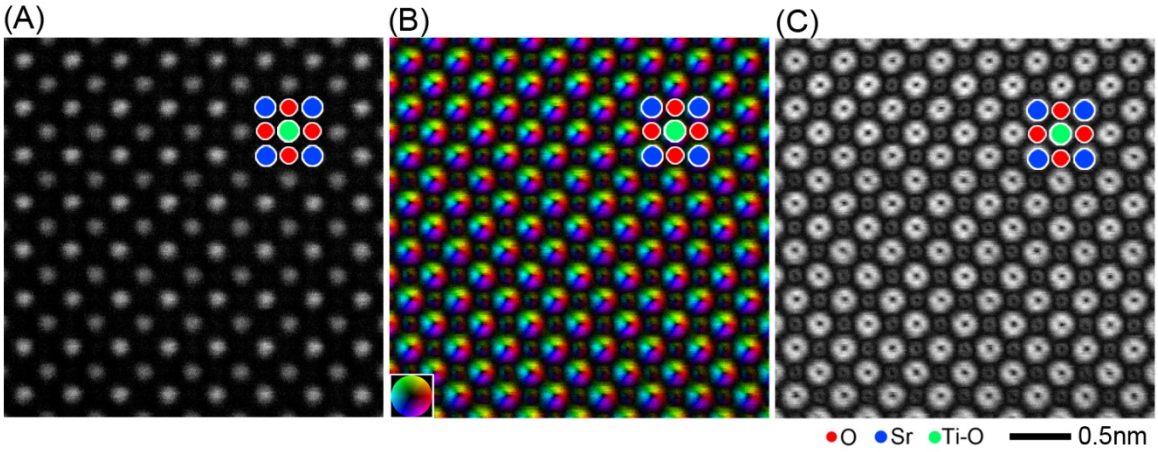

Fig. 1: (A) ADF image. (B) Projected electric field vector color map and (C) electric field strength map constructed from the segmented detector DPC images [3]. The inset color wheel indicates how color and shade denote the electric field orientation and strength in the vector color map.

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Sinclair, Bob – Stanford University

In Situ High Resolution and Environmental Electron Microscopy Studies of Material Reactions

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There has been a steady growth in the applications and breadth of *in situ* transmission electron microscopy (TEM) since the 1980's [1]. At that time, the procedures to carry out meaningful experiments were described (e.g. [2]) but it was thought that high voltage TEM and thick specimens were required to reproduce bulk behavior. However, in a series of studies, we established that this was not necessarily the case and that high resolution TEM recordings could be made in real time, *in situ* and that the atomic behavior associated with materials reactions at interfaces could be deduced (e.g. [3],[4]). Moreover, with the advent of thin film and nanotechnology, the investigation of thin and nano-scale materials became a necessity (e.g. [5]). In recent years, there has been an additional proliferation, most notably from *in situ* TEM in controlled environments such as in gases and liquids (e.g. [1], [6]), and from the application of aberration-corrected TEM (e.g. [7]).

This paper gives a review of the application of *in situ* high resolution TEM to investigate material reactions. An overarching theme of our work has been to ensure that the *in situ* studies are truly representative of the real behavior of the material system, and we have advanced a number of guidelines to ensure this. Moreover, we have also expanded our approach to environmental material-gas reactions such as carbon nanotube (CNT) oxidation [8], and the in situ study of CNT field emission for high intensity X-ray sources [9]. The influence of the imaging electron beam is more important for the gaseous reactions, as it ionizes the reacting gas species, and it is necessary to develop protocols to take this into account [10]. The procedures we have adopted to do this will be carefully described [11].

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Stach, Eric – University of Pennsylvania

Using Operando Characterization, Data Analytics, and Artificial Intelligence to Understand Mechanistic Links between Processing and Structure

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In this presentation, we will describe a vision for a future research paradigm, wherein a tight coupling of in-situ and operando experimental methods, data analytics and automated data analysis are coupled with artificial intelligence to direct how we use electron microscopy (and other forms of materials characterization) to characterize the mechanisms by which processing/structure/property relationships are determined. The presentation will be forward looking and will incorporate research results and ideas culled from a variety of sources and authors: it will not be a typical presentation reviewing research from just the co-authors.

First, we will describe the motivations for working towards this type of research paradigm. These

include the desire to speed up the rate of scientific discovery and time to market, as well as a more pedestrian desire to maximize the utilization of expensive instrument time.

Second, we will review examples of autonomous research methods, both through data mining of the literature [1,2], and through the use of real time feedback and artificial intelligence methods to direct experimental outcomes.[3] Specifically, we will discuss how these approaches may be utilized in electron microscopy research in the near future, and the developments needed to bring this to reality.

Third, we will describe how this approach can be used to explicitly and efficiently test operative hypotheses, and to efficiently understand the relevant experimental parameter space. This yields insight as to where detailed experimentation is most valuable. In this portion of the talk, the need for operando methods and correlative experimentation will be emphasized.[4]

Finally, we will discuss this evolving research paradigm as it exists both within and provides challenges to established theories of scientific discovery.[5,6]

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Stemmer, Susanne – University of California, Santa Barbara

Understanding Point Defects in Complex Oxides Using Quantitative STEM

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While methods to measure the global concentration of point defects exist, determining their spatial arrangement is significantly more challenging. Furthermore, most techniques cannot provide direct information about atom relaxations around a point defect even though these are crucial for many properties. HAADF-STEM images can provide three-dimensional information of the location of individual dopant atoms in $SrTiO_3$ from a single image. The contrast and interpretability can be improved using variable-angle HAADF-STEM (VA-HAADF). Detecting vacancies in STEM images is even more challenging than dopant atoms that have a large atomic number difference with the host. We have combined VA-HAADF and rigid registration methods to detect Sr vacancies in $SrTiO_3$ and their associated local atom relaxations. Lattice relaxation around the vacancies are detected with picometer precision. Rigid registration methods not only improve the precision in measurements of atom column positions but also the quantification of image intensities, which allows for the detection of point defects with low Z-contrast, such as vacancies. We also investigate atomic-scale structural relaxations upon doping a prototype Mott insulating material, $SmTiO_3$, with Sr. As the Sr content increases, the orthorhombic distortions gradually decrease. Measurements of the atomic column positions provide direct evidence of continuous and homogeneous structural changes with no signs of phase separation. The detailed structural information with high spatial resolution and precision and provides an important step toward a complete understanding of unconventional physical phenomena near Mott transitions.

Taheri, Mitra – Drexel University

Implications of Direct Detection Electron Energy-loss Spectroscopy (DD-EELS) for In Situ TEM: Challenges and Opportunities

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In situ transmission electron microscopy (TEM) and electron energy-loss spectroscopy (EELS) are powerful tools for the observation of real-time materials processes. The development of radiation hard direct detection (DD) electron sensors has enabled improvements in the quality of in situ data for TEM imaging, and recently, we have demonstrated that DD provides far-reaching benefits for EELS [1, 2]. Specifically, the sharper point spread function and reduced pixel size of DD provides a significant improvement in combined energy resolution and field of view (FOV). This combined resolution/FOV facilitates methodologies such as simultaneous elemental mapping (large FOV) and oxidation state analysis (high resolution). Additionally, the improved detective quantum efficiency (DQE) offered by electron counting greatly reduces spectrum noise, which has benefits for low-dose applications and time-resolved in situ studies.

Using the combined energy resolution and FOV and increased spectrum SNR offered by the DD EELS system, critical issues in both physical and biological sciences can be addressed. Additionally, dynamic ionic or defect-dependent phenomena can be quantified with the combination of in situ methods and the DD EELS system. This talk reviews some of our group's recent work with DD EELS in a wide variety of systems, including in situ investigation of chemistry-property relationships in a rapidly emerging family of 2D materials, MXenes, that show for energy storage and electromagnetic interference shielding; scattering and local structure of high entropy and other candidate metallic systems under radiation damage; dynamic semiconducting device failure, and finally, cervical tissue remodeling. These experiments enable new understanding of complex materials systems, including the first direct correlation of MXene surface terminations and conductivity, providing a predictive understanding of MXene properties for device development, as well as improved sensitivity in EFTEM imaging of biological tissues and the promise for low dose studies necessary for biological and soft matter applications.

In addition to these research topics discussed, an outlook for DD EELS in the context of emerging microscopy techniques and platforms (both in situ and ex situ) will be discussed. Finally, key challenges for high-throughput data analysis and artificial intelligence methodologies with next generation detection systems yielding more data at faster rates (over 400 frame/second acquisition).

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Twesten, Ray – Gatan

Electron counting detectors for imaging and spectroscopy

Ray D. Twesten, Gatan Inc.

The incredible improvements in instrumentation for electron microscopy over the last decade have opened new windows into the nanoscale world. Beyond simply making smaller, brighter electron probes, recent efforts have pushed the boundaries in other directions. Driving down the electron energy spread, creating beams in defined momentum states and pushing the limits of temporal scales have all but redefined the meaning of electron microscopy itself.
Complementary to these advancements in electron sources are the detector improvements required to support these unique applications. Focusing just on forward scatted electrons, the ultimate detector for the electron microscope would allow the collection of every transmitted electron while simultaneously recording its energy and momentum with resolution limited only by the uncertainty principle. While we are still a long way off

from this goal, strides have been made to drive up the collection and detection efficiency of electron detectors while at the same time pushing the energy resolution, stability and time resolution to match the source advances.
In this presentation, we will review the current state of detectors for electron microscopy with an emphasis on systems for beam sensitive materials and EELS measurements. By comparison with the ideal, we will explore how close we are getting to the ideal and what strides are being made to close the performance gap.

Villa, Elizabeth – University of California, San Diego

Opening Windows into the Cell: Opening Windows into the Cell: Bringing Structure to Cell Biology with Cryo- Electron Tomography

To perform their function, biological systems need to operate across multiple scales. Current techniques in structural and cellular biology lack either the resolution or the context to observe the structure of individual biomolecules in their natural environment, and are often hindered by artifacts. Our goal is to build tools that can reveal molecular structures in their native cellular environment. Using the power of cryo-electron tomography (CET) to image biomolecules at molecular resolution in situ, we are building tools to make compatible with, and directly comparable to, biophysical and cell biology experiments, capturing the structural behavior of macromolecules in action under controlled conditions. I will show how we use these techniques to study the molecular of the nuclear periphery, to understand Parkinson's disease at the molecular level, and to peek on the inner life of bacteria.

Ward, Andrew – Scripps Institute

Structure-based vaccine design

The HIV-1 envelope glycoprotein trimer on the surface of the virion is the target for all broadly neutralizing antibodies (bnAbs). Various soluble Env trimer reagents that structurally and antigenically mimic native Env have now been deployed and tested in animal immunization studies. We use these soluble Env trimers form complexes and solve structures of broadly neutralizing antibody (bnAb) lineages from infected patients, as well as neutralizing and non-neutralizing antibody complexes elicited via vaccination. In solving these complex structures, we aim to understand the structural basis of productive immune responses in humans and compare them to vaccination experiments in different animal models. We have developed several electron microscopic approaches to deal with complexity of both Env and polyclonal immune responses to large, glycosylated antigens that improve throughput and further enrich our understanding of immune responses. The structural information that we generate is then used for immunogen design and re-design, with the goal of programming immunity in a rational manner.

Wang, Peng – Nanjing University

Electron Ptychography

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Transmission electron microscopy (TEM) is a powerful tool for material science characterization due to its high spatial resolution. Coherent diffraction imaging (CDI) [1] is a "lensless" method that forms an image of an object by solving the phase problem from a single diffraction pattern with an iterative phasing algorithm. This approach can, in principle, overcome the current image resolution limiting factors and ultimately achieve wavelength-limited resolution. However, conventional CDI experiments require isolated samples and a priori knowledge about the sample shape or extent. Maiden and Rodenburg suggested an extended ptychographical iterative engine (ePIE), which does not need this prior information and overcomes many of the other issues of CDI, such as convergence stagnation and limited field of view. Recently, two-dimensional (2D) ptychographical reconstructions at atomic lateral resolution [2] have been achieved using ePIE [3] on a CeO_2 nanoparticle. However, when the sample becomes thicker, the multiplicative assumption of electron-sample interaction becomes invalid causing the ptychographical reconstruction to breakdown. To overcome this limitation, Maiden et al. incorporated the well-known multiple slice solution to multiple scattering into the ePIE algorithm (3PIE) [4]. In this approach the sample is split into axial sections, to produce 3D optically sectioned images of the sample. This 3D multiple-section reconstruction of ptychography has been successfully demonstrated with both light [4] and X-ray [5] optics recently. However, this method is still a challenge for use with the electrons. In this paper, we will firstly review our previous work on the capabilities of defocused probe ptychography to achieve a 2D phase reconstruction of a nanocrystal at sub-Å resolution [6] and a 3D reconstruction of nanostructured materials [7] using traditional CCD camera. Subsequently we will show results from focused and defocused electron ptychography using a fast direct electron detector to reconstruct the wavefunction of various low dimensional materials under different low dose conditions.

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Xin, Huolin – Brookhaven National Laboratory

Artificially Intelligent Scanning Transmission Electron Microscopy

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Artificially Intelligent-Scanning Transmission Electron Microscopy (AI-STEM) is an emerging area that encapsulates automated sample loading, STEM data acquisition, data selection, analysis, and reporting. The purpose of this is to increase throughput, achieve statistics, and remove human bias from the data acquisition/analysis process. The emerging field of deep learning introduces the potential to realize the "self-driving" characterization paradigm--a step toward unsupervised data acquisition and analysis through the use of convolutional neural networks.

In this talk, I will focus on discussing deep learning based image restoration and atom segmentation for high-resolution, low-dose, background-complex STEM image streams. The challenge in image restoration and atom segmentation for an arbitrary ADF-STEM image lies in five dominant unpredictable factors: 1) shot and scan noise, 2) thickness variation and background, 3) imaging condition, 4) crystal structure and zone axis, and 5) field of view. For our network to learn how to recognize atomic columns in all different possible scenarios, we have built a library of 15,000 atomic resolution ADF-STEM images by randomly combining different crystal structure, zone axe, field of view, imaging condition, thickness variation, background pattern, and shot/scan noise level.

These images are synthesized by computer algorithms so we know exactly where the atomic columns are, i.e. ground truth. This synthetic approach allows us to easily scale up and augment the training library to prevent the over-fitting of our neural networks. Apart from 2D projection imaging, in this talk, I will discuss using deep learning to reduce noise and missing-edge artefacts for 3D electron tomography datasets.

Xu, Qiang – DENS Solutions

Correlative imaging of catalysts at working state

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Conventional transmission electron microscopy (CTEM) has been widely used for characterizing the microstructure of the catalyst particles before and after the reaction. Knowledge of particle size distribution, chemical composition, crystal, atom and electronic structure, greatly helps us to understand the structure information of the catalyst at its stable states [1]. However, as the working structure of a catalyst is often a transient, metastable form, and highly dependent to the reaction environment, to fully understand the catalytic mechanism catalysts are needed to be studied during the catalytic reaction. This is not possible in CTEM, due to the high vacuum environment and room temperature limitation of CTEM.

Using the latest nano-reactor technology [2], we have realized the high-pressure gas and high-temperature environment (10 ^ 5 Pa, 1000 ° C) required for chemical synthesis and catalyst reaction inside the electron microscope maintaining the characterization power of TEM. The entire workflow of catalysis, starting from preparation/synthesis of the catalyst, evaluation of the reaction activity and characterization of the real-time structure dynamics can be observed directly in TEM at atomic level. Furthermore, in order to improve the correlation between in situ TEM imaging to chemical relevant properties, e.g. converge rate, activities, etc., mass spectrometry and calorimetry are used to get correlative information.

In this work, we studied the activity of palladium nanoparticles during the catalytic oxidation of methane and other gases using a FEI Titan TEM operated at 300 kV combined with a DENSsolutions Climate in situ gas & heating system. Carbon monoxide and methane, in different ratios, were used as reactive gases (Fig. 1, upper graph). The structure changes of palladium nanoparticles during the catalytic reaction were recorded using in situ STEM (Fig. 1, STEM images). Simultaneously, the catalyst performance was analyzed by monitoring the reaction products (CO2, CO) using the mass spectrometer (Fig. 1, lower graph). During the experiments, the changes in CO2 and CO product content while decreasing the oxygen composition were clearly visible. This indicates a decrease in the activity of the catalytic reaction, which corresponds well with the in situ STEM movie. Furthermore, chemical analysis via mass spectrometry and physical analysis via TEM imaging were combined with in situ calorimetry using power data from the MEMS-based heating device (sample carrier). It was observed that at certain experimental conditions the catalytic reaction exhibits oscillatory behavior that are strongly sensitive to the chemical environment, like methane concentration. (Fig. 2). The correlation between TEM data, chemical analysis of the catalytic reaction, gas composition, temperature and calorimetric data provides an intuitive tool helping us to probe the structure-property relationship of catalytic particles in the reactive environment, which can be easily applied to other heterogeneous catalytic reactions.

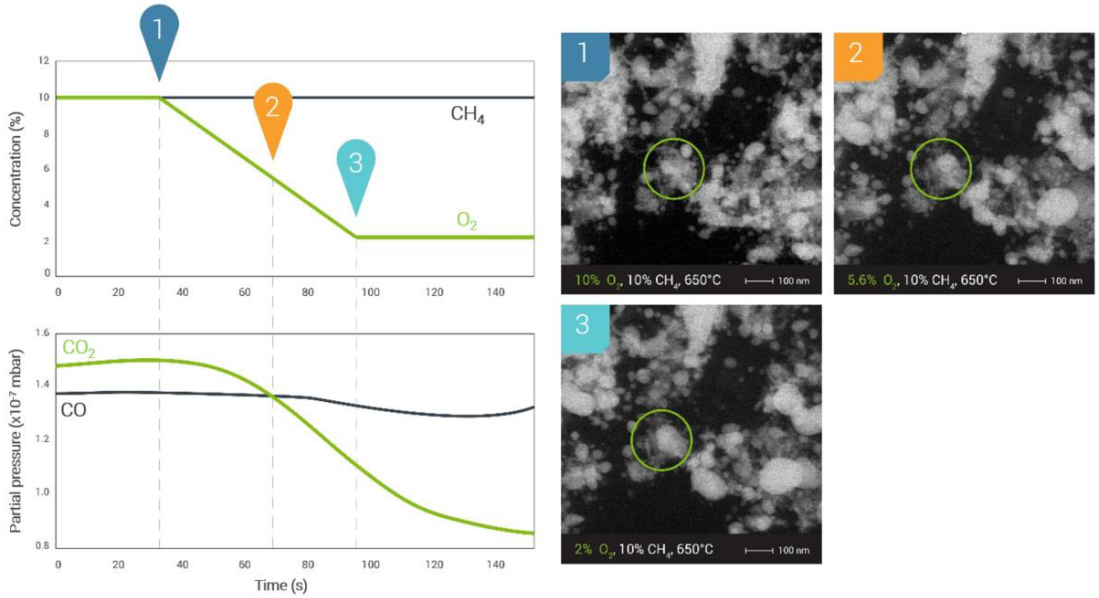

Figure 1: Upper graph shows the applied gas conditions during the in situ experiment, where we lowered the O2 concentration from 10% to 2%. Simultaneously, the mass spectrometry data in the lower graph shows a decrease in CO2 and CO reaction products. STEM images on the right (snapshots from the in situ STEM movie) correspond with the different conditions, showing changes in the morphology of the palladium catalyst (encircled region).

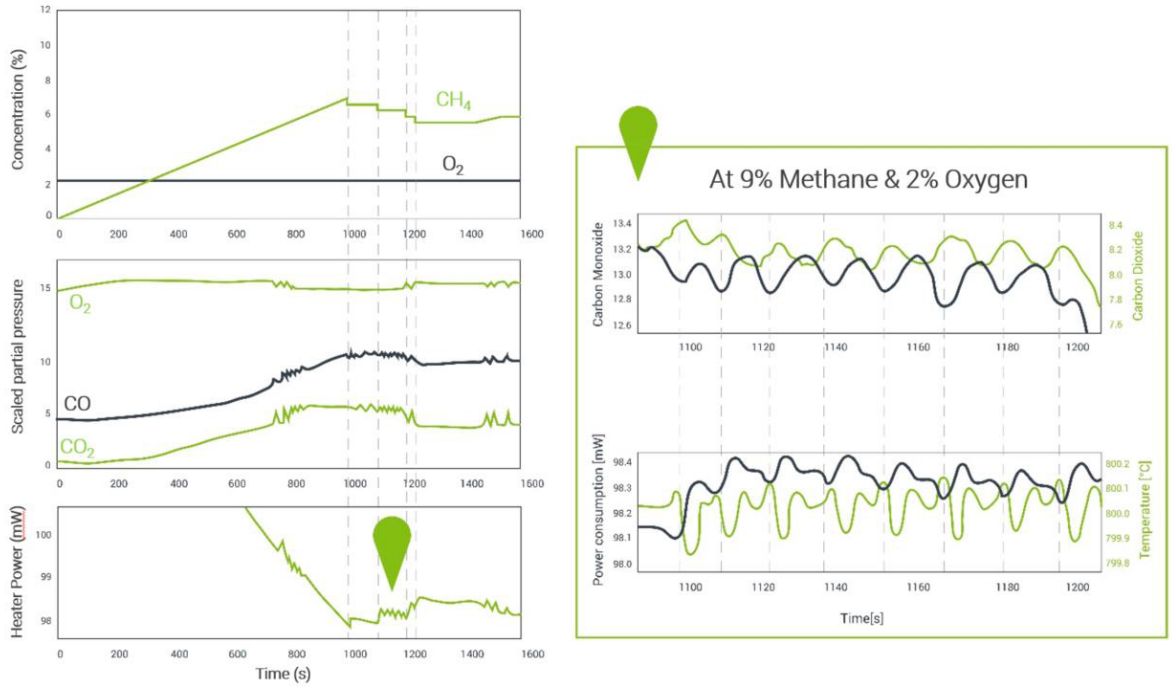

Figure 2. Top left graph shows the input reactant gas composition, with an increase ramp in CH4 concentration from 0% to 10% while O2 was kept stable at 2%. Middle graph shows the corresponding mass spectrometer signal, where oscillations are visible, showing that the catalytic reaction exhibits oscillatory behavior between a high- and low-activity phase. The oscillations observed in the mass spectrometer signal correspond with spikes in the MEMS heater power data (lower graph). Inset graphs on the right show the high temporal resolution of the mass spectrometer and corresponding MEMS power data, of the oscillations which occur at 9% CH4 and 2% O2.

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Zheng, Haimei – Lawrence Berkeley National Laboratory

Materials Transformations in Liquid and Gas Environment

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Materials transform, very often in responses to an external stimulus mediated by their liquid or gas environment. An atomic level understanding of the materials transformations in liquids and gases is significant for the materials applications in many functional devices. With in situ liquid (or gas) environmental transmission electron microscopy (TEM), we study a variety of nanostructured materials with a focus on solid-liquid or solid(liquid)-gas interfaces. In this talk, I will first show our recent liquid cell TEM experiments with the controlled reactions. In particular, it is a great challenge to study air sensitive and electron beam sensitive materials, such as PbSe nanocrystals during self-assembly, with liquid cell TEM. With systematic study and control, we have been able to overcome the issue and achieve in situ atomic scale imaging of PbSe nanoparticle interactions and self-assembly during ligand exchange. We have also been using gas environmental TEM to study catalytic nanoparticles and thin films during oxidation and reduction reactions. I will present the selective oxidation of Rh nanostructured thin films, as an example. Using in situ TEM in combination with the advanced electron microscopy techniques on data collection and image processing, we have been able to achieve an unprecedented level of detail on Rh nanograin oxidation.

Zhou, Wu – University of Chinese Academy of Sciences

Interplay of strain and lattice structure in novel 2D semiconductors

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Two-dimensional (2D) materials have attracted intense research efforts in the past decade due to many unique properties and promising applications of these atomically thin layered materials. Their physical properties can be further tailored by lattice strain, which provides new means to create new functionalities in 2D materials via strain engineering. In this presentation, I will discuss two examples from our recent studies to explore the interplay of strain and lattice structure in 2D semiconducting materials, using a combination of low-voltage aberration corrected scanning transmission electron microscopy (STEM) imaging and density-functional theory studies.

First, I will show that the van der Waals (vdW) interaction between two transition metal dichalcogenide (TMDC) layers can significantly modify the atomic arrangement in a mirror twin boundary (MTB) in bilayer TMDC films, leading to the development of a local strain field of a few nanometers in the vicinity of the MTB [1].

When stitching two different monolayer materials with lattice mismatch side-by-side, e.g. a selenide monolayer (WSe_2 or $MoSe_2$) and a sulfide monolayer (WS_2 or MoS_2), strain relaxation at the lateral mismatched interfaces often leads to misfit dislocation arrays, which are highly active during the high temperature chemical vapor deposition growth for the heterostructures. Insertion of metal and S atoms into the dislocation cores induces dislocation climb while concomitant selective substitution of Se atoms around the dislocation core by S atoms, driven by the local strain field, leads to the growth of WS_2 and MoS_2 quantum-well arrays embedded in the WSe_2 and $MoSe_2$ monolayers. Preliminary monochromatic electron energy-loss spectroscopy (EELS) analysis shows that the optical properties of such quantum well superlattices are considerably different from their parent 2D components. This misfit-dislocation-driven growth mechanism should be applicable to different combinations of 2D monolayers with lattice mismatch for the fabrication of a wide range of 2D lateral superlattices with width smaller than 5 nm, a regime where quantum size effect would come into play [2].

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Topological Vortices and Charge-Lattice Interactions in Multiferroic Oxides

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Topological structures emerging near spontaneous symmetry-breaking transitions are ubiquitously observed in wide branches of science. Topological defects are invariant under continuous deformations or perturbations, and thus considered to be protected by topology. Here, we report structural transformation of sixfold vortex domains into two-, four-, and eightfold vortices via a different type of topological defect in hexagonal manganites. Combining high-resolution electron microscopy and Landau-theory-based numerical simulations, we investigate the remarkable atomic arrangement and the intertwined relationship between the vortex structures and the topological defects. The roles of their displacement field, formation temperature, and nucleation sites are revealed. All conceivable vortices in the system are topologically classified using homotopy group theory, and their origins are identified.

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