Abstracts
Keynotes
Gold Room C

Paul G. Kotula **Physical**
Mark H. Van Benthem
Data Analytics in Materials Characterization.
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Recent developments in X-ray detectors for electron-beam instruments have made collecting data more straightforward while creating a new problem of analyzing these vast sums of data. This presentation will discuss collecting and analyzing X-ray spectral images in the scanning and scanning transmission electron microscopes with an emphasis on solving materials science problems.

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Jeanette Killius **Biological**
K.T. Nakamoto, J.G. Mellott, M.E. Storey-Workley, C.S. Sowick and B.R. Schofield
Limits and Advantages of Light and Electron Microscopy When Studying Neural Circuits
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The neural pathways associated with hearing are complex. Sounds reach the ear and are translated into frequencies by the cochlea. These frequencies are transmitted along the ascending auditory pathway through the inferior colliculus (IC) in the midbrain, then on to the auditory cortex (AC) of the brain. The AC sorts the frequencies and then alters nerve cell responses to the sounds in the IC through AC neural projections of the descending auditory pathway. Thus the IC is a very important component in hearing as it integrates ascending auditory input AND descending input.

In this talk, I will discuss light microscopy techniques used to identify AC axons and to classify IC neurons as excitatory or inhibitory. TEM ultrastructural studies will show further identification of synapses and neurons.

Invited Speakers
Gold Room C

Hessam Ghassemi **Physical**
Protochips™ in-situ Electron Microscopy Solutions: Capabilities and Applications
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New innovations are transforming the Transmission Electron Microscope (TEM) from a simple high-resolution image acquisition tool into a nanoscale materials research and development laboratory. Researchers can now better understand material behavior by analyzing samples in real-world gas or liquid environments, at high temperature and with ultra-low noise electrochemical and electrical biasing techniques. With the new in situ tools from Protochips, materials research occurs in highly controlled environments at high resolution without sacrificing the analytical capabilities of the TEM such as EDS. Applications for these tools include heterogeneous catalyst reactions, nanostructure nucleation and growth, battery and fuel cell materials, high temperature nanoparticle behavior, and semiconductor devices.

In this presentation we show the most recent results using the Protochips Atmosphere™ 200 Gas Environmental Cell, the newly released Protochips Poseidon™ Select flowing liquid and electrochemistry cell, and the newly released Protochips Fusion™ heating and electrical biasing system. The Atmosphere system combines the Protochips patented silicon carbide MEMS heating technology, closed-cell holder design, and gas handling manifold, with our innovative Clarity workflow software allowing for atomic-scale resolution at gas pressures up to 1 atm and sustained temperatures up to 1000°C. The system is compatible with analysis tools including EDS and EELS. Recent results on gas-phase catalyst reactions will be presented. The Poseidon Select liquid cell surrounds samples in a self-contained, fully hydrated, hermetically sealed chamber directly within the TEM. Poseidon comes with a wide range of applications, from life science to battery research and corrosion studies, just to name a few. It features in situ electrochemistry capabilities, which enables the observation and characterization of electrochemical reactions in realistic reaction environments in real-time, and now offers liquid heating, for experiments in growth and reaction kinetics and temperature sensitive samples.

Protochips is happy to announce our next generation heating and electrical biasing system called Fusion. It reduces thermal drift by over 90%, offers best in class electrical biasing performance, simultaneous heating and biasing (electro-thermal mode), and features closed-loop temperature feedback. Like Atmosphere, Fusion is based on the Protochips patented silicon carbide heating technology, and the user friendly Clarity workflow software.

Raja K. Mishra **Physical**
Electron Microscopy Characterization of Automotive Structural Metals at Multi-Scales and Microstructure-Based Plasticity Modeling
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Today, when automotive manufacturing has come to rely on computer aided engineering (CAE) tools to guide its metal forming technology, electron microscopy in general and quantitative microscopy methods in particular have come to occupy very important roles in materials development and usage. Deep knowledge of factors that influence the deformation mechanisms of metallic alloys at the atomic scale, lattice scale, grain scale and continuum scale have become indispensable for development of robust models and to aid faster implementation of advanced light metals. High resolution imaging, electron tomography, electron backscatter diffraction, conventional TEM, STEM and SEM imaging, etc. along with analysis tools to inform crystal plasticity models with quantitative microstructural data at different length scales have seen decisive advances. This has resulted in more accurate constitutive modeling of forming on the one hand and novel material design guidelines incorporating specific microstructural attributes on the other. For example, the nucleation and growth of twinning and how the texture of the material changes with the amount of deformation - topics that can be studied by in situ electron backscatter diffraction (EBSD), in situ TEM and electron tomography - are proving to be important for validating crystal plasticity models to serve CAE needs and for guiding the metal industry to engineer better materials. Examples of current applications and challenges of computational and experimental microstructural research using electron microscopy will be highlighted in this presentation.

Dr. Raj Mishra is a Technical Fellow at the GM R&D Center in Warren, MI. He received his Ph.D. degree in Materials Science and Engineering from University of California in 1977 and has been with GM R&D Center since 1984. He has served in the Steering Committee of the National Center for Electron Microscopy at the Lawrence Berkeley Laboratory for over a decade. Dr. Mishra holds adjunct faculty positions at McMaster University and University of Waterloo in Canada and at Xian Jiaotong University in China. He has authored over 200 publications and patents covering microstructure analysis of engineering materials

George Graham **Physical**
Applications of *In-situ* TEM to Catalyst Characterization
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Knowledge of the atomic-scale structure of a catalyst under reaction conditions is critical to the detailed understanding of its performance. Transmission electron microscopy (TEM) provides the most direct means of obtaining such knowledge, but special means are required in order to perform TEM *in situ*. In this talk, I will describe studies where an electron transparent gas cell was used to observe restructuring of catalysts at elevated temperature under atmospheric pressure with aberration-corrected TEM, thereby providing insight not previously available from *ex-situ* examination

George Graham received a B.S. degree in Engineering Physics from the University of Illinois at Champaign-Urbana and a Ph.D. degree in Solid State Physics from Cornell University. He spent most of his career at Ford Motor Company's Scientific Research Laboratory in Dearborn, where he worked in the Metallurgy, Physics, and Chemical Engineering Departments, primarily on characterization of materials related to automotive catalysis. After retiring from Ford, he joined the University of Michigan as Adjunct Professor of Materials Science and Engineering, where he has maintained an active interest in catalysis through interaction with the Pan Research Group. He is currently also a Project Scientist in the Department of Chemical Engineering and Materials Science at the University of California – Irvine.

Benjamin J. Perrin **Biological**
Characterizing Actin Dynamics in Auditory Hair Cells using Light and Electron Microscopy
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Auditory sensory hair cells detect sound waves through stereocilia, which are actin-based protrusions arranged in tiered rows on the apical surface of the cell. Since hair cells are not renewed in mammals, stereocilia homeostasis, including proper length regulation, is critical for maintaining the ability to hear. Stereocilia lengths are dictated by the assembly and disassembly of their actin-based cores. Actins are a family of very similar proteins encoded by different genes. Hair cells express two isoforms, b-actin and g-actin (encoded by *Actb* and *Actg1,*respectively), which are 99% identical in amino acid and can reversibly polymerize into polarized filaments (filamentous or F-actin). Actin dynamics are regulated by several different types of actin binding proteins including those that modulate assembly and disassembly at filament ends as well as crosslinking proteins that link adjacent filaments together. Using a variety of genetic and imaging approaches, we have found that actin is turned over rapidly at stereocilia tips but is incredibly stable in the remainder of the core with a half-life of more than several months. Finally, the stability of the actin core, and the ability to maintain stereocilia lengths, depends on actin crosslinker proteins.

Biological and Physical Science Talks
Gold Room C

Physical
Electrochemical Synthesis of Organic Nanorods on Gold Nanoparticles Seeds
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Chemical Engineering

This talk is focused on nanoparticle-directed synthesis of tetrathiafulvalene charge-transfer salt (TTFBr) and potassium tetracyanoplatinate (KCP) organic nanorods using electrochemical deposition. Gold nanoparticles (AuNPs) here acting as nucleation seeds are deposited on highly oriented pyrolytic graphite (HOPG) by electrochemistry method. The nanorods, either TTFBr or KCP, are deposited subsequently on the AuNPs-decorated HOPG for the study of seed- mediated crystallization process, which is also by electrochemistry. The deposition processes are monitored by cyclic voltammetry and in-situ atomic force microscope. The field-emission scanning electron microscope is also employed to characterize the seed-crystal nanostructures. The TTFBr and KCP crystals deposited on AuNPs-decorated HOPG show preferential nucleation on the AuNP in comparison with crystals electrochemically deposited on the planar HOPG. The size of the crystals is controlled predominantly by the solution concentration and the seed size. Nanorods as small as 7 nm in height are nucleated on AuNPs of 20 nm in height. We hypothesize that the local curvature of the seed particle, that limits the cross sectional dimension and introduces an interfacial strain, contributes to the nanoconfinement effect observed. For further application, we applied this seed-directed synthesis method to fabricate organic nanowire sensors on gold lithographical patterns. The prototype sensor shows potential to compete with commercial sensors in detecting toxic and flammable gases. This research contributes a better understanding of crystallization at the nanoscale and a possible low-cost technology for chemical sensing applications.

Biological
Morphological Outcomes of Misfolding Peptides and Proteins
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Protein misfolding is associated with certain diseases, such as degeneration [1]. A neurodegeneration, such as Alzheimer's disease, involves misfolding of neuronal peptides and proteins into toxic aggregates. The degeneration of the eye proteins and peptides has been associated with eye disease, such as cataract. The mechanisms of misfolding and aggregation of certain degeneration peptides and proteins still remain elusive, further preventing the identification of viable drug targets. In order to gain further understanding about this class of biomolecules, we have selected two model systems: 1) tau protein and its peptides as related to neurodegeneration, and 2) crystallin peptides as related to the eye disease. We evaluated their aggregation morphologies using TEM. The effects of the protein-protein interactions, and metal ions on the aggregation outcomes will be described.

References
[1] (a) A. L. Goldberg, Nature, 2003, 426, 895-899. (b) D. J. Selkoe, Nature, 2003, 426, 900-904.

Physical
3D Hands-On Virtual Scanning Electron Microscope
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Mechanical and Aerospace Engineering

The goal in developing the 3D virtual scanning electron microscope (SEM) was to address the need for some hands-on SEM experience for a large number of students, without the cost-prohibitive of providing an access to such physical instrument. It is also appealing to pre-college students in recruiting events and used for training before using the "real one." The computer simulation was developed by an international team of faculty and students with a clear objective of making it affordable (only a laptop is needed), and user friendly, so that the user does not need an instructor's help to successfully run the SEM. At Western Michigan University, the physical SEM is next door and is available for demonstrations, while the virtual one is hands-on. The simulation is available free of charge to individuals and institutions around the world and over 110 already requested and received the lab downloads. Assessment of the impact on student learning shows a significant positive impact.

Physical

Accessorizing your TEM/STEM and/or "Keeping up with the Joneses"

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A number of game changing technologies have been introduced in the last few years for the current generation of TEM/STEM's. Modern Cs probe forming correctors have made STEM spatially resolved atomic analysis possible, and the advances that have been made in the digital imaging realm for TEM imaging have been remarkable. New high resolution/high speed/high sensitivity cameras offer a plethora of advantages for today's modern, and not so modern TEM/STEM's. Another amazing development was the introduction of large area SDD EDS detectors that increased sensitivity and greatly reduced the time to acquire large data sets. In addition, a variety of holders have also been introduced and are constantly being improved upon to offer researchers more capabilities and control of the sample within the microscope. Some of the latest advances in hardware and software will be discussed.

Kevin Mcilwrath has worked in the JEOL TEM applications group as a TEM/STEM Applications Scientist for 6 years and has been in the industry for over 23 years. Concentrating on TEM/STEM applications development in both the Materials Science field as well as the Biological Sciences with a focus on Cs corrected applications as well as TEM/STEM and analytical electron Tomography.

Physical

Graphene Moire Pattern Ultra-High Resolution Atomic Force Microscopy

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The ultra-high resolution of AFM was demonstrated in a graphene/hexagonal boron nitride (hBN) sample evaluation conducted by AFM. The sample consisted of hBN substrate overlaid with a graphene layer and was scanned under ambient air. The purpose of the evaluation was to assess the AFM ability to characterize the topography of the moiré pattern that was created when one layer was set on top of the other and offset by rotation. Using non-contact AFM mode and a standard AFM probe tip, the AFM was able to successfully image the moiré pattern super lattice constant of the sample in scans as large as 500 x 500 nm (see Figure 1a). In the higher magnification image of Figure 1b which was taken at a scan size of 60 x 60 nm provides the clear evidence that not only are the super lattice constants of the moiré pattern about 15 nm [1] in width, but that the spacing between each striation on the moiré pattern is roughly 4-5 nm in length. Observations of such striations in graphene/hBN systems have been previously reported [2]. This latter distance is in line with the expected tip radius curvature values for the AFM tip used to acquire all sets of data.

[1] A. Zandiatashbar, B. Kim, Y. Yoo, and K. Lee, Microscopy Today 23(06):26-31 (2015)
[2] P. Gallagher, M. Lee, F. Amet, F, et.al. Nature Comm. 7 10745 (2016)

Physical

Image Texture Analysis and Application to Acicular Mullite Porous Ceramic Microstructure

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Acicular mullite porous ceramic can display a range of microstructures. The microstructure in turn impacts performance metrics such as strength, modulus and filtration back pressure. Assessment of microstructure was done in a completely subjective manner; a small group of experienced individuals assessed SEM images. The goal of the project described here was to develop an objective way to quantify aspects of this microstructure. The approach was to apply computational image analysis to SEM images. First, an image is divided into grids of systematically smaller regions (domains). The mean and standard deviation of gray levels were calculated within each domain. The set of values for each domain size were combined to yield three figures of merit: The standard deviation of the average gray-levels (variability of overall image brightness); the average of the standard deviations of gray-level within the domains (overall variability of the image brightness); and the standard deviation of the standard deviations of gray-level within the domain (the variation in the brightness variability across the image).The second step in the image analysis method is to process these data using principal component analysis. The model had four significant Principal Components based on the size of the eigenvalues. Principal Component 1 (PC1) accounted for nearly 81% of the variability in the original data. PC2 accounted for over 10% of the variability. The developed image texture analysis method was successful in objectively quantifying the microstructure of acicular mullite porous ceramic. Texture analysis metrics correlated with the important parameters of mullite crystal size and microstructure consistency.

POSTERS

Gold Rooms A & C

Biological 1

A Decrease in Rtca Protein Levels Promotes Robust Axonal Regeneration in Mouse Retina Optic Nerve

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A decrease in Rtca protein levels promotes robust axonal regeneration in mouse retina optic nerve Shravan K. Chintala, PhD Naveena Daram, Student Researcher Research Abstract In POAG patients, progressive degeneration of axons in the retina and the optic nerve (ON) leads to permanent vision loss. Despite the fact that elevated intra-ocular pressure (IOP) promotes axonal degeneration, the mechanisms leading axonal degeneration remain unclear. Consequently, there is no cure to regenerate axons in POAG patients. The purpose of this study is to investigate whether elevated levels of Rtca (RNA-3'phosphate cyclase) promotes axonal degeneration. Poly(I:C) (polyinosinic-polycytidylic acid), a specific activator of Toll-like receptor 3 (TLR3), or PBS was injected into the vitreous humor of C57BL/6J mice. At 24, 48, and 72 hours after treatments, axonal degeneration in the retinas was assessed by immunostaining of whole retinas with Tuj1 antibody. Axonal degeneration in the ONs was determined by anterograde labeling with Cholera Toxin B (CTB). Relative levels of TLR3 and Rtca proteins in retinal protein extracts was determined by western blot analysis. Cellular localization of Rtca in the retina and optic nerves was determined by immunostaining with antibodies against Iba1 (for activated microglia) and CD68 (for macrophages).Poly(I:C) promoted a progressive degeneration of axons both in the retina and the ON over a 72 h time-period. Poly(I:C) up-regulated Rtca protein levels in activated microglia in the retina, and CD68-positive cells and activated microglia in the ON. Interestingly, Poly(I:C) did not up-regulate Rtca protein levels in TLR3 knockout mice and promoted axonal regeneration. Results presented in this study indicate that the activated microglia and CD68-positive macrophages.

Biological 2

Remodeling of the Endoplasmic Reticulum in Meiotically-Arrested Germ Lines

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In many animal species, oocytes arrest in meiosis for an extended period of time. In *C. elegans* hermaphrodites, oocytes enter extended meiotic arrest when sperm become depleted at ~4 days of age. The oocytes maintain their quality for a short time, but as mothers age, the percent of embryos that die increases. We are investigating the mechanisms that promote oocyte quality and the cellular transitions that occur in old-aged oocytes. We demonstrate that the endoplasmic reticulum (ER) undergoes substantial remodeling in arrested oocytes. In hermaphrodites depleted of sperm, the ER distribution changes from being diffuse throughout the cytoplasm to being concentrated in large cortical patches. These changes also occur in the arrested oocytes of *fog-2* females; thus, it is the age of the oocyte, not the worm that triggers the ER remodeling. Moreover, the changes appear to be reversible. We have begun investigating the regulation of ER remodeling, focusing on *car-1* and *cgh-1*. CAR-1 appears to regulate the ER in the distal germ line. Interestingly, the CAR-1 protein assembles into irregular sheets in the distal core of *cgh-1* germ lines; therefore, we asked if ER accumulates in similar sheets after cgh-1(RNAi). Preliminary confocal and TEM results show an accumulation of membranes in the core. We hope these studies will shed light on the cellular responses to aging and meiotic arrest, and eventually address the intriguing connection between RNP complexes and the ER.

Biological 3

A Study of UVB-Induced DNA Damage in Cultured Human Lens Epithelial Cells

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Age related cataracts are the second leading cause of blindness in the United States. Cortical cataract (an opacification in the lens periphery) is a major type of this disease; however, little is known about its mechanism of formation. One cause is known to be solar UVB radiation, possibly through UVB-induced damage to DNA present in the lens epithelium. Here, we investigated DNA damage andrepair in cultured human lens epithelial cells (LECs) exposed to two different intensities of UVB light, 0.9mW/cm² and 0.09mW/cm². Following UVB exposure, cells were incubated for various times, and analyzed for DNA strand breaks (TMR Roche Red), reactive oxygen species (CellROX), cell viability (MTT), and fluorescence immunocytochemistry, with the latter assay employing antibodies for the DNA repair enzyme poly(ADP)-ribose polymerase 1 (PARP-1) and poly(ADP)- ribose (PAR) polymers. The results indicated a biphasic mechanism for the higher dose of UVB in which a first phase of DNA strand breaks were repaired immediately after exposure, and then followed by a second phase of strand breaks at 90 min, which were repaired again. For the lower dose of UVB there was only an initial occurrence of DNA strand breaks and repair. Furthermore, it was observed that 90 min after the high UVB dose, PAR polymers migrated from the cell nucleus to the cytoplasm, possibly to induce cell death. The results indicate that PARP-1, along with PAR polymers, assist in the repair of UVB-induced damage to DNA in the human lens epithelium, and may also induce cell death.

Biological 4

Olfactory Bulb Anatomy and Circuitry Following Ablation of p75NTR Expressing Cells

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Olfactory sensory neurons (OSN's) provide synaptic input to the olfactory bulb (OB). Reducing OSN activity induces OB neuroplasticity as measured by a reduction in tyrosine hydroxylase (TH) and dopamine levels. Similarly, pan-neurotrophin receptor 75 (p75NTR) activation also reduces TH levels. However, the location of p75NTR within the glomerular layer is still uncertain. First, we sought to localize p75NTR within the glomerular layer of the OB. ImageJ analysis revealed that p75NTR failed to colocalize with TH on interglomerular neurons, GFAP expressing glia, and olfactory marker protein (OMP) expressed on OSN's (N=3). In a second experiment, we sought to induce OB plasticity by ablating p75NTR expressing cells. A p75NTR-targeted saporin toxin was injected into the dorsal OB along with an untargeted control toxin. Immunohistochemistry was performed to determine the expression of p75NTR within the OB. Treatment reduced p75NTR signal by 56% (N=7) in the immunolesioned OB compared to the control OB. Treatment did not reduce expression of any neuronal markers (TH and OMP) or glial markers (GFAP) (N=3-4 each). Ongoing studies are being performed to determine if p75NTR colocalizes with GAD65+ interglomerular neurons. Also, confocal imaging will be performed to obtain a higher resolution of p75NTR immunofluorescent labeling for colocalization analyses.With these and future data, we can provide a better explanation of p75NTR's location and role in neural plasticity within OB circuits.

Biological 5

What is the Mechanism for Change in Olfactory Epithelium with Detergent Induced Damage?

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Chemosensory epithelia degenerate and regenerate throughout life. Damage to the olfactory epithelium (OE) induces anosmia (smell loss) and is associated with clinical depression. Previous studies have shown the ciliary layer to be disrupted with detergent in aquatic animals specifically exhibiting cleaved cilia. This study investigates OE damage following intranasal irrigation with a detergent solution, Triton X-100. We treated 8 mice: 3 were treated with phosphate buffer saline (PBS); 4 were treated with 0.1% Triton X-100. The tissues were then imaged using standard scanning electron microscopy techniques. In the detergent treated animals we did not find cleaved cilia as other studies have previously shown; instead we saw a matted ciliary layer. It is not yet clear if the ciliary layer is directly affected or if the mucus layer in which the cilia are dispersed within is affected by the detergent. Future studies will examine the damage to the OE through tissue staining and morphology analysis.

Physical 1

Production of Few Layers Graphene Sheets via Direct Exfoliation of Graphite Oxide

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Graphene, a 2D single atomic layer of sp2 bonded carbon atoms, has gained the attention of researchers over the globe owing to its exceptional properties such as high strength, transparency, electrical conductivity, chemical inertness, and high surface area. A lot of efforts have been put forward in devising the routes for the easy and scalable production of graphene. The present research focuses on a two-step production of graphene sheets through mechanical exfoliation of graphite oxide, produced using Modified Hummer''s method. Raman spectroscopy, X-ray diffraction, and Infra Red spectroscopy were used to validate the conversion of graphite oxide to graphene. Transmission electron microscopy was used to study the morphology of graphene sheets, and X-ray photoelectron spectroscopy was employed to identify the type of bonds on graphene sheets.

Physical 2

Strain- and Depth-Dependent Poisson's Ratio in Articular Cartilage

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Articular cartilage functions as a load bearing material in joints. Compression of cartilage changes its complex biomechanical environment, which are depth- dependent. Chondrons are the mechanical unit of living cells in cartilage, consisting of chondrocytes and its pericellular structure. We aim to measure the compressive modulus and Poisson's ratio of the chondrons in situ. Full–depth osteochondral slices with a thickness of 120±5μm

were prepared from the center region of canine humeral heads. A homemade glass fluid chamber, which sandwiched the cartilage slice, was placed under a Nikon microscope. Step wise unconfined stress relaxation tests were performed in situ using compression step of 30μm at 5μm/s under computer control. At the end of each relaxation, an image (pixel size of 0.435μm) was captured, where the cellular deformation was analyzed. The preliminary results reveal that both compressive modulus and Poisson's ratio increase for all depth region at all individual strains. Within five strain increments (max 25%), the strain-dependent Poisson's ratio increases from superficial zone to transitional zone and then decreases from transitional zone to radial zone. Our observation of the negative Poisson's ratios of chondron in transitional zone has never been reported before, which could result in enhanced toughness even if the material is compliant. Whether the orientation of collagen fibrils is responsible for auxetic behavior of chondron is an open question. Further studies will determine the relationship between mechanical response of cartilage and its cellular composition, in both healthy and osteoarthritic tissues.

We thank NIH for the support (R01 AR069047).

Physical 3

Synthesis and Characterization of Transition Metal-Doped $BaTiO_3$ Colloidal Nanocrystals

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Multiferroic materials possess at the same time more than one ferroic property such as ferroelectricity, ferromagnetism, and ferroelasticity. The coexistence of these physical properties is difficult to achieve, but it can open the door for developing new memory devices. Due to the important role that the surface plays on the properties of nanomaterials, we have developed a new synthetic route that allows a versatile control over the size of agglomerate free perovskite nanocrystals. Transmission electron microscopy studies have confirmed that the synthesis can be used to obtain monodisperse colloidal nanocrystals of $BaTiO_3$. Furthermore, we have demonstrated that it is possible to achieve high doping concentration (up to 6%) without the formation of secondary phases. The inclusion of the transition metal into the nanocrystals has been verified by electron paramagnetic spectroscopy and vibrating sample magnetometer studies. The ferroelectric behavior has been investigated with piezoresponse force microscopy, which it has revealed the possibility to read and write arbitrary pattern over nanocrystal thin films by applying an electric field.

Physical 4

When Ligand Exchange Leads to Ion Exchange: Exploiting Facets for Nanoparticle Conversion and Assembly

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Surface ligand chemistry is a key player in nucleation, growth kinetics, chemical and colloidal stability, solubility and self-assembly of nanocrystals (NCs). The insulating organic ligands used in colloidal synthetic routes preclude efficient electrical transport in assemblies limiting potential application in solid state devices. Ligand exchange with chalcogenide ions (S_2^-, Se_2^- and Te_2^-) or chalcogenidometallate ions (SnS_{44}^-, $Sn_2Se_{64}^-$, $In_2Se_{42}^-$, $Ge_4S_{104}^-$, etc.) leads to decreased NC spacing but electrostatic repulsions negatively affect self-assembly. We seek to use covalently crosslinking metal cations (Sn_4^+) as a means to make robust assemblies in 2 and 3D. PbTe NCs were prepared as cubes and cuboctahedra and exchanged with S_2^- or SnS_{44}^-. Ligand exchange proceeded as expected with the cubes and subsequent treatment with Sn_4^+ ions led to crosslinking gels. However, ligand exchange of cuboctahedra led to form PbS via an anion exchange process. The role of facets in dictating and limiting reactivity will be discussed along with the potential to access metastable phases via ion-exchange or cross-linking.

Physical 5

Solvothermal Synthesis of Mg-doped Li_2FeSiO_4/C Nanocomposites and their Electrochemical Performance for Advanced Lithium Ion Batteries

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Li_2FeSiO_4 is the potential cathode material for lithium ion batteries due to its high specific energy density (~330mAh/g), high thermal stability due to Si-O covalent bond, low cost because of abundant of iron and silicon ore and environmental friendly. However, it suffers from poor electronic conductivity and slow lithium ion diffusion in the solid phase. To address these issues, we have synthesized mesoporous $Li_2MgXFe1\text{-}XSiO_4$/C (where X=0.0, 0.01, 0.02 and 0.04) nano-composites synthesized by solvothermal method using tri-block copolymer (P123) as carbon source and structure directing agent. The structure and morphologies of nano-composites of $Li_2MgXFe1\text{-}XSiO_4$/C have been characterized by various techniques such as x-ray diffraction, SEM and TEM and surface area and pore size distribution were obtained by using Brunauer–Emmett–Teller (BET) and Barrett–Joyner–Halenda (BJH) analysis. The electrochemical properties were evaluated by Galvanostatic charge/discharge, electrochemical impedance spectroscopy (EIS), and cyclic voltammetry (CV) and the effect of Mg-doping was investigated. We found that 1% Mg doped Li_2FeSiO_4/C shows a better electrochemical performance among all the nano-composites. The improved electrochemical performance of 1% Mg doped Li_2FeSiO_4/C has been attributed to its enhanced electronic conductivity, higher surface area, higher lithium diffusion coefficient, and stable structure of material due to optimum amount doping of Mg in Li_2FeSiO_4/C. The reduction of capacity with higher percentage of Mg in Li_2FeSiO_4/C nanocomposites is attributed to decreased surface/porosity and an increase in the particle size as confirmed by TEM images.

Physical 6

Surfactant Assisted Synthesis of Monodisperse Fe Doped TiO_2 Nanocrystals and its Photocatalytic Activity

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TiO_2 is a well-studied photocatalyst due to its several advantages such as stability, low cost, non-toxicity and chemical inertness. The polymorph of TiO_2 known to be photocatalytically active is anatase TiO_2. An efficient photocatalyst should have high surface area and a narrow bandgap for solar energy conversion. Surfactant assisted synthesis regulates shape and size of the nanocrystals and doping with impurities manipulates the band gap. The coupling of impurity ion doping and hydrothermal synthesis has not been explored previously. In this work, we employ hydrothermal synthesis for obtaining uniform monodisperse nanocrystals of TiO_2 as well as dope with different Iron (Fe) concentration. X-Ray diffraction studies performed is in agreement with Raman spectroscopy determining formation of phase pure anatase TiO_2. Electron microscopy is used to monitor the shape and size of the particles as a function of increasing dopant ions concentration. Diffuse Reflectance spectroscopy (DRS) is employed to understand the effect of dopant ion on band structure of TiO_2. Fe doped monodisperse TiO_2 nanocrystals will be analyzed for its photocatalytic activity with the aid of UV-Vis spectroscopy. With uniform morphology and phase composition of TiO_2 obtained, the effect of doping at nanoscale needs to be understood in detail.

Physical 7

Titanium Dioxide Nanotubular Structures

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Titanium dioxide is widely used in many applications owing to its exceptional properties. These properties can be enhanced by changing the processing parameters. In this study, we manipulated the process parameters to obtain different titanium dioxide nanotubular morphologies. Scanning electron microscopy (SEM) was used to investigate the nanostructures. The revealed nanostructures showed different nano topographies. Moreover, the contact angle goniometry was used to determine the wettability of the formed nanostructures. X-ray diffraction revealed the amorphous nature of titanium dioxide nanotubes. The nanostructure was electrochemically tested in 1 wt % NaCl saline solution.

Physical 8

α-MnO_2 Nanorod-composites as Electrode Material for Supercapacitors

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MnO_2-based supercapacitors, as electrochemical storage systems, have attracted intense research due to their low cost, natural abundance, high theoretical specific capacitance and environmental friendly nature.In the current work, two different kinds of nanocomposites have been prepared using chemical solution based co-precipitation method to synthesize α-MnO_2 and α-MnO_2/-CNF (carbon nanofibers, 5 wt%). The XRD studies have provided evidence for the formation α-MnO2 single phase.The nanocomposites have been characterized using SEM, TEM, and BET methods. Both SEM and TEM studies reveal the formation of nanorods of α-MnO_2, but with a bigger size in the case of α-MnO_2-CNF nanocomposites. Further, BET studies reveal a higher surface area of 266 m²/g for α-MnO_2 nanocomposite (without CNF) compared to 131 m²/g of α-MnO_2 nanocomposites with CNF. Cyclic voltammetry (CV) studies and galvanostatic charge/discharge studies have been performed on α-MnO_2 nanocomposites, coated on Ni foam, using a potential ranging from -0.02 to 0.8 V, in a 1 M Na_2SO_4 aqueous solution.Although α-MnO_2-CNF exhibited two orders of magnitude higher electrical conductivity (0.67 S/cm), it showed a lower specific capacitance of 192 F/gas an electrode, compared to 245 F/g for pure α-MnO_2 electrode. We attribute this to a reduced surface area in α-MnO_2-CNF nanocomposites. Further research is underway to optimize the amount of CNF in α-MnO_2-CNF nanocomposites to enhance supercapacitor performance.