Ontario Centre for the Characterization of Advanced Materials (OCCAM)

World-Class, Open-Access Materials Characterization Facility

"solving problems by enabling information and collaborative research"

What is OCCAM? •$23 million, multi-disciplinary material characterization facility housed and co-managed by the Departments of Chemical Engineering & Applied Chemistry and Material Science & Engineering at the U of T •internationally-unique, world-class cadre of leading-edge surface characterization, electron microscopy, and scanning probe tools, supported by collaborative research staff

What is OCCAM's role? •to provide leading-edge characterization capabilities (both instrumental and expertise) for the materials-based research community in the GTA and beyond •to serve as a training facility, exposing students, post-docs, industrial/government researchers, and faculty to world-class characterization kit in a structured and supported research environment •to serve as a nexus for materials research and collaboration

Who makes use of OCCAM? • >300 separate research projects/year, representing a diverse portfolio of academic, government, and industrial research •impact at all points along the innovation chain , from fundamental research, to product, process, and technology development

Fields of application and expertise:

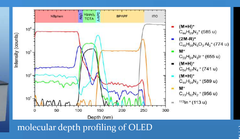
- •catalysis
- •medical devices and biomaterials
- •biology
- •microelectronics
- •semiconductors
- •electronic and communication materials
- •energy materials (harvesting, storage, etc...)
- •geology
- •mining
- •polymers
- •corrosion
- •sensors
- •forestry/pulp & paper
- •metallurgy
- •structural materials
- •environmental studies
- •nuclear sciences
- •aerospace engineering
- •coating industry
- •dentistry
- •ceramics
- •*and many, many more*

Surface Characterization

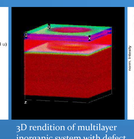
The surface properties of a material greatly influence its interaction of its environment. Understanding these properties is critically important. Surface characterization infrastructure is optimized to probe the surface/near-surface region of materials (~top 50nm).

Time-of-flight Secondary Ion Mass Spectrometry: mass analysis of ions ejected by sputtering ion beam. Optimized for surface specific characterization (1-2 monolayers). Depth profiling with multiple ion sources. Highly spatially localized (laterally (<100 nm) and in-depth (1 nm)) elemental, isotopic, and chemical (molecular) information with very high accuracy and sensitivity.

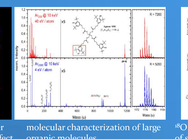
ION-TOF ToF-SIMS IV (to be upgraded, vendor TBD) • Bi liquid metal ion source and several complementary mono-atomic and polyatomic ion sources •time of flight analysis (low duty cycle for surface characterization) •charge compensation •heating/cooling •pulsed secondary electron detector



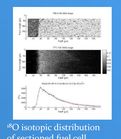
molecular depth profiling of OLED



3D rendition of multilayer inorganic system with defect



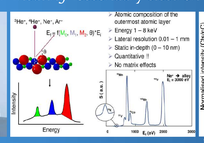
molecular characterization of large organic molecules

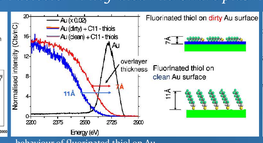


^{18}O isotopic distribution of sectioned fuel cell

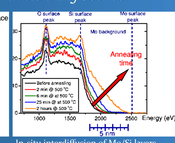
Low Energy Ion Scattering: energy analysis of low-energy (few keV) noble gas ions scattered from surfaces, to provide elemental and structural information regarding the absolute **top atomic layers**.

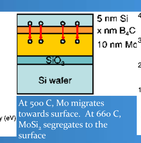
vendor TBD • noble gas ion source •high sensitivity ion energy analyzer and parallel energy detection (enabling non-destructive surface characterization) •time of flight mass filter •heating •secondary electron detector •integrated microwave plasma cleaning source



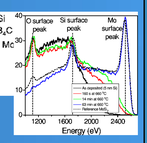


behaviour of fluorinated thiol on Au





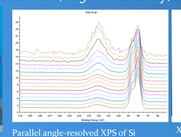
In-situ interdiffusion of Mo/Si layers.



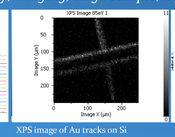
At 500 C, Mo migrates towards surface. At 660 C, $MoSi_2$ segregates to the surface

X-ray Photoelectron Spectroscopy (3 tools): energy analysis of photo-ejected electrons. Quantitative elemental and chemical information from the top 8-10 nm, quantitative depth profiles of organic and inorganic systems via angle-resolved photoemission measurement (~2-10nm) or sputter erosion. Imaging at spatial resolution of ~5um.

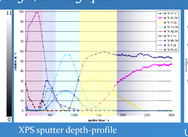
Thermo Fisher Scientific ThetaProbe and K-Alpha, imaging XPS vendor TBD •Al/Mg/Ag x-ray sources •mono-atomic and polyatomic ion sources (sputtering) •charge compensation •heating/cooling •azimuthal rotation (high resolution profiling) •parallel angle resolved detection (ThetaProbe) •field emission electron source (low spatial resolution Auger characterization and SE imaging) •multiple instruments, each emphasizing a different application: angle-resolved characterization, high-sensitivity/imaging, large sample/high/throughput



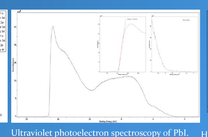
Parallel angle-resolved XPS of Si surface oxide



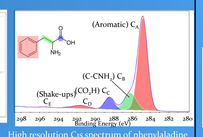
XPS image of Au tracks on Si



XPS sputter depth-profile



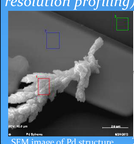
Ultraviolet photoelectron spectroscopy of PbI. Insets show onset and Fermi edge regions



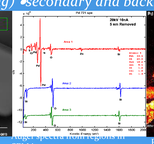
High resolution C1s spectrum of phenylaladine

Auger Electron Spectromicroscopy: providing quantitative elemental and chemical state information from the top 0.5-5 nm of the sample. With ion sputtering sources, quantitative depth profiling is possible. Chemical imaging at spatial resolution of ~10 nm.

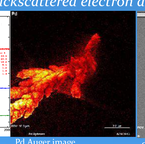
vendor TBD •high resolution, field-emission electron source •mono-atomic ion sources (sputtering) •charge neutralization •azimuthal rotation (high resolution profiling) •secondary and backscattered electron detectors



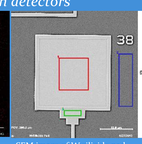
SEM image of Pd structure



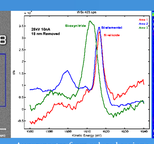
SEM image



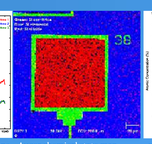
Pd Auger image



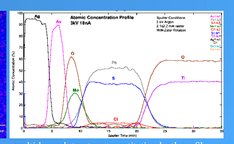
SEM image of W silicide pad



Augerspectra from colored regions



Auger chemical state map



multi-layered structure, quantitative depth profile

Sample Preparation and Handling

Critical to modern material characterization is preparing and handling materials for analysis. This includes not only transferring samples to/between analytical tools with minimal perturbation, but also preparing samples for analysis by means consistent with material properties and analytical goals.

Sample preparation:

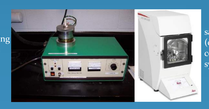
Metallographic polisher/ grinder

Targeted small area sectioner/ polisher

bench-top ion milling system

Cryo-ultramicrotome

stand-alone and integrated prep chambers, supporting heatin/cooling, gas dosing, crystal cleaving, auxiliary chambers



sample coating (carbon and conductive metal systems

Sample transfer: transfer of samples in controlled environments (inert/vacuum/cold) to/from labs/glove-boxes, as well as between analytical instruments

XPS sample transport shuttle

SIMS sample transport shuttle

tool-mounted glove-box

FIB bulk sample air protection holder

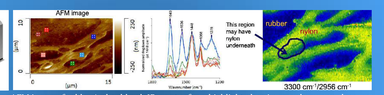
stand-alone glove-box

Cryogenic, inert atmosphere/vacuum transfer shuttle

TEM air protection holder

Scanning probe tools

thermomechanical nanoIR: atomic force microscope measures thermal response of surface to tuned IR radiation. IR spectral characterization and imaging at resolution below diffraction limit.



AFM image of rubber/nylon blend, IR spectra from highlighted regions, and wavenumber maps

stylus profilometer: stylus drawn across surface and vertical displacement is measured as a function of lateral position for profilometry/metrology (e.g. roughness, step heights, waviness)

2D measurement of step height

SIMS sputter crater 3D image

waviness (red), roughness (blue) profiles

Other characterization infrastructure: (i) inverse-photoemission spectroscopy, (ii) temperature-programmed desorption

Electron Microscopy

Morphology, topography, crystallography, elemental/chemical composition are critical in determining material properties. Characterizing these properties and understanding structure-property-behaviour relationships, (especially in in-use environments) is critical. Electron microscopy utilizes focused beams of electrons to probe and characterize materials from the mm to the sub-nm range. By monitoring different interactions of the electron beam with material, different properties can be probed.

Variable-Pressure Scanning Electron Microscope: optimized for analytical characterization of broad selection of materials. Low-vacuum mode for analysis of a variety of samples, including insulators (w/o coating), non-vacuum compatible samples, etc..

Hitachi SU3500 •VP SE detector (SE imaging) •energy dispersive analysis (quantitative elemental analysis and mapping) •electron backscatter diffraction pattern detector (microstructural crystallographic characterization and mapping)

glass fibers in resin

flower pollen

EBSD map of Cu/Sn .

EDX compositional map

Real-time 3D image of insect

Field-emission Scanning Electron Microscope (2 tools): optimized for ultra-high resolution (down to 0.6 nm) imaging applications.

Hitachi SU8230 •7 electron detectors (optimized contrast for different purposes, e.g. comp., topo., surface, cryst.) •2 energy dispersive x-ray detectors (quant. elemental analysis and mapping) •electron backscatter diffraction pattern detector (microstructural crystallographic characterization/mapping) Hitachi S4500 •backscattered and secondary electron detectors • energy dispersive x-ray detector •electron backscatter diffraction pattern detector

mesoporous silica

mesoporous silica

surface voltage contrast; Li ion battery negative electrode

EDX map of Au and Sn nanoparticles Au (red), Sn (green), Al (blue)

EBSD map of Cu

Transmission Electron Microscope: high energy electrons and thin samples allow transmission of electrons through material. ~atomic resolution and providing bulk e.g. morphological, crystallographic, chemical, electronic information

Hitachi HF3300 •300 keV electron source •in-column environmental capability (T, P) allowing reaction process analysis and observation •high pressure SE detector •nano diffraction •energy dispersive x-ray detector (quantitative elemental analysis and mapping) •spatially-resolved electron energy loss filter (chemical state analysis and imaging)

semiconductor transistor observed along Si[001] zone axis

Dynamic in-situ annealing of Pt/C catalyst (400C,4.3e-2 Pa air). Most of the Pt particles actively coalesced, grew larger and migrated.

EELS used to track change in Ti across diffusion interface

Focussed Ion Beam: high current ion beam for sample milling, sectioning, thinning, polishing. Sample preparation tool for TEM/STEM/SEM/surface analysis, as well as integrated 3D characterization of samples.

Hitachi NB5000 •40 keV, >50 nA Cs ion source •high resolution electron column (0.8 nm) •high pressure SE detector •energy dispersive x-ray detector (quantitative elemental analysis and mapping) •TEM/STEM and bulk sample handling

Nano-fabrication

TEM sample thinning

Milling & monitoring

3D reconstruction of SI device from mill & monitor slices

Who is OCCAM?

R.N.S. Sodhi, tel: 416.978.1470 email: rns.sodhi@utoronto.ca

P.M. Brodersen, tel: 416.978.8950 email: peter.brodersen@utoronto.ca

S. Boccia, tel: 416.978.5640 email: s.boccia@utoronto.ca

C. Mims, tel: 416.978.4575 email: charles.mims@utoronto.ca

D. Perovic, tel: 416.978.5635 email: doug.perovic@utoronto.ca