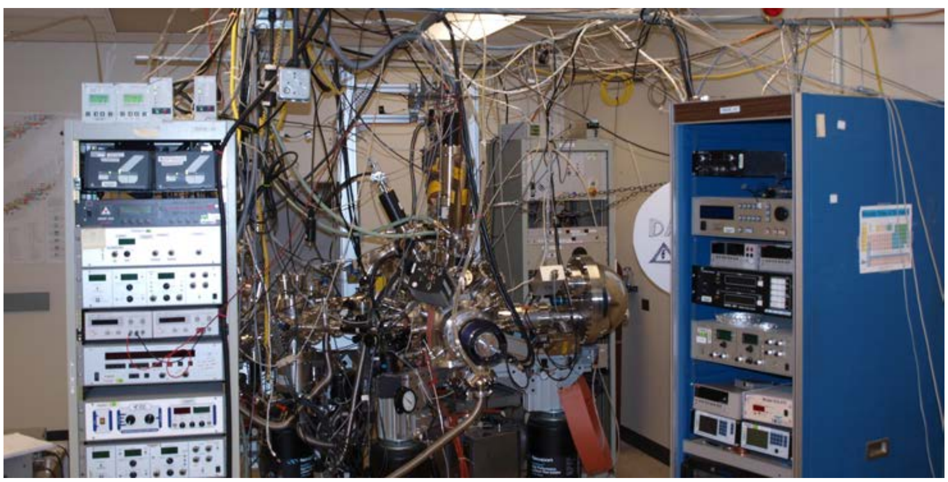
CMUXE IMPACT LAB

Training Manual
Purdue University



The Interaction of Materials with Particles and Components Testing (IMPACT) experimental facility at Purdue University is designed to study *in situ* dynamic heterogeneous surfaces at the nanoscale exposed to varied environments that modify surface and interface properties. The philosophy behind experiments in IMPACT relies heavily on its ability to provide a wide array of characterization techniques and conditions that properly simulate complex environments. The IMPACT experiment achieves this by atomic-scale characterization of the evolution of elemental, chemical, and thermodynamic states of ultra-thin film surface and interfaces using complementary surface-sensitive characterization techniques. In situ techniques used in the IMPACT experiment include the following:

- Low-energy ion scattering spectroscopy (LEISS) with simultaneous forward and backward scattering modes
- Direct recoil spectroscopy (to study impurity levels in the film)
- X-ray photoelectron spectroscopy (XPS)
- Auger electron spectroscopy (AES)
- Secondary electron emission (SEE) spectroscopy
- Extreme ultraviolet/UV photoelectron spectroscopy (EUPS)/UPS
- Extreme ultraviolet reflectometry (EUVR)
- Mass spectrometry using both quadrupole and magnetic sector analyzers.
- Scanning probe microscopy (SPM)

LEISS can give surface elemental information, whereas EUPS is useful to study surface chemical property of a few monolayers. While the low energy ions are bombarded at a grazing angle to study ISS, EUV light (92 eV photons in our setup) is used for EUPS studies. On the other hand, elemental profile and the composition can be detected by AES while XPS can be utilized to study the chemical states. The information can be achieved from a depth of 2-3 monolayers up to about 10–15 nm into the bulk. The system is also capable of measuring SEE spectroscopy by using an electron source with variable energies from 30-5000 eV. In fact, we use the same electron gun for AES study. All these spectroscopies are conducted by using a highly sensitive Specs Phoibos-100 hemispherical electrostatic energy analyzer (HEA). High-resolution depth profiles are obtained by using a unique low-energy ion source delivering 100 eV ions of any desired inert gas species at current densities of 2.5 $\mu A/cm^2$. Simultaneous to surface analysis of the irradiated sample, the total erosion flux is measured *in situ* using an ultrasensitive temperature-compensating quartz crystal nanobalance — dual crystal unit (QCN-DCU) with resolution better than 0.005 Å/sec. During ion etching the sample can be tilted at any desired angle with respect to its surface normal from 0 to 60 degrees, with a resolution of better than 0.1 degrees. Dynamic effects induced by energetic charged particles can range from induced surface morphology evolution to physical sputtering. IMPACT is designed to primarily study the effects of the latter by means of mass loss techniques. Photos of the IMPACT facility at CMUXE are given in Fig. 1. A pre-chamber which is also attached with the load lock (Fig. 2) is used for thin film growth using mini-electron evaporator and nanopatterning by ion beam erosion using a high current, compact plasma ion gun from Tectra.

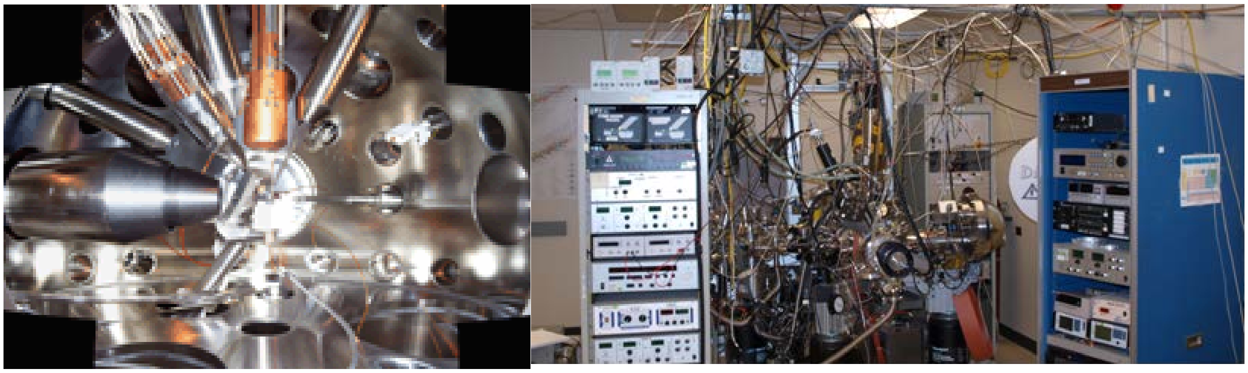


Fig. 1. IMPACT surface science facility at CMUXE. Right side image shows the inside view of the IMPACT chamber.

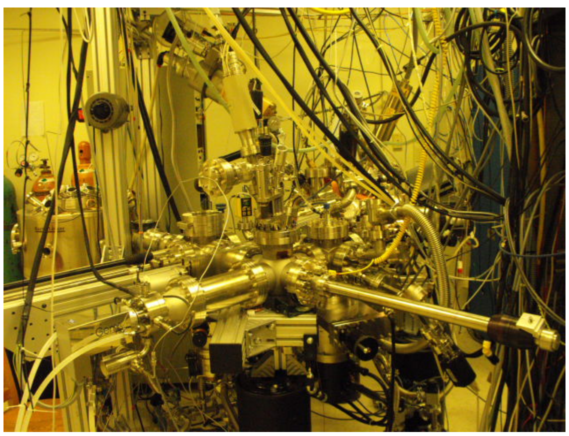


Fig. 2. Pre-chamber, which is connected with the load loack

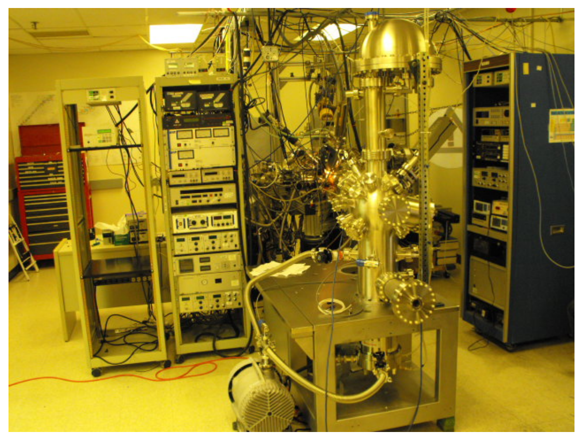


Fig. 3. IMPACT-2, which will be connected with the existing one, called IMPACT-1

We are integrating one more ultrahigh vacuum (UHV) chamber with the existing IMPACT facility for conducting surface analysis in ultraclean atmosphere (Fig. 3). The new chamber will consist of UPS, scanning-AES for further understanding of the surface properties. The emitted electrons from the target surface will be analyzed by a fast scanned HEA from Omicron. This HEA is also suitable for two-photon photoelectron (2PP) spectroscopy. The IMPACT-1 and IMPACT-2 will be connected through a single 8" flange where the sample can be travelled between these 2 chambers with the help of a long manipulator. The new facility allows us to characterize the surface properties by varying the substrate temperature from 25 to 900 ^{o}C.

The surface morphology of a solid sample is investigated *ex situ* by SPM, which can work in different modes such as atomic force microscopy (AFM), conducting mode AFM and scanning tunneling spectroscopy/spectroscopy (STM/STS). Depending on its application, AFM can be used either in contact or tapping mode.

General Lab rules

To access the Impact facility through the ATR National Scientific User Facility, a researcher will need to submit a Rapid Turnaround Experiment proposal through the ATR NSSFU website. If the experiment is awarded, the Impact facility will work with the user to set up a schedule for the research.

All CMUXE IMPACT lab users must read and understand the information in this document with regard to laboratory safety and emergency procedures prior to the first laboratory session. Your personal laboratory safety depends mostly on YOU. Effort has been made to address situations that may pose a hazard in the lab but the information and instructions provided cannot be considered all-inclusive**.**

1. Obtain proper training before starting to work at IMPACT lab. You will need Chemical safety and/or Radiation, depending on your assignment.

2. Read Training and Safety Manual to familiarize yourself with the basics of lab hazards and safe and effective equipment handling.

3. Read the manuals and procedures carefully for safe handling of equipment. If you have any questions you can contact your supervisor or senior lab members.

4. The laboratory door needs to be closed at all times. Designated entrances and exits are indicated on the doors.

5. Wear lab coats and shoes. Also use gloves whenever you handle ultrahigh vacuum (UHV) components.

Selected equipment operation and good practices

Also users can download manuals and operating procedure from CMUXE web page (https://engineering.purdue.edu/CMUXE/labdoc.html). The web page is password protected and the user can obtain the password from IMPACT lab officer.

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1. X-ray gun

The XR-50 is a high intensity twin anode X-ray source optimized for XPS experiments. The anode base is made of silver to avoid any CuLα stray radiation. The electron optical design of the anode, filament and source housing guarantees maximum X-ray intensity and very low cross-talk between the magnesium and aluminum anodes. The X-ray source is equipped with a 2.75" (NW38CF) port for differential pumping. In addition to the anode, the anode housing is very efficiently water-cooled to reduce the thermal stress on the specimen. Even during long- term operation the sample temperature is not increased by more than 5°C.

IMPORTANT PRELIMINARY INFORMATION:

· Be sure to degas the filament before use if the chamber has been vented.
· The chamber needs to be below 5E-6 torr prior to degassing filament, and 5E-7 torr prior to operating the unit for data collection.
· Prior to operating the X-ray source, turn the Neslab CFT-25 chiller on. The X-ray source will not operate without this unit running. Do not leave this unit

running unnecessarily however.

2. Ion guns

CMUXE IMPACT lab possesses several ions guns. The detailed SOP and manual can be found at CMUXE web page. Most of the ion sources utilize electron impact ionization as its source for ions derived from a gaseous input, giving a small spot size, typically 0.4 mm and high beam currents, a typical density range of 15 to 50 mA/cm2. It can provide beam energies in the range of 50 eV to 5 kV, and the incident particle species will consist of: H, He, Ar, Ne, Kr, Xe, N, and O. CMUXE also possess one plasma ion gun which provides very higher currents. However, a broad ion beam with current can be achieved from Tectra Gen-II plasma ion source.

3. Electron guns

The Specs EQ 22/35 electron gun is designed for stable and reliable operation in electron source applications including SEE, AES, scanning applications, imaging, EELS and electron pulse or desorption experiments. In combination with Specs PHOIBOS it can be used for Auger Electron Spectroscopy (AES). Detailed SOP and manual of electron gun can be downloaded from CMUXE web.

4. Phoibos electrostatic energy analyzer

The Specs PHOIBOS hemispherical electrostatic energy analyzer allows recording of energy spectra for electrons and ions emitted from a target material, with an energy range from 0eV to 3.5keV. This allows IMPACT to utilize XPS, AES, and ISS material analysis techniques. The unit has four voltage connections, which are supplied by the HSA3500 analyzer. These voltages have a maximum potential of 40, 400, 1500, and 3500 volts, these vary with the operation of the unit. Read manual carefully before operation.

5. Phoenix EUV gun

The Phoenix EUV gun is a compact, debris-free, stable and user friendly system, which utilizes electron-induced EUV emission from Si target. In particular, electrons are generated by a tungsten filament (cathode) and accelerated in a high-voltage electric field toward a Si target (anode). The characteristic emission in the EUV spectral range is produced due to electron-impact ionization (excitation) of atomic inner shells, which is followed by radiative decay. In fact, the narrow band at about 13.5 nm is associated with the L-edge Cherenkov emission induced by relativistic electrons in Si.

6. Mini e-beam evaporator

The EGN4 is a mini e-beam evaporator designed for use in a UHV environment. It provides the capability to evaporate high melting-point materials in a controlled manner at rates between <1 monolayer/min to over 5 nm/min. This is achieved by the use of electron-beam-induced heating of the target material to the temperature at which the desired evaporation rate is reached.

- The evaporator has four separate pockets, each of which can be loaded with material either in the form of rods, or contained inside mini crucibles. Each pocket is equipped with its own dedicated filament.
- The target rod (or crucible) is held at a high potential of 2 kV. Electrons emitted from the dedicated filament, at earth potential, are accelerated onto the tip of the rod by the high field gradient between the tip and the filament. This emitted

electron beam (emission current) can run at up to 100 mA, giving the total available power as 200W.

- During electron beam induced thermal evaporation of materials, some ionisation of the target vapour occurs. This positive ion current can be used as an indicator of the evaporation rate.
- The turret, or hearth, of the instrument is *directly* water cooled, keeping the temperature of adjacent pockets at near ambient, even during tungsten evaporation.

7. Photodiodes

There are two photodiodes in the UHV chamber and each of them is designed for the EUV wavelength, especially for 13.5 nm.

8. CCD cameras

These are very sensitive light detection instrument. Care must be taken when handling them.

9. Vacuum systems

The IMPACT chamber consists of two UHV chambers such as pre-chamber and main chamber. They are connected by load-lock which is mainly used for changing sample without breaking the UHV condition of the pre and main chambers. The pre-chamber is equipped with mini e-beam evaporator for growing thin films and an ion gun for cleaning or etching the sample surface. Following the sample processing, one can transfer the sample from the pre-chamber to the main chamber without breaking the vacuum for subsequent analysis with the help of several techniques such as XPS, AES, ISS, EUV reflectometry and EUV Photoelectron Spectroscopy. All of them are pumped using turbomolecular pumps, while the main chamber has further been pumped down by an ion pump. There is no extra protection for any of these chambers (for a rapid rise of pressure). So extreme care should be taken during operation of these chambers.

10. Scanning probe microscopy

The Innova atomic force microscope provides more performance and flexibility at a greater value than any other SPM. The proprietary closed-loop scan delivers noise-levels that approach those of high-end, open-loop systems and offers a wide range of functionality for physical, materials, and life sciences, from sub-micron levels up to 90 microns.
Only trained users are allowed to operate AFM. Please contact your supervisor if you are interested to use this equipment.

User arrangement:

In order to write any proposal or do some experiments, it is recommended to contact Prof. Ahmed Hassanein or Prof. S. S. Harilal.