Limits of electron and ion beam analysis and their application to nanoscience

4th Annual Joint Workshop

Microscopical Society of Canada

& Japanese Society of Microscopy

Canada (Edmonton) June 11, 2023

日本 (virtual): 2023 年 6 月 12 日

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Hybrid meeting: virtual and in-person

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JSM-MSC Annual Joint Workshop

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&
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MSC·SMC

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Limits of electron and ion beam analysis and their application to nanoscience

Electrons and ions allow imaging, chemical and structural analysis at sub-nanometer scale in many materials of interest to physical and biological sciences. The practical limits can arise from the instrumentation, the interactions responsible for the measured signal and, ultimately, by radiation damage inflicted on the studied sample by the incident beam. Presentations in this year's symposium discuss the practical aspects of pushing the boundaries of electron and ion microscopy instrumentation, and the practical problems applying the electron and ion beam analysis to real-world samples in physical and biological sciences.

Workshop sponsors

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time Edmonton Sunday June 11, 2023	time 東京 Monday June 12, 2023	Presenting author	
4:55 – 5 pm	7:55 – 8 am	Ken Harada Marek Malac	Opening remarks
5 – 5:30 pm	8 – 8:30 am	Takehito Seki	Direct Electromagnetic Field Imaging at Defects by Differential Phase Contrast Scanning Transmission Electron Microscopy
5:30 – 6 pm	8:30 – 9 am	Nadi Braidy	Encapsulation of dyes in carbon nanohorns
6 – 6:30 pm	9 – 9:30 am	Cathal Cassidy	Gas-based charge compensation measured by off-axis holography in environmental TEM
6:30 – 7 pm	9:30 – 10 am	Martin Couillard	Disentangling EELS signals from optical modes in photonic and plasmonic nanoparticle dimers and trimers
7 – 7:30 pm	10 – 10:30 am	Kodai Niitsu	Magnetic configurations of a skyrmionic vortex stabilized in FeGe nanoparticles
7:30 – 8 pm	10:30 – 11 am	Nabil Bassim	Insights about Atomic-Scale Heteroepitaxy based on Correlative Electron Microscopy of Van der Waals Heterostructures
8 – 8:30 pm	11 – 11:30 am	Makoto Schreiber	Lensing charged particles with the magnetic vector potential
8:30 – 9 pm	11:30 – 12 noon	Makoto Kuwahara	Time-Resolved Measurement in TEM Using a Semiconductor Photocathode
9 – 9:30 pm	12 – 12:30 pm	Alyssa Williams	Improved Visualization of Bone Ultrastructure in 3D FIB-SEM
9:30 – 10 pm	12:30 – 13:00 pm	Natalie Reznikov	The ultrastructure of bone in 3D: A twist of twists
10 – 10:10 pm	13:00 – 13:10 pm	Shigeo Mori Misa Hayashida	Closing remarks

On behalf of the Microscopy Society of Canada- Société du Microscopie du Canada (MSC-SMC), I am honoured to welcome you to Canada and the Fourth Japan-Canada Microscopy Societies Joint Symposium held at the in Edmonton, Alberta, Canada. Together, our two societies have been pushing the frontiers of electron microscopy with this exciting symposium, now in its fourth year.

This year's symposium theme "Limits of electron and ion beam analysis and their application to nanoscience" will bring together the latest developments and eminent speakers from across Japan and Canada presenting ground-breaking research focusing on the practical aspects of pushing the boundaries of electron and ion microscopy instrumentation, overcoming challenges, and applying electron and ion beam analysis to real-world samples across the physical and biological sciences.

Hosted this year as a satellite conference in association with the annual MSC meeting, the JSM-MSC symposium has become a hallmark of our national meeting and an event looked forward to by all. Its continued offering is strong evidence of the advancements in microscopy our societies are making together.

I extend my deepest thanks to the sponsoring organizations and volunteer organizers in Canada and Japan, especially Ken Harada, Shigeo Mori, Misa Hayashida, and Marek Malac, who worked so tirelessly to bring you this exciting meeting year after year.

We look forward to welcoming you to Canada (again!) next year for the 50th Anniversary of the MSC-SMC and look forward to our next joint meeting in Japan.

I give my warmest welcome to all attendees, in-person or virtually, and wish you a good scientific meeting.

Best wishes,

Kathryn Grandfield

President, Microscopy Society Canada

The Japanese Society of Microscopy

On behalf of the Japanese Society of Microscopy, it is my great pleasure and honor to welcome all the great scientists, young researchers, and students to the Fourth Annual Joint Workshop of the Microscopy Society of Canada- Société du Microscopie du Canada (MSC-SMC) and the Japan Microscopy Society (JSM), which will be held on June 11th, 2023 in Edmonton, Alberta, Canada both in person and online.

This workshop, "Limits of electron and ion beam analysis and their application to nanoscience," shares an insight into the latest research and state-of-the-art technologies in electron and ion beam analysis, which gains immense interest from a broad range of scientists and engineers. Nine renowned speakers from Canada and Japan will provide their cutting-edge research and discuss current limitations and future breakthroughs in the field. It will also provide an excellent networking opportunity for young researchers and students to develop lifelong friendships.

The strength and quality of this year's workshop reflect the success of the multiple preceding joint events that began in 2020. Each year, the relationship between the volunteered organizers and the two societies strengthened, despite the difficulties in organizing international meetings due to the COVID-19 pandemic. This year's meeting marks the start of a new stage in the post-pandemic collaboration between MSC-SMC and JSM.

I look forward to an excellent meeting with great scientists from two countries and sharing new and exciting research in electron microscopy and related technologies.

Yours sincerely,

Shigeo Okabe

President-Elect
The Japanese Society of Microscopy

National Research Council Canada | Conseil national de recherches Canada

I am very pleased to welcome representatives from Japan and Canada to the 4th annual workshop of the Japanese and Canadian Microscopy Societies here at the NRC's Nanotechnology Research Centre (NRC-NANO) in Edmonton, Alberta. NRC is committed to supporting R&D in microscopy and working with Japan as a priority international partner. Our Developmental and Analytical Microscopy team not only maintains and improves a suite of microscopes on our site, but they have also convened NRC researchers across Canada to bring more awareness of microscopy to the broader research community. NRC-NANO has also developed the first open-source electron microscope, known as the NanoMi, to build awareness of microscopy and also provide a platform for development of new microscopy capabilities in a plug and play system.

We view Japan as a key partner in advancing microscopy in Canada, and I hope this workshop can seed new collaborations between our countries to advance the field as a whole. We have an excellent track record of hosting and collaborating with Japanese students and researchers from both industry and academia, and I am very interested to expand on this success going forward.

Enjoy the workshop and best of luck with your research endeavors.

Andrew J. Myles, Ph. D.

Director, Research and Development, Nanotechnology Research Centre
National Research Council of Canada | Government of Canada

A message from the workshop organizers

The 4th joint symposium of the Japanese and Canadian microscopy societies, JSM and MSC, is about to take place.

The 4th symposium is being held only a short time after the Kurashiki (Japan) meeting. We are a satellite symposium of the joint conference of MSC with the International Union of Microscopy Societies (IUMAS). Taking into account the short time gap between the Kurashiki meeting and the limitations on number of attendees at IUMAS, we aimed at a modest size meeting in Edmonton.

The JSM-MSC is held in hybrid format to accommodate for the covid19 restrictions at the time of meeting planning. It is our understanding that the convenience of hybrid meetings has become important consideration for participants. According to the tradition of the previous meetings, the online participation JSM-MSC symposium is free of charge and open to everyone. It is our hope that the JSM-MSC meetings will result in one-on-one researcher interactions between Japan and Canada, resulting in new methods and applications of microscopy.

This year, we are pleased to have excellent speakers presenting at the JSM-MSC. The topics range from physics underlying electron and ion microscopies, to applications in biological and materials sciences.

We strive to make the JSM-MSC symposium a long-term feature for our societies. Please look forward to the information on the 2024 meeting, likely in conjunction with the 50th anniversary meeting of MSC.

Marek, Misa, Ken and Shigeo
At your service
March 25, 2023

Direct Electromagnetic Field Imaging at Defects by Differential Phase Contrast Scanning Transmission Electron Microscopy

Takehito Seki[1,2*] and Naoya Shibata[1,3,4*]

1. Institute of Engineering Innovation, The University of Tokyo, Tokyo 113-8656 Japan
2. PRESTO, Japan Science and Technology Agency, Kawaguchi, Saitama 332-0012, Japan
3. Nanostructures Research Laboratory, Japan Fine Ceramic Center, Nagoya 456-8587, Japan
4. Quantum-Phase Electronics Center (QPEC), The University of Tokyo, Tokyo 113-8656, Japan.
* Corresponding author: seki@sigma.t.u-tokyo.ac.jp

Aberration-corrected scanning transmission electron microscopy (STEM) is a powerful technique for directly observing atomic-scale local structures inside materials and devices. However, in ordinary STEM, atomic-resolution observation of magnetic materials is essentially difficult, since high magnetic fields are inevitably exerted on samples inside the magnetic objective lens. In recent years, we have succeeded in developing a new magnetic objective lens system that realizes a magnetic field free environment at the sample position. Using this new objective lens system combined with the state-of-the-art higher order aberration corrector, direct atom-resolved imaging of magnetic materials is realized in STEM [1]. This novel electron microscope (Magnetic-field-free Atomic Resolution STEM: MARS) is expected to be used for research and development of advanced magnetic materials and devices.
On the other hand, new imaging possibilities in STEM has been widely explored owing to the rapid development of segmented and pixelated detectors. Differential phase contrast (DPC) imaging is considered to be one of the most useful imaging techniques because electromagnetic field distribution inside materials and devices can be directly imaged in real space. Applying DPC imaging for atomic-resolution STEM [2], it has been shown that the electric field distribution within single atoms and magnetic field distribution of single atomic columns can be imaged [3,4].
By combining the magnetic-field-free STEM with DPC imaging technique, new possibility for characterizing local electromagnetic fields at crystalline defects has been opened. The current on-going developments and material applications especially for interfaces will be presented in the talk.

Acknowledgment:
The authors thank all the collaborators of this research, especially Y. Kohno, S. Toyama, S.D. Findlay and Y. Ikuhara for their contribution to the works shown in this presentation. The authors acknowledge JST ERATO Grant Number JPMJER2202, Japan. A part of this work was supported by JST SENTAN Grant Number JPMJSN14A and the JSPS KAKENHI Grant numbers 20H05659, 19H05788, 17H06094.

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[1] N. Shibata et al., *Nature Comm.* **10**, 2380 (2019).
[2] N. Shibata et al., *Nature Phys.*, **8**, 611-615 (2012).
[3] N. Shibata et al., *Nature Comm.* **8**, 15631 (2017).
[4] Y. Kohno et al., *Nature* **602**, 234-239 (2022).

Encapsulation of dyes in carbon nanohorns

Fanny Casteignau[1], Louka Yilmaz[1], Mathilda Dano[1], Armand Soldera[2], Christian Ricolleau[3], Richard Martel[4], Nadi Braidy[1,5]*

[1] Department of Chemical and Biotechnological Engineering, Université de Sherbrooke, Sherbrooke, Canada
[2] Department of Chemistry, Université de Sherbrooke, Sherbrooke, Canada
[3] Laboratoire Matériaux et Phénomènes Quantiques, Université de Paris, Paris, France
[4] Department of Chemistry, Université de Montréal, Montréal, Canada
[5] Institut Interdisciplinaire d'Innovation Technologique 3IT, Université de Sherbrooke, Sherbrooke, Canada
[*]nadi.braidy@usherbrooke.ca

Carbon nanohorns (CNHs) are ~50 nm long graphenic cones typically capped by a C_{60} and arranged in inseparable aggregates of about 100 nm in diameter with a sea urchin structure[1]. Thanks to their hollow structure, dyes can be encapsulated in CNHs to serve as chemical sensors. Giant Raman scattering was observed with α-6T dyes encapsulated in carbon nanotubes. A similar enhancement of the Raman signal was observed by our group when this dye was encapsulated in CNHs. It was proposed that Giant Raman scattering is due to the α-6T chains arranged in a J-configuration[3]. However, direct imaging coupled to simulation is needed to better understand the Raman scattering phenomenon and help tailor new sensing functionalities.

Here, we couple spherical aberration-corrected high-resolution electron microscopy (Cs-HRTEM), molecular dynamics, and TEM multi-slice methods to help understand the encapsulation behavior of dyes in CNHs. We investigate the impact of the CNHs morphology and the thermal oxidation annealing step on the encapsulation yield and α-6T configuration within the horns. We show that α-6T chains align along the walls of the CNHs (Fig. 1). However, it was difficult to provide direct experimental evidence of their J-configuration. Furthermore, our molecular dynamics study shows that the α-6T chains are preferentially attracted to the tip of the CNHs and other defects in the graphenic structure.

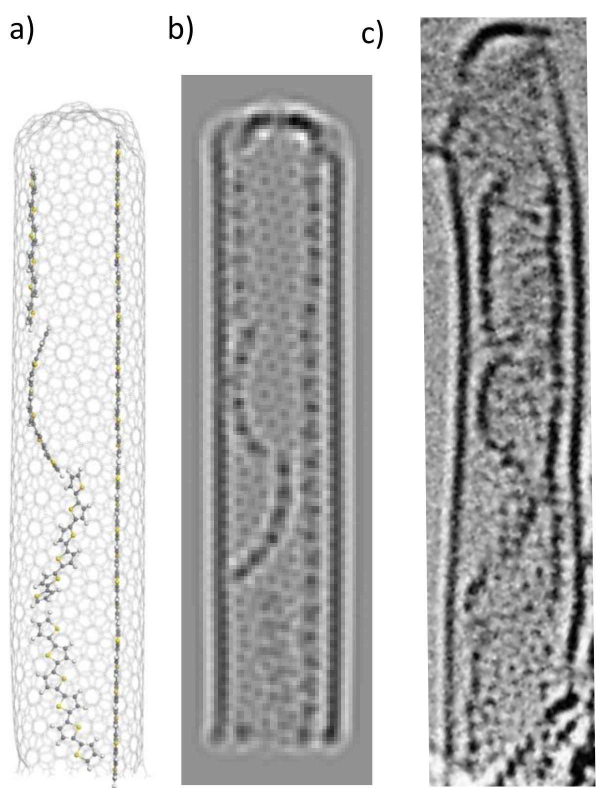

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Fig. 1 a) Eight α-6T in a CNH model that was used for b) multi-slice TEM image simulation to compare with c) Cs-HRTEM micrograph of an α-6T encapsulated in a CNH.

Gas-based charge compensation measured by off-axis holography in environmental TEM

Cathal Cassidy[1*], Makoto Tokoro Schreiber [2]

[1] OIST Graduate University, Onna-son, Okinawa, Japan.
[2] Department of Physics, University of Alberta, Edmonton, Alberta, Canada
[*]c.cassidy@oist.jp

When imaging samples with low electrical conductivity in an electron microscope, the sample can accumulate charge and distort the measurement. Charging effects have been well investigated in scanning electron microscopy (SEM) but have been less studied in transmission electron microscopy (TEM). One method of reducing charging effects is to coat the sample of interest with a thin conductive film such as carbon. This is for the most part an irreversible treatment and is not desirable for all samples such as vitreous ice films in biological electron microscopy. Another method to compensate beam-induced charging is through the introduction of gas flow over the sample. This technique has been established in SEM [1] but has not been well investigated in TEM.

In this work, we observe and quantify how different partial pressures of gas in an open-cell type environmental TEM affect the charging process on dielectric thin films through off-axis electron holography. We observe that the large phase-ramp present at the edge of a film in high-vacuum conditions (Figure 1a) is largely eliminated when a certain partial pressure of gas is introduced (Figure 1b) – indicating a significant reduction of charge on the film. Through this work, we extend on previous studies of how gaseous environments affect the electron holography process [2] and TEM imaging in general.

In this talk, we present some initial results on the effect of electron dose, gas pressure, specimen material, and thickness upon the observed charging and compensation behavior, as observed by off-axis electron holography. We discuss the utility of gas-flow as a reversible charge-modulation method which may aid studies of other processes such as mean inner potential measurement and charging-induced specimen vibrations.

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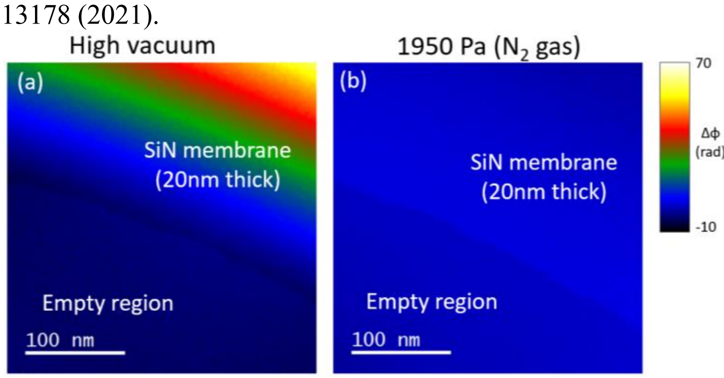

Fig. 1. Phase maps, reconstructed from 300kV off-axis holograms, from a 20nm SiN membrane. (a) Standard high vacuum case. (b) Same film region as (a) but with 1950 Pa N_2 gas in the specimen area. There is a clear, charging-induced phase ramp in (a), which has been substantially reduced by the introduction of N_2 gas in (b).

Disentangling EELS signals from optical modes in photonic and plasmonic nanoparticle dimers and trimers

Martin Couillard [1*]

[1] Energy, Mining and Environment Research Centre, National Research Council Canada, Ottawa, ON, Canada
[*]martin.couillard@nrc-cnrc.gc.ca

Advances in electron-beam spectroscopies performed in an electron microscope with a sub-nanometer probe have enabled unique access to optical properties for a wide range of nanostructures [1]. If we first consider a simple system, an individual sphere, two distinctive types of optical modes are expected: the plasmonic and the dielectric resonances. Plasmonic resonances relies on the response of *free electrons*, whereas dielectric resonances originate from displacement currents due to oscillations of *bounded electrons*. By bringing spheres in close proximity, unique optical features emerge from mode splitting and shifting. In this presentation, we explore using EELS the optical properties of monomers, dimers and trimers of plasmonic ($Ag@SiO_2$) and dielectric (SiO_2) quasi-spheres. For plasmonic dimers and trimers (figure 1), EELS measurements display multiple intertwined optical modes. Maps extracted from an independent component analysis (ICA) allow for the identification of local modes (bulk, vertices and facets) along with delocalized modes (coupling of spherical modes). Five components, however, is not sufficient to properly describe such a system. To analyze a larger number of components, we propose to reintroduce spatial information, which is absent in ICA, in order to classify the various contributions. For dielectric dimers (figure 2) and trimers, sharp peaks are observed below the silica bandgap, but at different energies than predicted by simulations. Nonetheless, trends such as blue-shift displacements or energy broadening are well reproduced, and confirm the coupling of optical whispering gallery modes [2]. In summary, our results emphasize the importance of experimental measurements, because of the high sensitivity of photonic resonances to optical constants of materials and the high sensitivity of plasmonic resonances to morphological details.

References:

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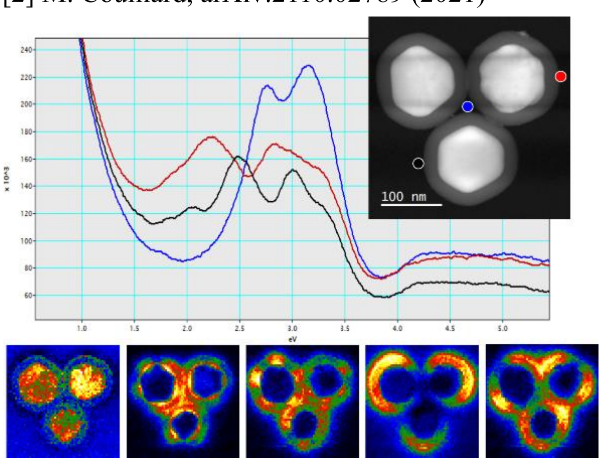

Fig. 1 STEM image and EELS spectra (0.5eV – 5.5eV) for 3 probe locations (top). Maps for 5 principal components extracted from ICA (bottom).

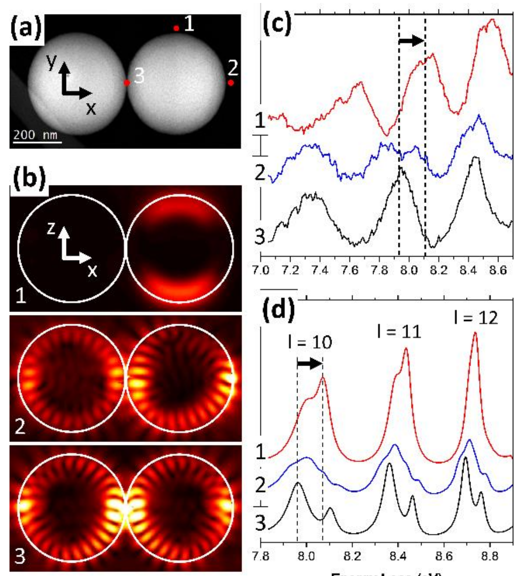

Fig. 2. (a) STEM image of silica bisphere with electron probe locations for simulated |H| maps in (b), experimental spectra in (c) and simulated spectra in (d).

Magnetic configurations of a skyrmionic vortex stabilized in FeGe nanoparticles

Kodai Niitsu[1*]

[1] National Institute for Materials Science, Research Center for Advanced Measurement and Characterization, Tsukuba 305-0047, Japan
*NIITSU.Kodai@nims.go.jp

For magnetic materials, confined geometries have become a versatile platform for engineering their micromagnetic structures. In particular, the engineering of magnetic structures has been intensively studied in two- and one-dimensional ferromagnets, wherein the magnetic domain structures accommodate their own physical and microstructural features into reduced dimensions. In contrast, when confined in zero dimensions (0D), i.e., in nanoparticles, sophisticated magnetic structures can still develop under appropriate conditions, but such visualization remains challenging.

A magnetic skyrmion is an emergent topological magnetic texture with a string-like structure in three dimensions but the form confined in 0D hosts is not evident. I and collaborators performed multiangle electron holography (EH) on chemically synthesized isolated tetrahedral particles of B20-type FeGe to reveal their internal magnetic configurations. Integrating EH observations and micromagnetic simulations uncovered real-space magnetic configurations. Nanoparticles with sizes comparable to the helical wavelength (~70 nm for FeGe) can lead to the emergence of a novel skyrmionic vortex structure (Fig. 1) [1]. This texture shows excellent robustness against temperature without applying a magnetic field; thus, a zero-field skyrmionic ground state is realized for a certain size range of tetrahedral particles.

Acknowledgment: The author thanks to all the collaborators of Y. Liu, A. C. Booth, X. Yu, N. Mathur, M. J. Stolt, D. Shindo, S. Jin, J. Zang, N. Nagaosa and Y. Tokura. This work was partly supported by a Grant-in-Aid for Scientific Research (B) (19H02418) and for Challenging Research (Exploratory) (19K22052) from the JSPS.

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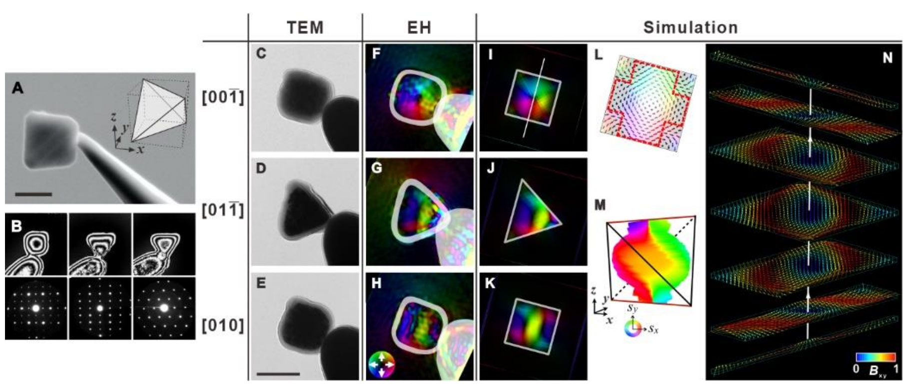

Fig. 1 Skyrmionic vortex confined in a 145-nm FeGe tetrahedral nanoparticle. (A) A scanning electron micrograph showing a nanoparticle. (B) Phase (φ) map in the form of cos(φ) from the [010], $[01\bar{1}]$, and $[\bar{1}1\bar{1}]$ directions (upper column) and corresponding nanobeam diffraction patterns (lower column). (C to E) TEM images along the $[00\bar{1}]$, $[01\bar{1}]$, and [010] directions. (F to H) Observed in-plane magnetic flux distributions for the corresponding projections at 10 K. (I to K) Corresponding projections of the micromagnetic simulations. (L) Magnetic configuration of (I) integrated along the $[00\bar{1}]$ direction. (M) Equi-spin surface with the z-component of magnetization $S_z = 0$. (N) Magnetic configurations in cross-sectional planes normal to the z-axis. All scale bars are 100 nm.

Insights about Atomic-Scale Heteroepitaxy based on Correlative Electron Microscopy of Van der Waals Heterostructures

Hesham El-Sherif[1], Joshua Robinson[2], Nabil Bassim[1,3]

[1.] McMaster University, 1280 Main Street West, Hamilton, ON, L8S 4L8, Canada
[2.] Pennsylvania State University, Old Main, State College, PA 16801, USA
[3.] Canadian Centre for Electron Microscopy, Hamilton, ON, L8S 4L8, Canada

Recently, the synthesis of various 2D materials confined at the epitaxial graphene/SiC interface has been realized through confinement heteroepitaxy (CHet). In this technique, atoms intercalate at the interface of epitaxial graphene (EG) and silicon carbide (SiC) substrates via a thermal evaporation process, typically at 800 °C. While the EG is deliberately damaged using plasma to open intercalation holes prior to the CHet process, the EG is found to be healed after the metal intercalation due to a metal catalytic effect. The CHet process facilitates scalable and environmentally air-stable 2D metals and alloys over millimeter-scale. CHet metals and alloys exhibit novel properties, such as enormous second harmonic generation, superconductivity, and epsilon-near-zero behavior. Understanding the atomic structure of these van der Waals heterostructures yields insights into their growth dynamics, can help explain their properties, and point to factors influencing their scaleability for device applications.

Here, we use the contrast from scanning electron microscope (SEM) images to unlock the metal intercalation positions as well as the EG thicknesses. We applied multiple correlative electron microscopy techniques for understanding the secondary electron (SE) emission that generates the contrast in SEM images. Our correlative electron microscopy includes surface chemical maps in plan view using auger electron spectroscopy (AES) and with Scanning-Transmission electron microscopy (STEM) imaging and electron energy-loss spectroscopy (EELS) obtained from multiple site-specific cross-sections. We also understand the origin of the SE emission related to the heterostructure's local work function by correlating both experimental measurements and theoretical calculations of the surface potential of these heterostructures. We also optimized the SEM imaging conditions – current, voltage, contrast/brightness, and the SE detector – to efficiently augment the differential SEM contrasts, thus providing a rapid characterization path using only the SEM to identify van der Waals heterostructure growth morphology variations. Finally, we examine implications on materials growth based on the local measurement of the top atomic layer of SiC and its positional relationship with the metal 2-D film above it.

Lensing charged particles with the magnetic vector potential

Makoto Schreiber[1,*], Cathal Cassidy[1], Menour Saidani[1], Matthias Wolf[1]

[1] Okinawa Institute of Science and Technology, 1919-1 Tancha, Okinawa, Japan.
[*]mschreib@ualberta.ca

The Aharonov-Bohm (AB) effect [1] demonstrates that charged particles passing in regions free of electromagnetic fields are affected either by the magnetic vector potential in a field-free region or non-locally by an inaccessible field. The effect is usually considered with respect to an infinite-length solenoidal coil. In this geometry, it has been shown analytically that charged particles passing by the solenoid experience a step-shift in their phase. This step-shift in phase has been considered the defining experimental signature of the AB effect.

In the present work [2], we show from theoretical considerations that in the case of a toroidal solenoid coil, a small curvature is present in the phase profile of particles passing through the central hole of the torus (Figure 1a). This curvature is quite small relative to the phase step, and has not been noted previously in either theoretical or experimental work. This phase curvature causes a lensing effect which can be made convex or concave depending on the particle charge polarity and current flow direction (Figure 1b). Additionally, the spherical aberration coefficient is of opposite polarity to the focal length. Although these properties would appear to violate Scherzer's conditions [3], they do not as the underlying principle is different - lensing is caused by the magnetic vector potential rather than a magnetic field. We present experiments with electron holography and Fresnel diffraction through toroidal micro-magnets which support the existence of this effect. The physics of this effect and its consequences are discussed.

References:
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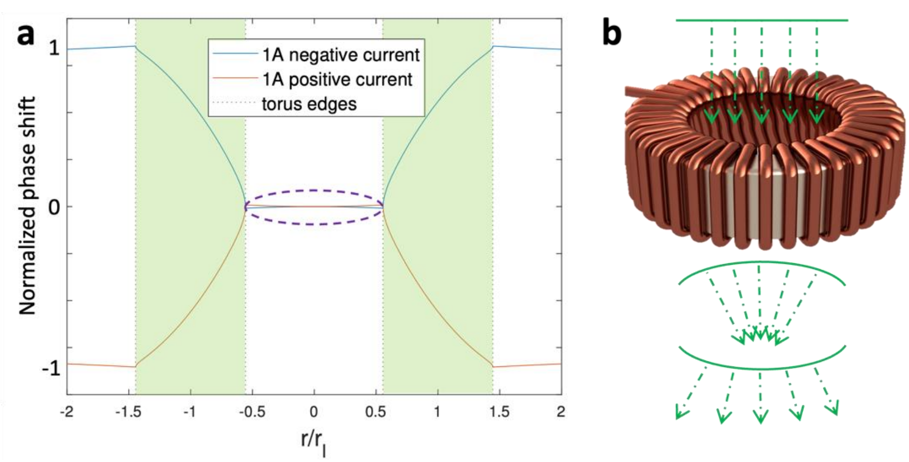

Figure 1. a) Calculated phase profiles for an electron plane wave passing an ideal toroidal solenoid. The light green areas within the dotted vertical lines represent regions inside the torus volume where magnetic field is present. The dotted ellipse highlights the central hole of the torus which is field-free yet exhibits lensing curvature. b) Schematic of how a toroidal solenoid coil can behave as a concave or convex lens for particles with negative or positive charge.

Time-Resolved Measurement in TEM Using a Semiconductor Photocathode

Makoto Kuwahara（桑原 真人）[1, 2*], Takafumi Ishida（石田 高史）[1, 2], Koh Saitoh（齋藤 晃）[1,2], Hideo Morishita（森下 英郎）[3], Shota Kuwahara（桒原 彰太）[4], Toshihide Agemura（揚村 寿英）[5]

[1] Institute of Materials and Systems for Sustainability, Nagoya University, Nagoya, 464-8601, Japan.
[2] Graduate School of Engineering, Nagoya University, Nagoya, 464-8603, Japan.
[3] Research and Development Group, Hitachi, Ltd., Kokubunji 185-8601, Japan.
[4] Department of Chemistry, Faculty of Science, Toho University, Funabashi, Chiba 274-8510, Japan.
[5] Hitachi High-Tech Corporation, Hitachinaka 312-8504, Japan.
*kuwahara@imass.nagoya-u.ac.jp

Dynamic observations of nanoscale materials provide important information for the time evolution of optical couplings, phase transitions, and energy relaxations in a local site. Ultrafast measurement in electron microscopy using pulsed electron beams is a promising candidate for investigating high-speed phenomena on the nanoscale. Furthermore, intense pulsed electron beam in transmission electron microscope (TEM) enables single-shot imaging, which is expected to observe biological specimens or organic material without electron-beam damage due to extracting information before thermalization of injected energy into specimen.

We had begun developing a spin-polarized pulse-TEM (SPTEM), which comprises a semiconductor photocathode with a negative electron affinity (NEA) surface for a polarized electron source and a 30-keV TEM [1]. The SPTEM showed that high-quality electron beam, e.g., high brightness, high spin-polarization, long coherent lengths, picosecond response time and narrow energy width, is realized by the semiconductor photocathode with an NEA surface (NEA-PC) in electron microscope [1,2]. However, observable thickness of specimen in the SPTEM is restricted due to the beam energy. Therefore, we have newly developed time-resolved TEM (TRTEM) using an NEA-PC as pulsed electron source equipped to the TEM column based on a 100-kV TEM instrument (HT7830, Hitachi High-Tech Corp.). The TRTEM is operated with beam energies of up to 100 keV, which improves transmittance in a specimen and provides higher brightness compared with previously developed 30-keV SPTEM. A thin GaAs film on a glass plate (Hamamatsu Photonics Inc.) was employed as a backside-illumination-type NEA-PC, and surface treatments of this photocathode were conducted in an NEA surface preparation system in the TRTEM. The electron beam was extracted from the flat surface of the photocathode with a high electric field gradient of 8 MV/m at an acceleration voltage of 100 kV along with suppression of the space charge effect (as a consequence of the high acceleration field gradient) [3].

Ultrafast phenomena in gold nanotriangles (AuNTs) stimulated by pulsed laser were investigated using transient electron energy-loss spectroscopy (TEELS) technique. This investigation was conducted in the TRTEM under the average pumping laser power of 200 mW. The pulse durations of the pumping laser for specimen irradiation and electron beam emission were 150 fs and 7 ps, respectively. Intensity enhancement and energy width broadening of the energy loss peak were observed at the EEL peaks associated with surface and bulk plasmons in the AuNTs [3]. The TEELS data showed two decay processes on decay-times of 7.8 ps and longer than 100 ps that compensated for the relaxation times of excited surface plasmons using transient absorption spectroscopy. The results also indicated that excited electrons on the surface and in the bulk had the same relaxation processes during both electron–phonon and phonon–phonon interactions.

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Improved Visualization of Bone Ultrastructure in 3D FIB-SEM

Alyssa Williams[1,2*], Tengteng Tang[3], Aurélien Gourrier[4,5], Nabil Bassim[1,3,6], Kathryn Grandfield[1,6]

[1.] School of Biomedical Engineering, McMaster University, Hamilton, Ontario, Canada
[2.] Fibics Incorporated, Ottawa, Ontario, Canada
[3.] Department of Materials Science and Engineering, McMaster University, Hamilton, ON, Canada
[4.] Univ. Grenoble Alpes, LIPHY, F-38000, Grenoble, France.
[5.] CNRS, LIPHY, F-38000, Grenoble, France
[6.] Canadian Centre for Electron Microscopy, McMaster University, Hamilton, ON, Canada
[*]willia16@mcmaster.ca

The nanoscale architecture of bone is founded on the arrangement of collagen fibrils and mineral platelets. Elucidating the spatial relationship between these two constituents faces great challenge largely due to the requirement for nanometer resolution and a large tissue volume of imaging in the range of tens of micrometers. Focused ion beam scanning electron microscopy (FIB-SEM) nanotomography allows for 3D high-resolution imaging of nanoscale features and, coupled with advanced image processing tools, key structural information can be extracted from datasets. Recent FIB-SEM studies indicate that mineral clusters in ellipsoid shapes. Yet, the collagen fibril organization across the ellipsoid and its presence at the periphery are unclear. In this study, human tibial osteonal bone tissue was imaged using FIB-SEM nanotomography and analyzed using fast Fourier transforms (FFT), inverse FFT and 3D segmentation tools. FIB-SEM imaging revealed previously reported mineral clusters or "mineral ellipsoids" and characteristic collagen banding. FFT and inverse FFT analysis was implemented to enhance the 67 nm periodicity associated with type I collagen fibrils, while 3D segmentation was used to elucidate their spatial arrangement with respect to mineral ellipsoids. Collectively, these data show the interwoven structure of the collagen fibrils and demonstrate the fibrils traversing the mineral ellipsoid boundary from the periphery to the center. This work provides greater structural insight into the fundamental building blocks of bone tissue.

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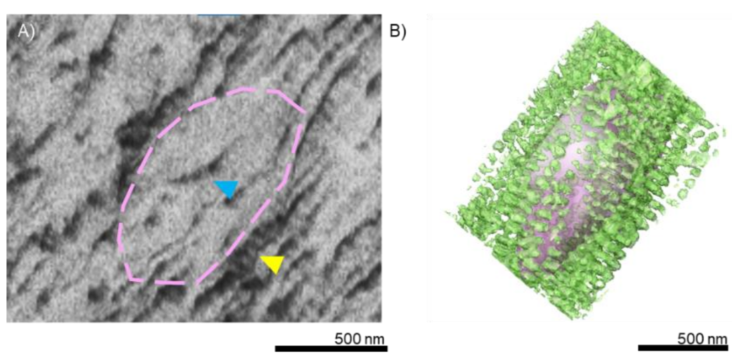

Fig. 1 Collagen fibril and mineral ellipsoid association in osteonal bone tissue. A) Mineral ellipsoid structure with collagen fibrils in (blue arrowhead) and around (yellow arrowhead) the ellipsoid. B) 3D view of collagen fibrils in and around the mineral ellipsoid structure.

The ultrastructure of bone in 3D: A twist of twists

Daniel J. Buss[1], Roland Kroeger[2], Marc D. McKee[1,3], Natalie Reznikov[1,3,4]*

[1]Department of Anatomy and Cell Biology, Faculty of Medicine and Health Sciences, McGill University, Montreal, Quebec, Canada; [2]Department of Physics, University of York, Heslington, York, United Kingdom; [3]Faculty of Dental Medicine and Oral Health Sciences, McGill University, Montreal, Quebec, Canada; [4]Department of Bioengineering, Faculty of Engineering, McGill University, Montreal, Quebec, Canada
*natalie.reznikov@mcgill.ca

Structural hierarchy of bone – observed across multiple scales and in three dimensions – is essential to its mechanical performance. While the mineralized extracellular matrix of bone consists predominantly of carbonate-substituted hydroxyapatite, type I collagen fibrils, water, and noncollagenous organic constituents, it is largely the 3D arrangement of these inorganic and organic constituents at each length scale that endow bone with its exceptional mechanical properties. Based on recent volumetric imaging studies of bone using FIB-SEM tomography and STEM tomography, and earlier works spanning scales up to the level of whole bones, we illustrate the self-similarity of nested structural motifs across several orders of magnitude and link the hierarchical organization of bone to the ubiquity of spiral structure in Nature. We discuss the omnipresence of twisted, curved, sinusoidal, coiled, spiraling, and braided motifs in bone in at least nine of its twelve hierarchical levels – a visualization undertaking that has not been possible until recently with advances in 3D imaging technologies. We hypothesize that the twisting motif occurring across each hierarchical level of bone is linked to enhancement of function, rather than being simply an energetically favorable way to assemble mineralized matrix components. We propose that attentive, further consideration of twists in bone tissue and in the skeleton at different scales will likely enhance our understanding of structure–function relationships in bone.

Acknowledgment: Object Research Systems Inc., Natural Sciences and Engineering Research Council of Canada

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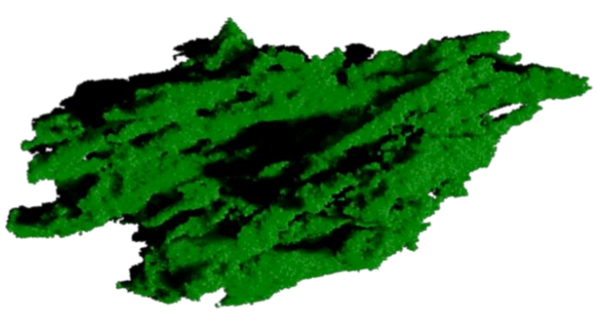

Fig. 1. This figure shows a micrometer-size mineral tesselle – a typical space-filling motif in mature bone, imaged by FIB-SEM Slice&View tomography. The spindle-shaped tesselle shows a gentle longitudinal twist following the texture of co-aligned collagen fibrils in bone. Scale bar equals 500 nm.

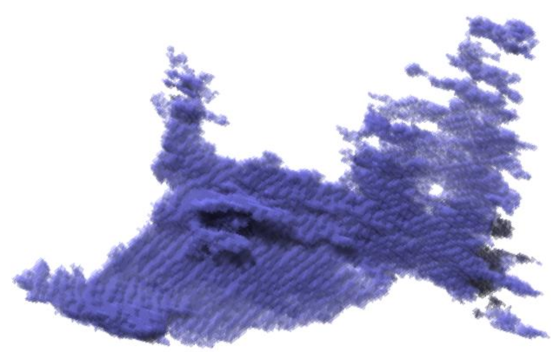

Fig. 2. This figure shows a few hundred nanometer-sized aggregate of bone mineral crystallites, imaged by STEM tomography. The layers of merging and splitting crystallites also subtly twist, and the overall shape of the mineral aggregate is similar to that of the tesselle in Fig 1. Scale bar equals 50 nm.

Bassim, Nabil

Brady, Nadi

Cassidy, Cathal

Coulliard, Martin

Harada, Ken

Hayashida, Misa

Kuwahara, Makoto

Malac, Marek

Mori, Shigeo

Niitsu, Kodai

Reznikov, Natalie

Seki, Takehito

Schreiber, Makoto

Williams, Alyssa