Performing a scientific experiment on an electron microscope involves two components: time spent in the lab preparing the sample and using the microscope; and time spent at the desk performing analysis of the acquired data to reveal and understand the information that it contains. Considerable effort is put into learning to use the microscope properly and ensuring that it is operational and works as expected, but less often is the same attention applied to the data analysis software used post-acquisition. This software can be commercial, and is typically very costly with a small set of fixed data processing routines; or open source or open access, and frequently unreliable or difficult to use by anyone except the original programmer. The application of modern software engineering documentation practices to the development and design of scientific software, such as the programs used for data analysis, is meant to ensure that each piece of software is documented to the same standard, and therefore more easily understandable and useable by an outside party or by developers in the future.

The suggested methodology outlines a set of documents (requirements, design, code, and verification) to guide the developer through the software design process [1] and to help the user of the software understand the purpose, capabilities, and limitations of the software, promoting trust in the results obtained through using the program. Key software engineering principles promoted include the separation of concerns, abstraction, and design for change. Recommended tools include version control, issue tracking, unit testing frameworks, and documentation generation. Two case studies are presented. The first, SpectrumImageAnalysisPy [2], is an open-source Python library of functions used for visual and numerical analysis of 3D spectrum images. While originally designed for electron energy loss spectroscopy and cathodoluminescence data, the software is highly modular and easily adaptable to allow the analysis of any spectrum image data, from any spectromicroscopy technique. The modularity of the program enables new functionality, such as a new data processing routine, to be swiftly incorporated into the overall hierarchy of the software. The second case study is an open-source interactive software, STEM_Moire_GPA [3], for converting STEM Moire holograms into deformation maps using the Geometric Phase Analysis (GPA) algorithm. The ability to quantitatively track the errors from the GPA process is demonstrated at the module level; such information is essential to judge the proper execution of a processing method and must be shared with the informed user. The source of errors can also be tracked and revealed that the Fourier Transform algorithm and the masking process are major contributors to the error propagation. Document-driven methodologies, used to design data analysis software for electron microscopy, provide immense benefits and could be the next move for a community already facing challenges in reliably and accurately processing the large high-resolution datasets acquired on today's instruments.

**References**


Surface modification of 3D-printed Ti alloys to improve osseointegration

Chiara Micheletti¹, Bryan E. J. Lee², Simon Coulson¹, Hatem Zurob¹, Kathryn Grandfield¹,²
¹ Department of Materials Science and Engineering, McMaster University, Hamilton, Canada
² School of Biomedical Engineering, McMaster University, Hamilton, Canada

Introduction
Titanium and titanium alloys are commonly employed materials for hard tissue replacements in bones and dental implants, thanks to their biocompatibility and high corrosion resistance. However, a non-negligible percentage of implants is prone to failure, mainly due to poor osseointegration, namely poor implant-host tissue interaction¹. In particular, surface topography and micrometre roughness have been shown to have a significant effect on cell adhesion, proliferation and migration². The objective of the study described is to achieve microscale surface topography by manufacturing titanium alloys using selective laser melting (SLM), which generates micro-rough surfaces, thanks to the presence of spherical microparticles randomly arranged on the surface³. Moreover, this manufacturing technology allows for further surface modification treatments aimed at the creation of nanoscale features. Therefore, a second objective of the study is to electrochemically anodize the 3D-printed samples to produce an array of titanium oxide nanotubes on their surface. The hypothesis is that the dual topography obtained in this way will improve cellular responses and osseointegration of Ti alloys-based implants³.

Materials and Methods
Different titanium alloys, Ti-6Al-4V (Ti64), Ti-1Al-8V-5Fe (Ti185) and Ti-5Al-5Mo-5V-3Cr (Ti5553), were 3D-printed by SLM. The topography of the 3D-printed samples was visualized using scanning electron microscopy (SEM). Roughness was measured using a focus variation microscope. Surface wettability was evaluated by water contact angle measurements. In vitro tests were performed using the Saos-2 cell line. Cell metabolism and alkaline phosphatase activity were evaluated with AlamarBlue and alkaline phosphatase (ALP) activity assays. The adhesion and morphology of the cells on the substrates were evaluated using SEM. Electrochemical anodization of titanium samples used a solution of ethylene glycol with 0.3 wt% of NH₄F and 2 vol% of H₂O.

Results
The SLM process resulted in surfaces with micro-scale roughness in the range of 15-35 µm (R₅). SEM imaging of Ti64 and Ti5553 samples showed the presence of microspherical particles arranged on the surface, while Ti185 samples had irregular surface features, which is due to the lower quality of the powder feedstock used compared to the other two alloys (Figure 1). Water contact angle measurements of Ti64 revealed a certain degree of hydrophobicity, while Ti185 and Ti5553 appeared to be slightly hydrophilic. In vitro experiments revealed cell metabolism and ALP activity after 3 days, which were higher in case of Ti5553 especially if compared to Ti185. According to in vitro tests results, SEM imaging showed the presence of cells adhered to the surface (Figure 2).

Discussion and Conclusions
This study reached its objective of using SLM to obtain surfaces with microscale roughness, as proved by the characterization performed on the samples. In vitro tests and following SEM imaging provided encouraging results in support of the hypothesis of having better cellular responses with surfaces obtained by SLM. Thus, this technology seems to be a valid method to optimize implant surface for improved osseointegration, and it is believed that subsequent electrochemical anodization of the samples synthesized by SLM will allow for a combination of their characteristic micro-rough surfaces with the so-produced titanium oxide nanotubes. In fact, the creation of a dual topography is believed to be an important surface modification strategy to achieve an optimal osseointegration and to increase the success of Ti-based implants³.
References

Figures

![Figure 1. SEM micrographs of 3D-printed Ti64 (left), Ti5553 (centre) and Ti185 (right).](image1)

![Figure 2. SEM images of cells on 3D-printed Ti5553 (left) and Ti185 (right).](image2)

Acknowledgements
We would like to thank NSERC for funding, and the Canadian Centre for Electron Microscopy, a facility supported by NSERC and other government agencies, staff for microscopy assistance. *In vitro* studies were performed at the Biointerfaces Institute at McMaster University.
Plasma Focused Ion Beam Curtaining Artifact Correction by Fourier-Based Linear Optimization Model

Christopher W. Schankula¹, Christopher K. Anand¹ and Nabil D. Bassim²

¹. Department of Computing and Software (CAS), McMaster University, Hamilton, Ontario, Canada
². Department of Materials Science and Engineering (MSE), McMaster University, Hamilton, Ontario, Canada

Focused ion beam scanning electron microscope tomography uses a destructive slice-and-image technique for obtaining three-dimensional structural information. Xenon-based plasma focused ion beam technology replaces the traditional gallium source, resulting in much higher material removal rates. This increases the feasibly analyzable volume of material [1], allowing for richer microstructural and statistical analyses.

However, in heterogeneous samples, plasma milling rates vary, causing vertical ripples (“curtains”) to develop on the cutting surface of the sample. In backscatter electron images, curtains manifest as dark or light vertical streaks. This leads to an inaccurate automated quantitative analysis of the image tomogram. A “rocking mill” technique can be used to reduce these artefacts by varying the cutting angle of the plasma beam. The result is images with less pronounced curtains; however, curtains are created at two discrete angles corresponding to the milling angles used.

We present a new algorithm aimed at correcting these multi-angle artefacts using a Fourier-based linear optimization model implemented using Anaconda Python 3.5.2 and the Gnu Linear Programming Kit (GLPK). The algorithm was testing on an ultra-high performance concrete tomogram taken on an FEI PFIB taken with a voxel resolution of approximately 48nm × 48nm × 50nm. A rocking mill was used during imaging, creating curtains at approximately 1° and -7° rotations from the vertical.

Comparisons of results with similar algorithms show good curtaining correction performance. Future work includes taking advantage of the parallelisable nature of the algorithm and other techniques to increase the efficiency of the algorithm [2].

References:

[2] The authors acknowledge funding from NSERC and McMaster’s MSE department. We thank Prof. Kay Wille for preparing the UHPC sample and Ron Kelley & Brandon Van Leer of FEI-Thermo Fisher for the imaging, as well as Dr. Jan Henrik Fitschen from the University of Kaiserlautern for agreeing to run their algorithm on our data.
Investigation of Gap Zone Mineral in Bovine Bone using Transmission Electron Microscopy

D. Binkley\textsuperscript{1}, L. Lou\textsuperscript{2}, K. Grandfield\textsuperscript{3,4} and H. Schwarcz \textsuperscript{4,5}

\textsuperscript{1}School of Interdisciplinary Sciences, McMaster University, Canada
\textsuperscript{2}Department of Health Science, McMaster University, Canada
\textsuperscript{3}Department of Materials Science and Engineering, McMaster University, Canada
\textsuperscript{4}School of Biomedical Engineering, McMaster University, Canada
\textsuperscript{5}Department of Geography and Earth Sciences, McMaster University, Canada

Bone is a naturally occurring composite material that is comprised of both mineral (apatite) and collagen. Many models have been proposed to describe the intricate interplay between the mineral and collagen \textsuperscript{1,2}, with most recent studies suggesting that the mineralized component of bone surrounds collagen fibrils in plate-like shapes, a feature termed mineral lamellae \textsuperscript{3}. It is widely accepted that collagen fibrils have a gap zone, a region of space approximately 40 nm wide between each collagen molecule \textsuperscript{4}; however, the exact composition of the gap zone is currently debated. Research has suggested that the material within the gap zone is crystalline hydroxyapatite, as determined from elemental analysis \textsuperscript{5}. However, the presence of calcium and phosphorous does not confirm that the material in the gap zone is completely crystalline. This study aims to investigate the crystallinity of the gap zone mineral through dark-field transmission electron microscopy (TEM).

A longitudinally sectioned femoral diaphysis of a mature bovine bone was prepared for TEM using ion milling techniques. Bright field and dark-field images to highlight the 002 reflection were obtained using a Philips CM-12 microscope operated at 120 kV. Images were aligned and stacked using ImageJ to reveal crystallinity information in the gap zone.

Preliminary results (Fig. 1) suggest that some areas in the gap zones are not crystalline in nature, supporting our hypothesis that the gap zone material may not be crystalline apatite. It can instead be hypothesized that this material could be amorphous calcium phosphate (ACP). Further studies, including high-resolution TEM and spectroscopy are planned to determine the composition of the gap zone material. If indeed this material is not crystalline, this work will contribute to more accurate
models of the ultrastructure of bone, and the interplay between collagen and mineral components in mineralized tissues.

Figure 1. BF-TEM image (a) overlapped with corresponding DF images along the 002 direction that have been colored red (b,c). Arrows indicate the gap-overlap zones, and lack of material aligned in the c-direction in the gap zone although mineral lamellae are clear.

References

Keywords: bone, ultrastructure, gap zone, crystallinity, TEM
Electron Microscopy of 2D Gallium, Indium and GaN and InN Layers Synthesized by Confinement Heteroepitaxy (CHet) Technique

Hesham El-Sherif¹, Brian Bersch², Natalie Briggs², Joshua A. Robinson² and Nabil D. Bassim¹

¹ McMaster University, 1280 Main Street West, Hamilton, ON, L8S 4L8, Canada
² Pennsylvania State University, Old Main, State College, PA 16801, USA

Two Dimensional (2D) materials have great potential because of many superlative properties: spin-orbit coupling, massless Dirac fermions, flexibility, thermal and chemical stability. Besides graphene, many other 2D materials with a hexagonal structure, such as h-BN and more recently 2-D GaN, have been synthesized and many more predicted by simulation. The synthesis of 2D GaN was first reported in 2016 by applying Confinement Heteroepitaxy (CHet). In this technique, Silicon Carbide (SiC) substrate is graphitized and deliberately damaged using oxygen plasma to provide an intercalation path between two graphene sheets. Then, Ga flakes located below the SiC substrate are evaporated in a tube furnace at 700 °C. The Ga atoms intercalated the defected graphene and deposited between the graphene and the SiC substrate. To complete the half reaction, nitrogen is introduced by ammonia annealing at 700 °C.

In this study, we use electron microscopy to characterize the microstructure and chemistry at the atomic scale of Ga, In, GaN and InN, using scanning transmission electron microscopy (STEM). Focused ion beam milling was employed for site-specific characterization to examine the role of surface steps and graphene damage sites on the intercalation behaviour. TEM techniques such as bright-field, dark-field, aberration-corrected STEM-HAADF imaging, electron energy loss spectroscopy (EELS) and Energy Dispersive X-ray Spectroscopy (EDS) mapping were all applied to characterize the interface.

The results of this study had three observations. Firstly, HAADF images corresponding to “dark” gray levels in SEM imaging confirm the existence of 1-2 atom thickness of In encapsulated between the graphene and the SiC substrate. Secondly, we found that in some case, the oxygen plasma also intercalated oxygen to the substrate, altering the crystallinity of the formed 2-D layers. Finally, Both EDS and EELS maps show an In-peak encapsulated between the SiC substrate and a carbon layer. For InN layers, mapping of Nitrogen “N-K” was challenging and ambiguous because of the overlapping between In-M edge (443 eV), N-K edge (401 eV) and a large carbon background (284 eV). We continue to explore the processing envelope, namely by studying growth at lower temperatures, and using different plasma processing techniques, which could enhance nitrogen incorporation.

Figure (a): HAADF image from the interesting shows 2 atom thickness layer of Indium Nitride (white color) covered with graphene sheet.

Figure (b): EELS spectrum showing C-K, N-N, In-M, O-K, and Si-K edges, the magnification on the N-In-O region shows that there is N edge at 401 eV and a delayed Indium edge at 443 eV.
Surface Plasmon Resonance Modes of Single and Coupled Upright Split Ring Resonators

Isobel Bicket, Edson Bellido, Sophie Meuret, Toon Coenen, Albert Polman, and Gianluigi Botton

1. Dept. Materials Science and Engineering, McMaster University, Hamilton, ON, Canada
2. AMOLF, Science Park 104, 1098 XG Amsterdam, The Netherlands
3. DELMIC BV, Thijsseweg 11, 2629 JA, Delft, The Netherlands
*email: bicketic@mcmaster.ca

The split ring resonator (SRR) is a well-studied device known for its ability to produce a magnetic dipole moment [1]. When the SRR is of sub-wavelength dimensions, its surface plasmon resonance modes allow for it to be used to create a left-handed metamaterial, which possesses a negative permittivity and permeability and provides many interesting options for the manipulation of light [2]. The planar SRR, fabricated in a planar geometry on the substrate, has received much attention scientifically, but holds some drawbacks in terms of the coupling geometry between the magnetic dipole moment of the SRR and the magnetic field of light. Fabricating the SRR in a vertical geometry is more challenging, but reveals more opportunities for coupling the magnetic dipole moment to photons or to neighbouring plasmonic structures.

We present a correlative study using monochromated electron energy loss spectroscopy and cathodoluminescence to probe the full spectrum of plasmon resonance modes in a single upright SRR [3]. We show detection of the magnetic dipole moment and provide a full classification of the higher order plasmon resonance modes present on the pillars of the upright SRR. We also show the full polarization state of the emitted light of each resonance mode using polarization-resolved cathodoluminescence. We find that the higher order resonance modes are coupled rim modes present on the pillars of the upright SRR and we discuss the advantages of using the correlative techniques of EELS and CL in conjunction with each other.

References
Synthesis of Gold Nanoparticles from Aqueous Solutions by Dielectric Barrier Discharge (DBD) Plasma Treatment

Jean-François Sauvageau¹,²,³ (MSc Student), Marc-André Fortin¹,²,³* (PhD, Ing)
¹Département de Génie des Mines, de la Métallurgie et des Matériaux, Université Laval, Québec
²Laboratoire de Biomatériaux pour l'Imagerie Médicale (BIM), CR-CHUQ, Québec
³CQMF: centre Québécois des Matériaux Fonctionnels (CQMF), Université Laval. Québec
jean-francois.sauvageau.1@ulaval.ca

Plasma reactors operating at atmospheric pressure such as dielectric barrier discharge (DBD) plasma systems can be used for the fabrication of nanoparticles (NPs).¹ They are remarkable candidates for the treatment of large volumes of industrial aqueous solutions, such as electrolytic baths and effluents from the gold mining industry, for the fast and efficient recovery of gold ions in nanoparticle form.² For this study, two types of aqueous solutions were plasma-treated using the same methodology. The first solution was prepared from a gold chloride salt (HAuCl₄), while the second precursor solution was directly collected from an industrial gold electroplating process (KAu(CN)₂). Physicochemical studies were conducted on both samples of NPs in order to compare the influence of the composition of the precursor solutions on the properties of the synthesized NPs. The size, morphology and crystal structure of the NPs were studied in transmission electron microscopy (TEM), while the hydrodynamic diameter of the NPs was measured in dynamic light scattering (DLS). The chemical composition of the NPs was analyzed in X-ray photoelectron spectroscopy (XPS) and in Fourier-transform infrared spectroscopy (FTIR). Finally, the elemental recovery of gold ions as NPs was assessed by microwave plasma atomic emission spectroscopy (MP-AES). The mean diameter of the Au NPs observed in TEM was 24.7 nm for the HAuCl₄ precursor solution and 14.8 nm for the electroplating solution. Both types of NPs were mostly spherical and exhibited a face-centered cubic crystal structure. Aggregates of NPs were observed in DLS for both samples. For 15 min of plasma treatment, calculations from MP-AES results revealed that 33.2% of the Au ions were recovered as Au NPs. Overall, the results confirm the quick and efficient recovery of gold ions as NPs possessing distinct physicochemical properties by DBD plasma treatment of industrial and synthetic aqueous solutions.

References
2) Bednar, N.; Matovic, J.; Stojanovic, G., Properties of surface dielectric barrier discharge plasma generator for fabrication of nanomaterials. J. Electrost. 2013, 71 (6), 1068-1075.
Improved Fiber-Chip Coupling in Waveguides by Focused Ion Beam Modification

K. M. Kiani, A. P. Knights, and J.D.B. Bradley, Department of Engineering Physics, McMaster University
S. Norris, N. D. Bassim, Department of Materials Science and Engineering, McMaster University

We demonstrate efficient optical coupling between a single mode fiber (SMF) and integrated silicon nitride (Si$_3$N$_4$) waveguide using a focused ion beam (FIB) milling process. This led to efficient matching of the fundamental mode of the fiber to that of waveguide. We have also measured the coupling efficiency using a laser beam at 1550 nm wavelength. The optical coupling using a tapered fiber when the end facet has been FIB milled, is 35% on average more efficient than with coupling between the fiber and the waveguide without FIB milling.

Figure 1 - On left, as received waveguide end facets. On right, waveguide end facets after FIB processing.

Figure 2 - Cross section SEM image of Si$_3$N$_4$ waveguide

It has been shown that an improved optical coupling efficiency between a single-mode fiber and Si$_3$N$_4$ waveguide can be achieved by FIB milling. With plasma ion sources and automation, this process has the potential to be developed into a wafer-scale technique which could easily be implemented by photonic chip manufacturers.


Thin-Film Dewetting Prevention by Ion Implantation

Longxing Chi1 Nabil Bassim1
1. McMaster University, Hamilton, Ontario, Canada

Dewetting is a nontrivial challenge in thermal and chemical processing of thin films.1 Conventional methods in dewetting prevention concentrate on interfacial modification and surface capping.1-7 The first approach suppresses dewetting by placing a new chemical species at the interface between films and substrates to decrease their interfacial energy; the later one achieves the goal through exerting additional tensile strain on the film with a capping layer.

Here we develop a new way to prevent dewetting by doping with ion implantation. Silicon atoms of different doses are used at dopant-level concentrations (1, 2, 4×10^{15} atoms/cm^2) implanted into 100 nm Ag thin films grown directly on single crystal sapphire substrates by high-vacuum sputtering to research their contribution to anti-dewetting. SEM, TEM, AFM and Raman are used to characterize film morphology before or after vacuum annealing treatment (803K, 30 - 1300 min). The interfacial energy of the Ag film during heat processing is calculated based on Miller’s close-packed hexagonal cylinder model; influence of dopant ions on grain growth is measured as well as simulated according to Cahn’s solute drag theory.

It is found that 10^{14}cm^{2} Si^{4+} ions are sufficient to stop 100nm-thick Ag film from rupture even after 21 hours annealing, which can be attributed to those additional ions successfully retard Ag grain growth during heating resulting from their solute drag effects. Downsized Ag grains significantly decrease grain diameter-to-height ratio of the film, thereby enabling the continuous Ag film to possess a lower interfacial energy than the discrete Ag islands and to maintain its initial shape. Furthermore, a simulation of the solute drag effect on grain growth successfully matches our experiments, proving that small amounts of dopants are capable of decelerating grain growth and shrinking average grain size and thus preventing the thin film dewetting. These results show the potential for novel contact processing for advanced semiconductor device applications.

Electrospun Polymer-Niobium Containing Composite Scaffolds for Bone Regeneration

Natália H. Marins1,2, Ricardo M. Silva1,2, Bryan E. J. Lee3, Neftalí L. V. Carreño2, Kathryn Grandfield1,3*

1Department of Materials Science and Engineering, McMaster University, Hamilton, Canada
2Graduate Program in Materials Science and Engineering, Federal University of Pelotas, Pelotas, Brazil
3School of Biomedical Engineering, McMaster University, Hamilton, Canada

The development of biocomposite scaffolds with enhanced properties has attracted great attention for repairing and regenerating bone tissue. Recently, scaffold composites of organic matrices and bioactive inorganic nanofillers have been studied as promising candidates for bone tissue engineering applications due to their improved physical, biological, and mechanical properties. Electrospun membranes have been used in bone tissue engineering because they can provide these features in addition to promoting cell migration and proliferation. Electrospinning is a simple method and it has been used to create continuous polymeric nanofibres structures very similar to extracellular matrix. Polycaprolactone (PCL) and gelatin (GL) are two biodegradable and biocompatible polymers widely applied in medical applications1. Over the last decade, hydroxyapatite (HA) has been commonly used in biocomposites for bone regeneration due to its bioactive and osteoconductive properties. Several types of nanoparticles are being used in biomaterials, but little attention has been given to niobium pentoxide (Nb2O5)-based (Fig. 1A-B) biomaterials although they have shown improved alkaline phosphatase (ALP) activity, cell spreading, and increased collagen production2. The objective of the present work is to develop an electrospun PCL/GL/HA/Nb2O5 (Fig. 1C) to improve bone regeneration.

Figure 1. SEM micrographs of Nb2O5 nanoparticles (A), PCL/GL/HA/Nb2O5 scaffold (C), and HRTEM micrographs of Nb2O5 nanoparticles (B).

References:
Neutron scattering facilities at the McMaster Nuclear Reactor: a complimentary probe to electron microscopy

Patrick Clancy (1), Zin Tun (2), Maikel Rheinstadter (1), Chris Heysel (3), Bruce Gaulin (1,4)

1. Dept. of Physics & Astronomy, McMaster University, 1280 Main St. West, Hamilton, Ontario L8S 4K1
2. Canadian Neutron Beam Centre, Chalk River, Ontario KOJ 1JO
3. McMaster Nuclear Reactor, 1280 Main St. West, Hamilton, Ontario L8S 4K1
4. Brockhouse Institute for Materials Research, 1280 Main St. West, Hamilton, Ontario L8S 4K1

The McMaster Nuclear Reactor (MNR) is Canada’s only source of neutron beams for materials research. MNR is currently home to two neutron scattering beamlines: the McMaster Alignment Diffractometer (MAD) and the McMaster Small Angle Neutron Scattering beamline (MacSANS, coming online in Spring 2019). In many respects, neutron scattering is highly complimentary to electron microscopy, providing a bulk, momentum-space probe of structure and magnetism on length scales ranging from ~0.1 to 100 nanometers. This poster will describe current (and future) neutron scattering facilities at the MNR, and discuss how these instruments may be of use to CCEM users.
Focused Ion Beam Fabrication of a Chiral Infrared Polarizer

S. Norris, N.D. Bassim
Department of Materials Science and Engineering, McMaster University, Hamilton ON L8S 1J1
norriss@mcmaster.ca

T. Folland, J.D. Caldwell
Department of Mechanical Engineering, Vanderbilt University, Nashville TN 37212

As fiber-optic communication becomes more integrated and all-optical circuits become more complex, the need for compact and inexpensive micro-optical components has increased rapidly. Many valuable new devices have been made from patterned structure-based metamaterials. Components active in the relevant infrared (IR) wavelength regimes require nanometer-scale features in three dimensions. A specifically suited substrate material is required as well, making fabrication by conventional means challenging. Resistless, direct-write focused ion beam (FIB) patterning can fulfill these requirements and is widely used as a robust and versatile tool for prototyping metamaterials.

Here we present a novel, chiral metamaterial fabricated via FIB that transforms an unpolarized mid-infrared source into either right-handed circular (RCP) or left-handed circular (LCP) polarized light. Simulations of the electromagnetic near-field associated with this structure verify a net change in polarization, though the efficiency is highly dependent on the fabrication process.

Each array element consists of opposing 90° stepped wedges. Each is comprised of three steps in Z, with the deepest step penetrating through the substrate (see fig 1). The directionality of this “spiral staircase” provides chirality to the overall structure. For instance, if the direction of the descending steps is reversed from clockwise to counterclockwise, the transmitted light would be LCP instead of RCP.

A substrate with high anisotropy is required to eliminate optical loss from reflection and interference. The substrate material chosen was hexagonal boron nitride (hBN). hBN has the advantage of exhibiting extreme birefringence throughout the IR and, in specific spectral ranges, is naturally hyperbolic (see fig 2). Dose, dose rate, and etching gas selection are optimized to fabricate highly accurate devices by direct-write FIB.

3 J.D. Caldwell et al., Nature Communications 5, 5221 (2014).
Untangling the electron beam damage from specimen damage in the FIB-SEM system

Weiwei Zhang¹, Lis Melo², Dr. Adam Hitchcock² and Dr. Nabil Bassim¹

¹ McMaster University, Department of Materials Science and Engineering, ² McMaster University, Department of Chemistry

Abstract

When coupled with focused ion beam (FIB), scanning electron microscopy (SEM) expands FIB applications to include high precision site-specific lamella sample preparation for transmission electron microscopy (TEM) and FIB tomography. Though SEM has often been considered as a relatively non-destructive technique for materials surface morphology characterization and FIB is used to locally image within a structure, radiation damages caused by both electrons and ions can strongly affect materials chemistry and properties. This is especially significant for soft materials. There are known strategies, like lowering beam voltage and current during final polishing steps and optimized patterning for milling. However, damage mechanisms for specific materials have not been explored in detail and the relationship between beam parameters and damage mechanisms remain unknown. Previous work from our group explored damage mechanisms and tried to untangle the different levels and types of damage from the ions and electrons. We found that the ions and electrons result in distinct chemical changes to soft material damage in FIB-SEM. In this research, we separated the damage from the electrons and ions by irradiating the samples with different beam sources, and later characterizing with near edge X-ray absorption fine structure (NEXAFS) in a scanning transmission X-ray microscopy (STXM) in a synchrotron. We found that, even with low electron fluence condition, the electron beam can still damage some polymer samples. We also found that even with the same fluence, larger electron beam electron fluence rate can lead to greater damage. We present here a detailed study by FIB-SEM and STXM of polyvinylidene fluoride (PVDF), poly(methyl methacrylate) (PMMA) and Embed812 epoxy resin.
Scanning electron microscope 3D surface reconstruction via optimization

Yasamin Sartipi, Weiwei Zhang, Samuel Norris, Hesham El-Sherif, Aidan Ross, Nabil Bassim, Christopher Anand

McMaster University, Hamilton, Ontario, Canada

Abstract

In this paper, we present an optimization method for three-dimensional (3D) reconstruction of scanning electron microscope (SEM) images based on shadowing effects. This method could extend the use of SEM images from surface morphology to surface topography and roughness. Therefore, there would be less need to perform further tests such as atomic force microscopy (AFM), which is quite challenging and time consuming, so as to analyze the 3D shape of surfaces. Many efforts have been made to measure depth in SEM images. Stereo-photogrammetry is one of the well-known methods to generate 3D images out of SEM 2D images. This approach, however, requires collecting hundreds of SEM 2D images which takes plenty of time. Using our proposed method, one could take advantage of both Backscattered Electron (BSE) and Secondary Electron (SE) modes of an Everhart-Thornley Detector (ETD) from different angles accessible via rotation of the sample stage. The optimized model is capable of creating 3D images from noisy SEM 2D images which could be capture quickly and consequently decreases the time of imaging. Furthermore, the method allows us to use only eight BSE-mode SEM images in order to achieve a 3D model. Subsequently, SE-mode images could be utilized to modify the small features and details on the output of the optimization problem. We have tried the optimized method on images of fabricated silicon test objects. Our results show the obtained 3D images nearly correspond to the AFM results, taking the limitations of our AFM probe into account.