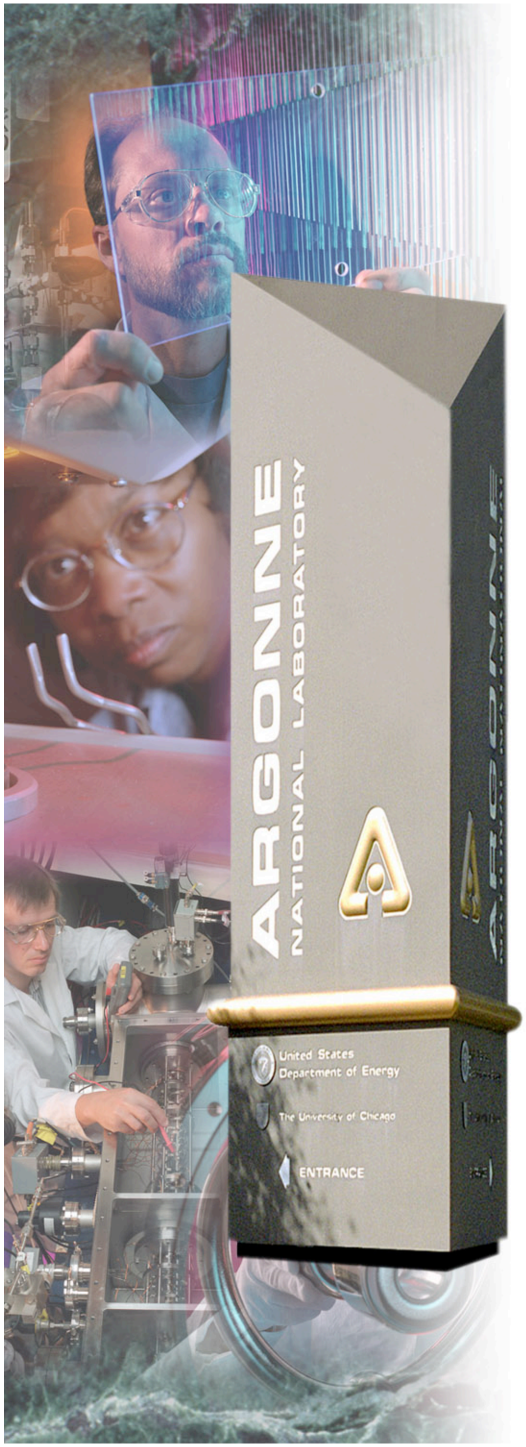

Lithium thin-film coatings erosion and surface analysis

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Outline of talk

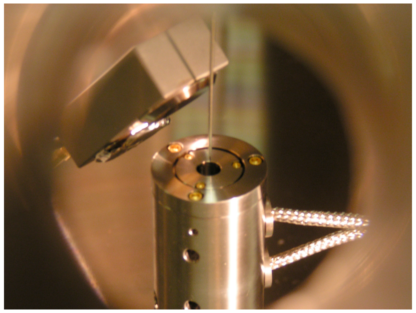
- **Motivation for work with lithium thin-film coatings on graphite**
- **Work Plan**
- **Experiments in: *IMPACT* (Interaction of Materials with charged Particles and Components Testing) at Argonne**
- **Experiments in: *ARIES* at Sandia National Laboratory in Livermore**
- **Conclusions from *IMPACT* and *ARIES* work**
- **Future work**

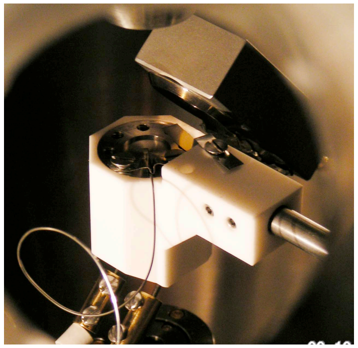
Motivation for proposed work

- **Lithium thin-film coatings on graphite tiles are proposed as "Module A" static liquid lithium divertor for NSTX to enable new confinement regime operation.**
  - Alternatively, W or Mo substrates are also proposed.
- **Experiments at Argonne and Sandia/Livermore study thin-film lithium-based systems with respect to:**
  - Bombardment-induced sputtering (He^+, D^+, etc…) on graphite, Mo or W substrates
  - Lithium coating evolution on graphite substrates as a function of Li coating thickness and substrate temperature
  - Physical sputtering of lithium/graphite system as a function of: incident flux, incident angle, surface temperature
- **Other issues:**
  - Role of boron in Li/graphite kinetics
  - Deuterium retention/kinetics in Li-C system

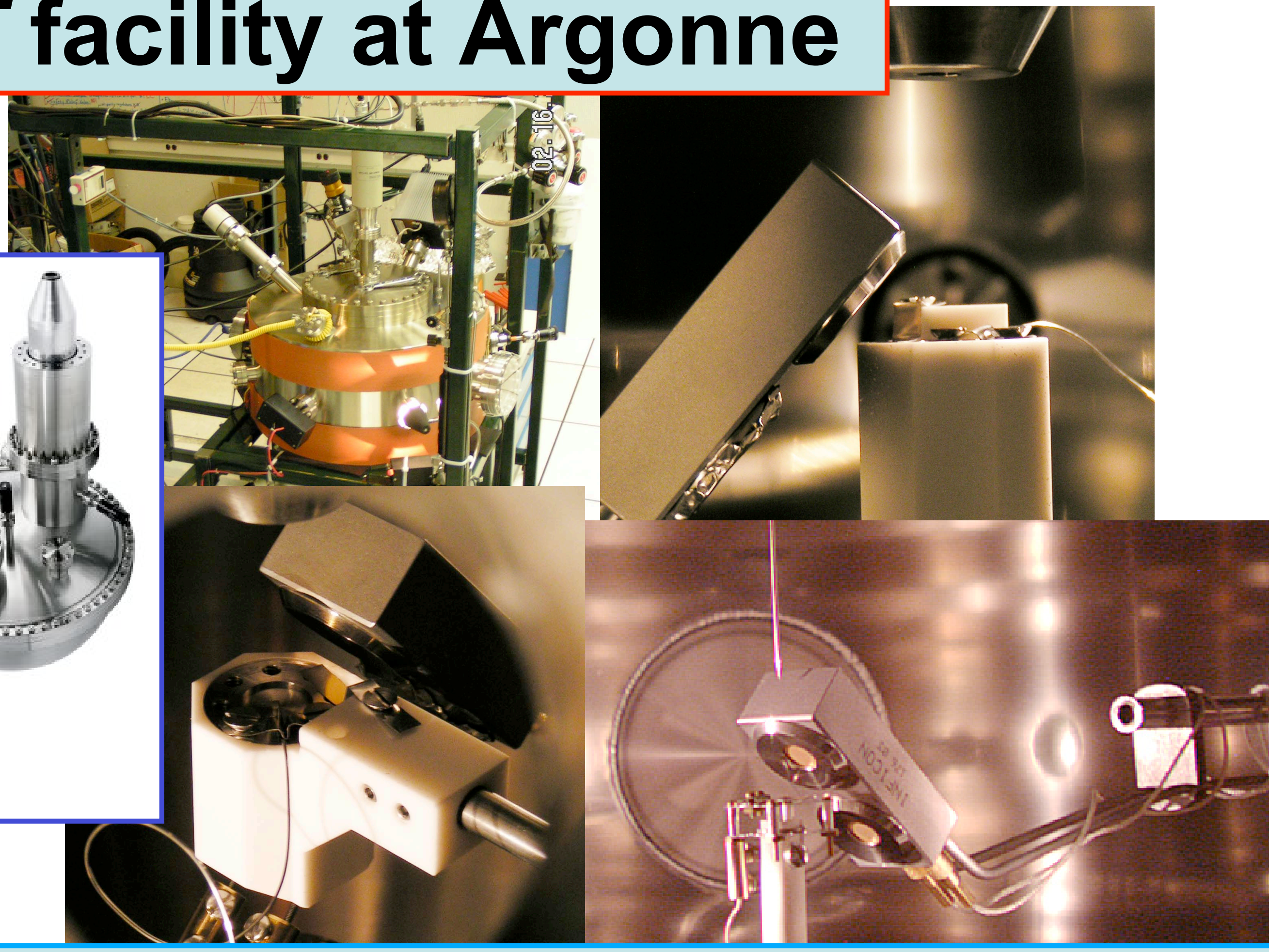
IMPACT Experiment Setup

- Singly-charged particle source providing flux range from 10^{11}-10^{17} ions/cm^2/sec
- H,D,He,Ne,Ar,Kr, and Xe ion beams
- In-situ UHV heater (25-1200 ºC) on rotary manipulator for angle-of-incidence studies
- Base pressures: 10^{-6} Pa
- Studies with background gases: H_2O, O_2
- Quartz crystal microbalance technique for total erosion measurements.
- RGA-QMS Inficon system up to 100 amu
- In-situ Faraday cup for beam profile analysis to conduct flux-dependent erosion measurements
- New transfer-lock chamber will allow for sample transfer in vacuo

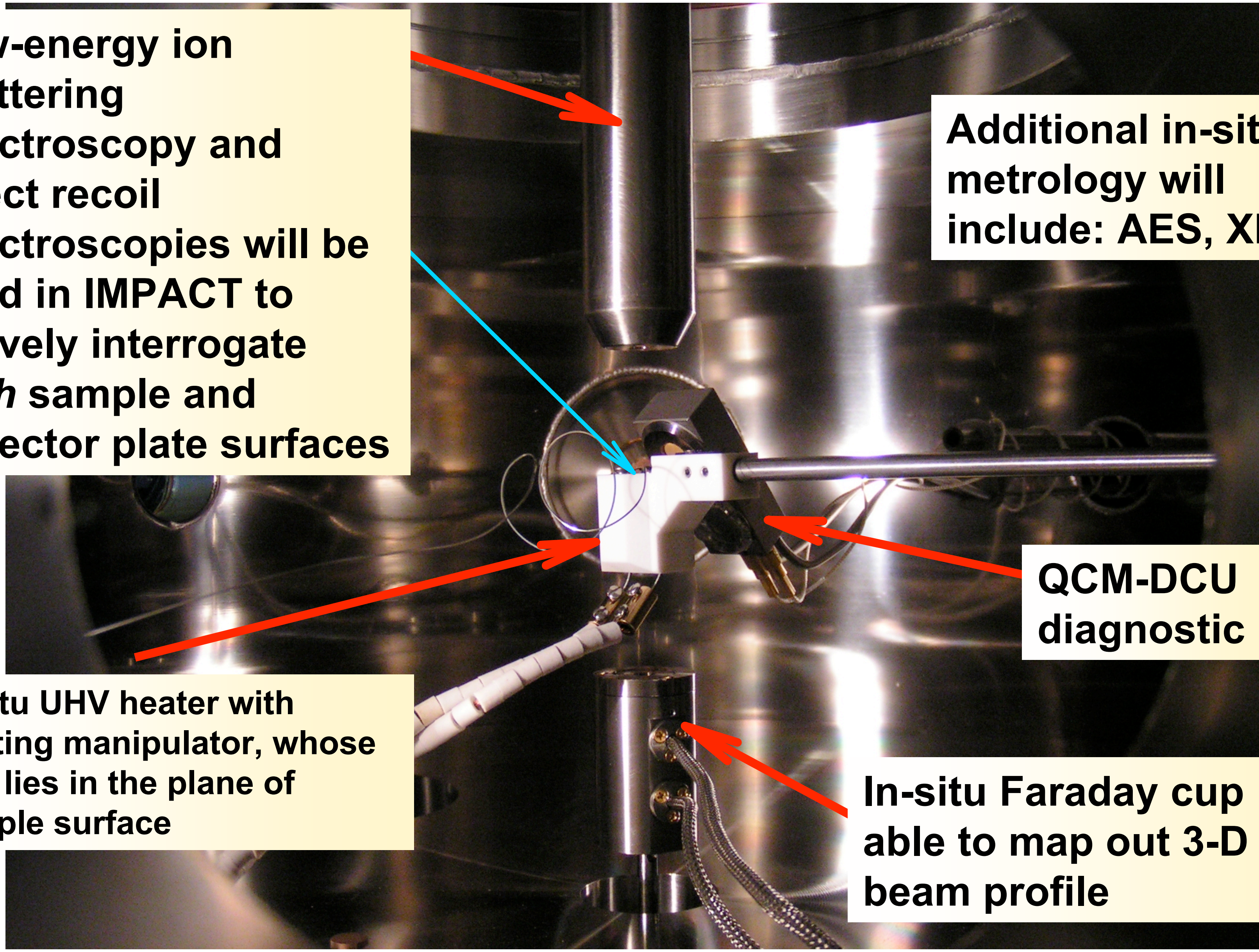




IMPACT facility at Argonne



- QCM-DCU diagnostic already in use measuring physical sputtering (e.g. Xe, Kr and Ne sputtering of Sn, Mo, W, Ru, and liquid lithium)
- Future IMPACT ion guns: Li^{+}, Sn^{+} and LEIG (0.13 mA/cm^{2} at 20 eV)
- Full hemispherical energy analyzer on line 6/04 for: in-situ ISS, AES and XPS



Low-energy ion scattering spectroscopy and direct recoil spectroscopies will be used in IMPACT to actively interrogate *both* sample and collector plate surfaces

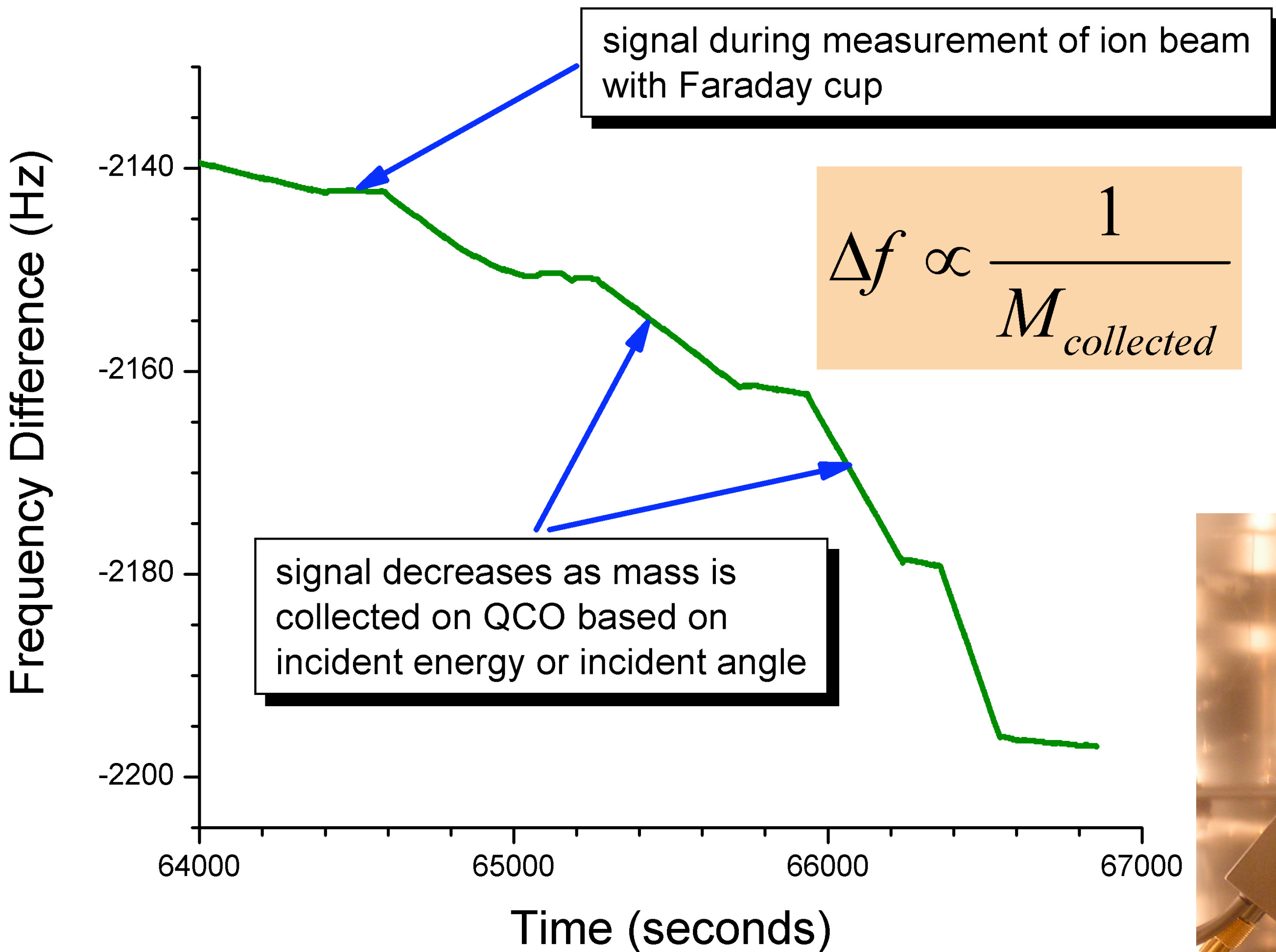
Additional in-situ metrology will include: AES, XPS

QCM-DCU diagnostic

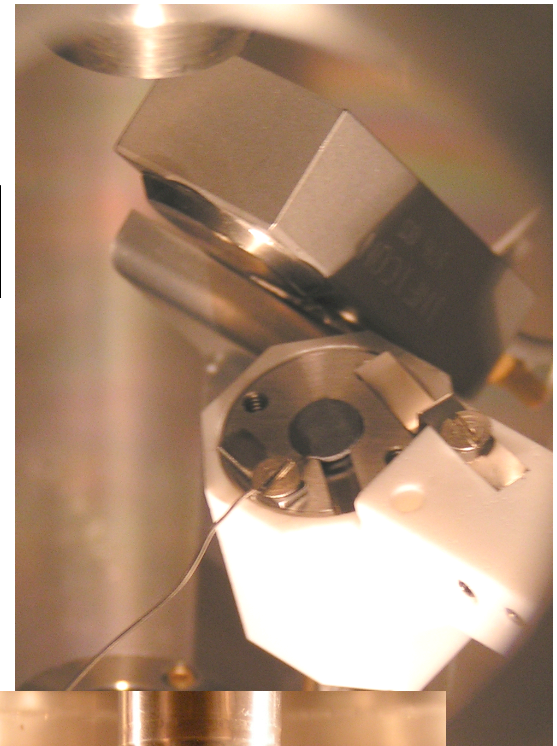
In-situ UHV heater with rotating manipulator, whose axis lies in the plane of sample surface

In-situ Faraday cup able to map out 3-D beam profile

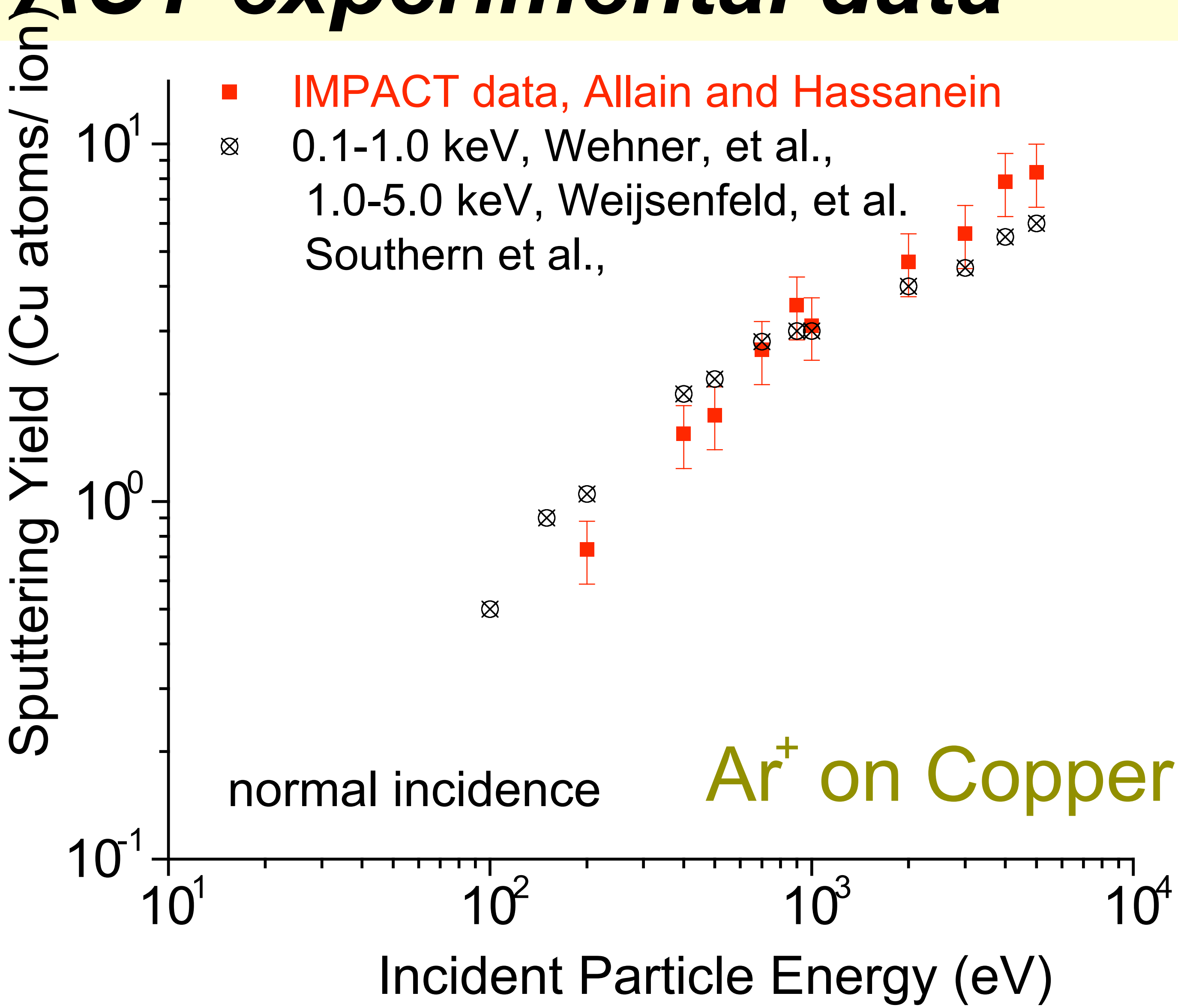
QCM-DCU total erosion measurements



$$\Delta f \propto \frac{1}{M_{collected}}$$



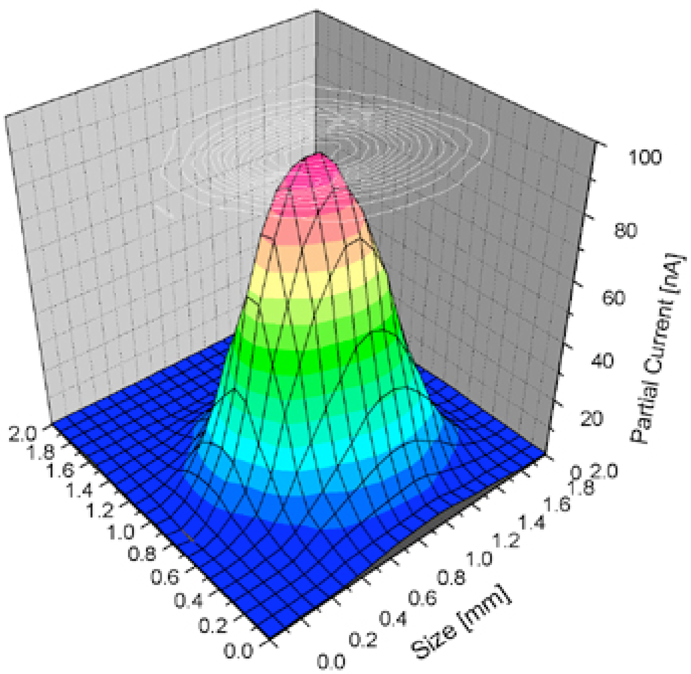
IMPACT experimental data



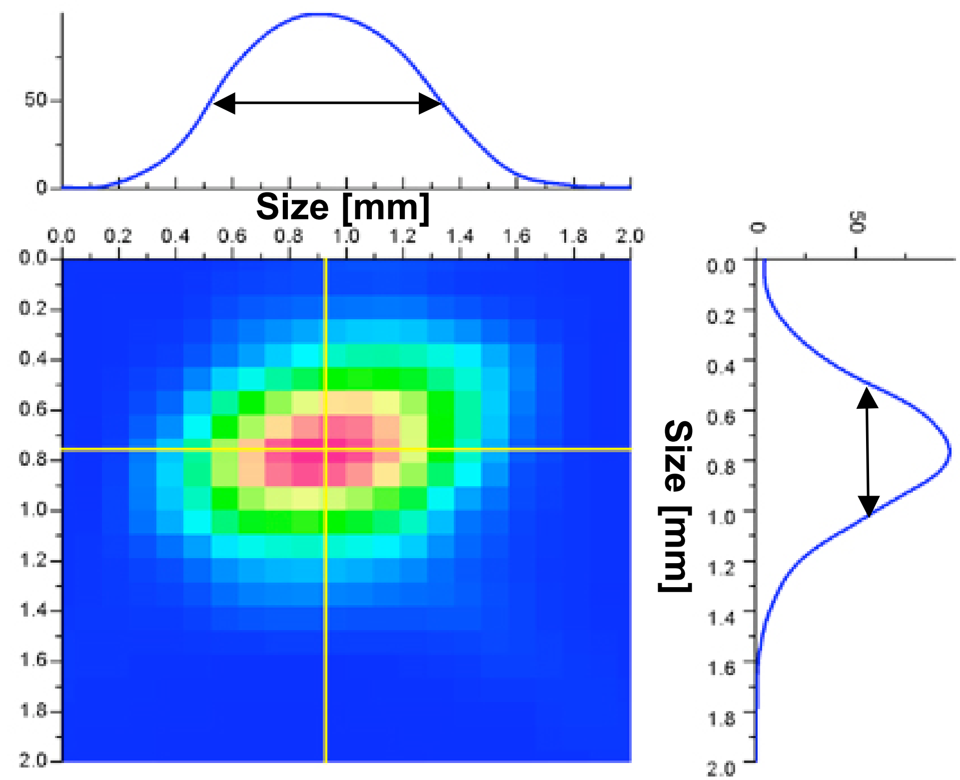
High Flux Helium Beam

Energy: 1 keV
Total beam current: 2.0 µA

Flux = 3.25 _ 10^{15} ions/cm^2/s



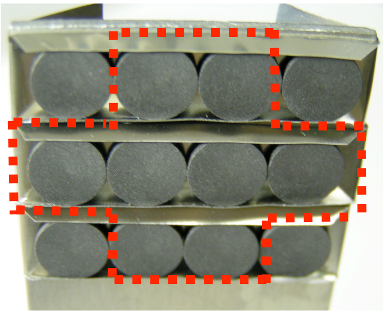
FWHM = 800 µm

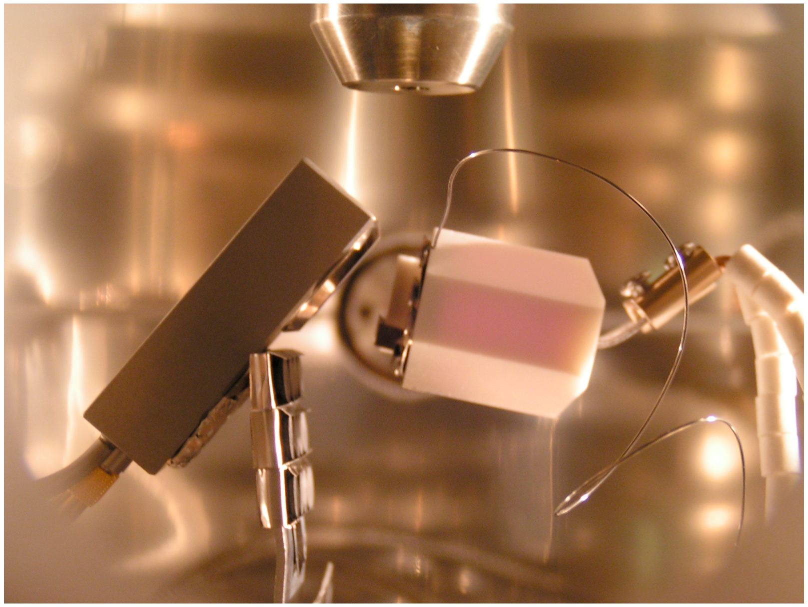


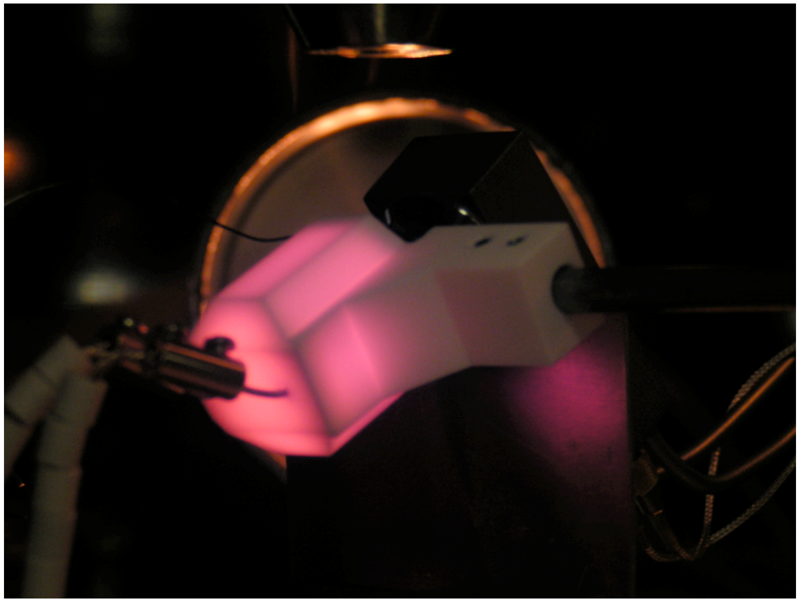
FWHM = 600 µm

Preparation of Li thin-films on ATJ graphite samples

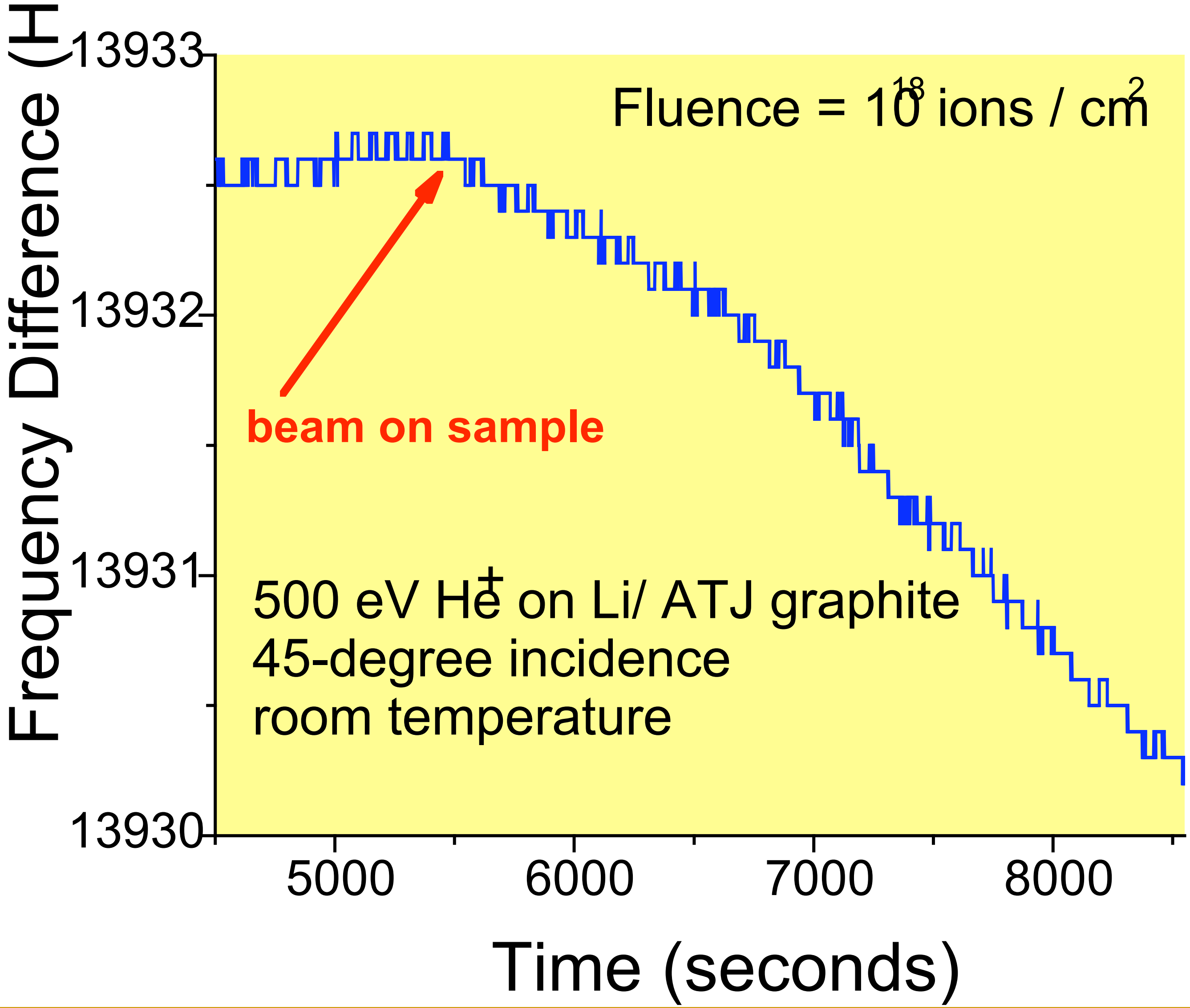
- About 200-400 nm lithium thin-films evaporated onto ATJ graphite samples provided by SNLL
- 8 samples delivered under argon atmosphere to SNLL for surface analysis



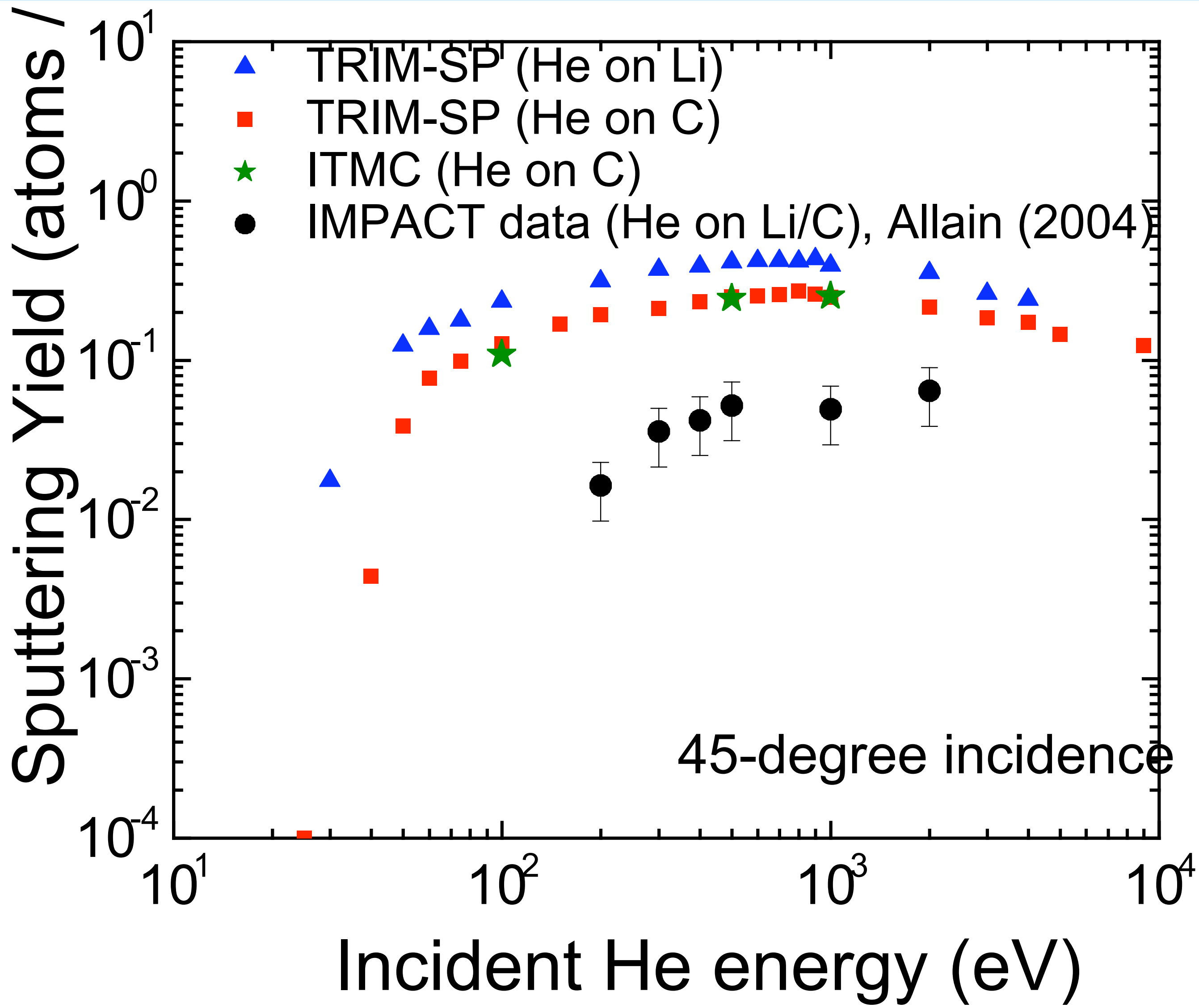




QCM-DCU measurements



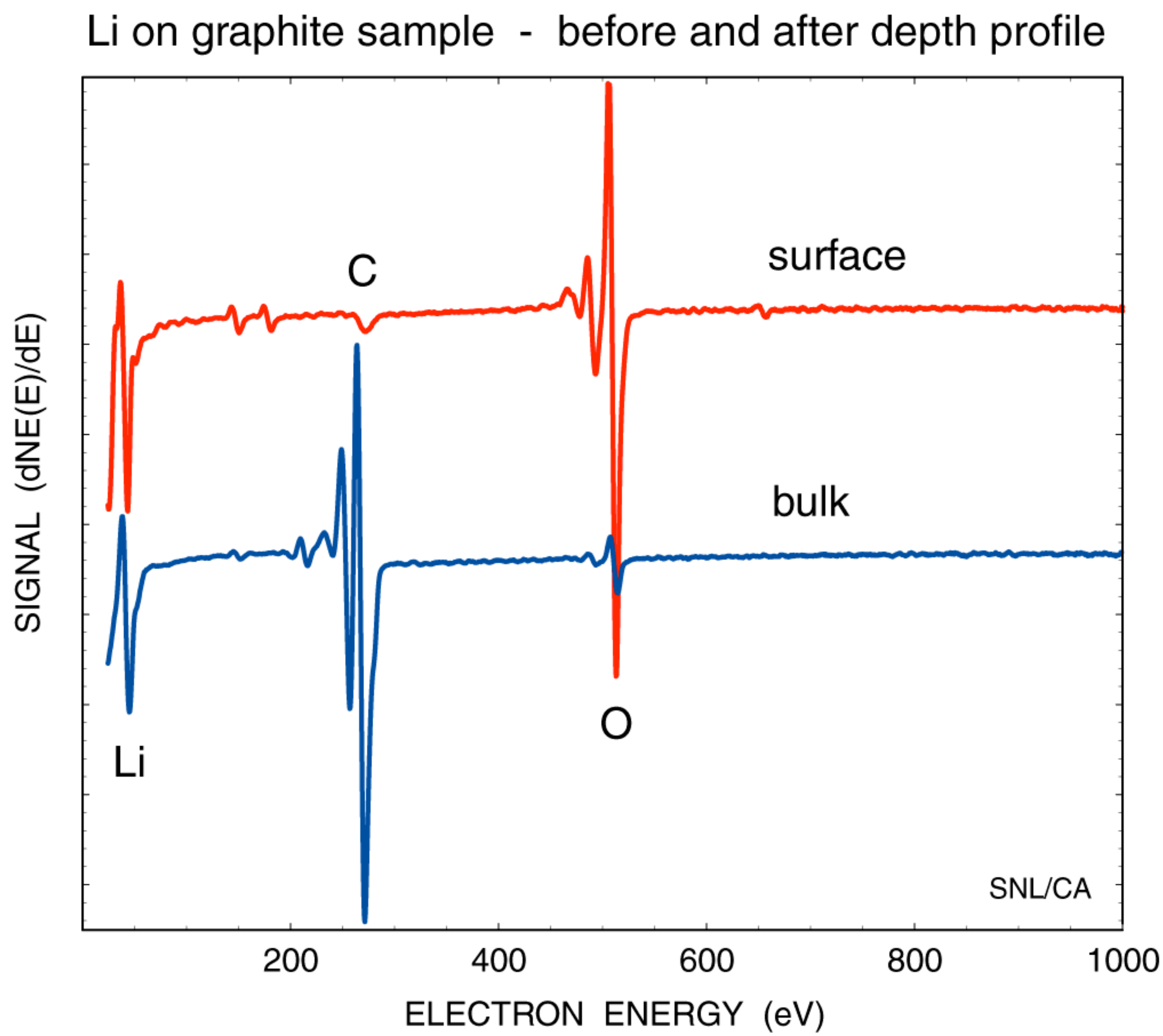
He^+ on ATJ graphite in IMPACT



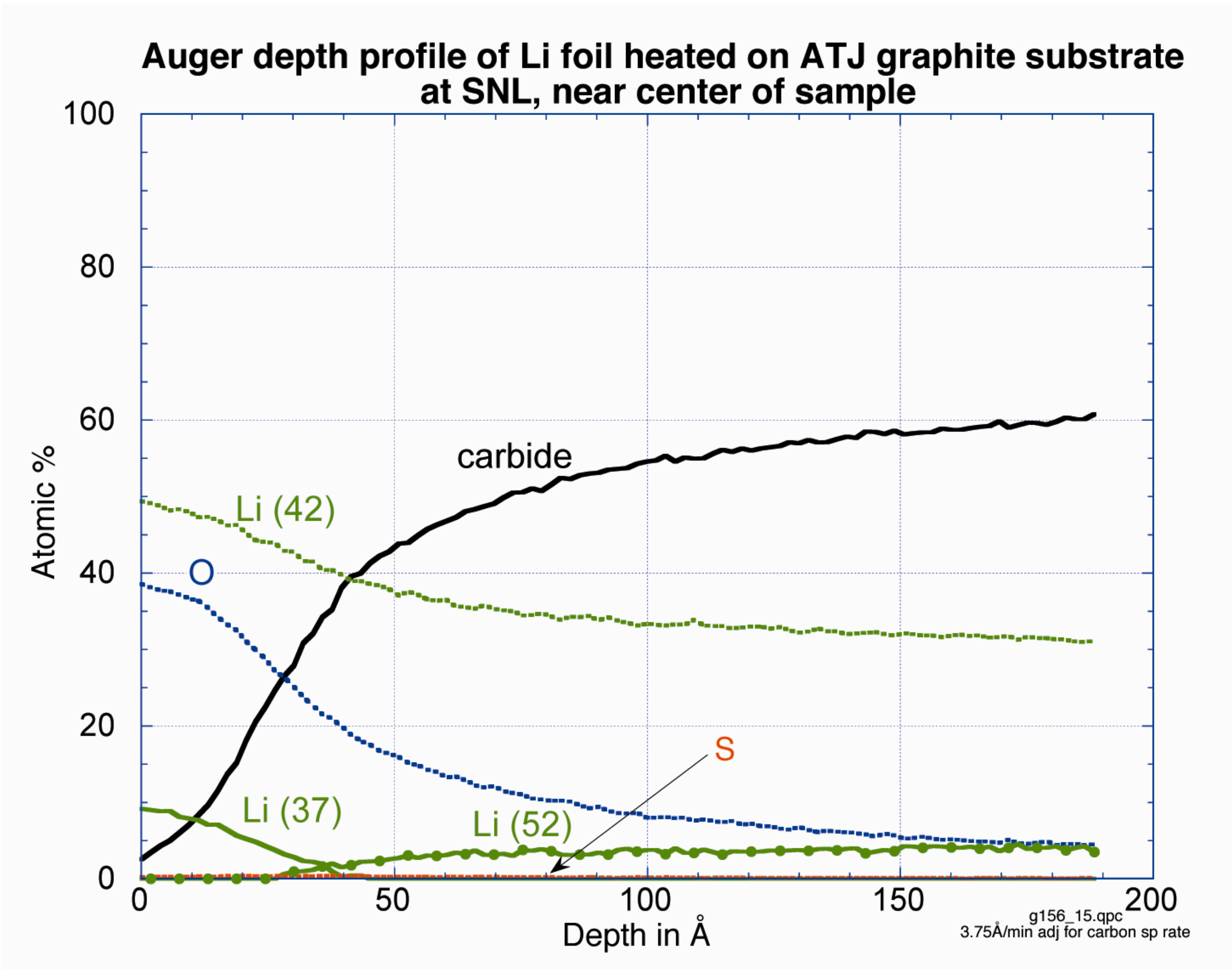
Angle-resolved ion energy spectrometer (ARIES) at SNL/CA

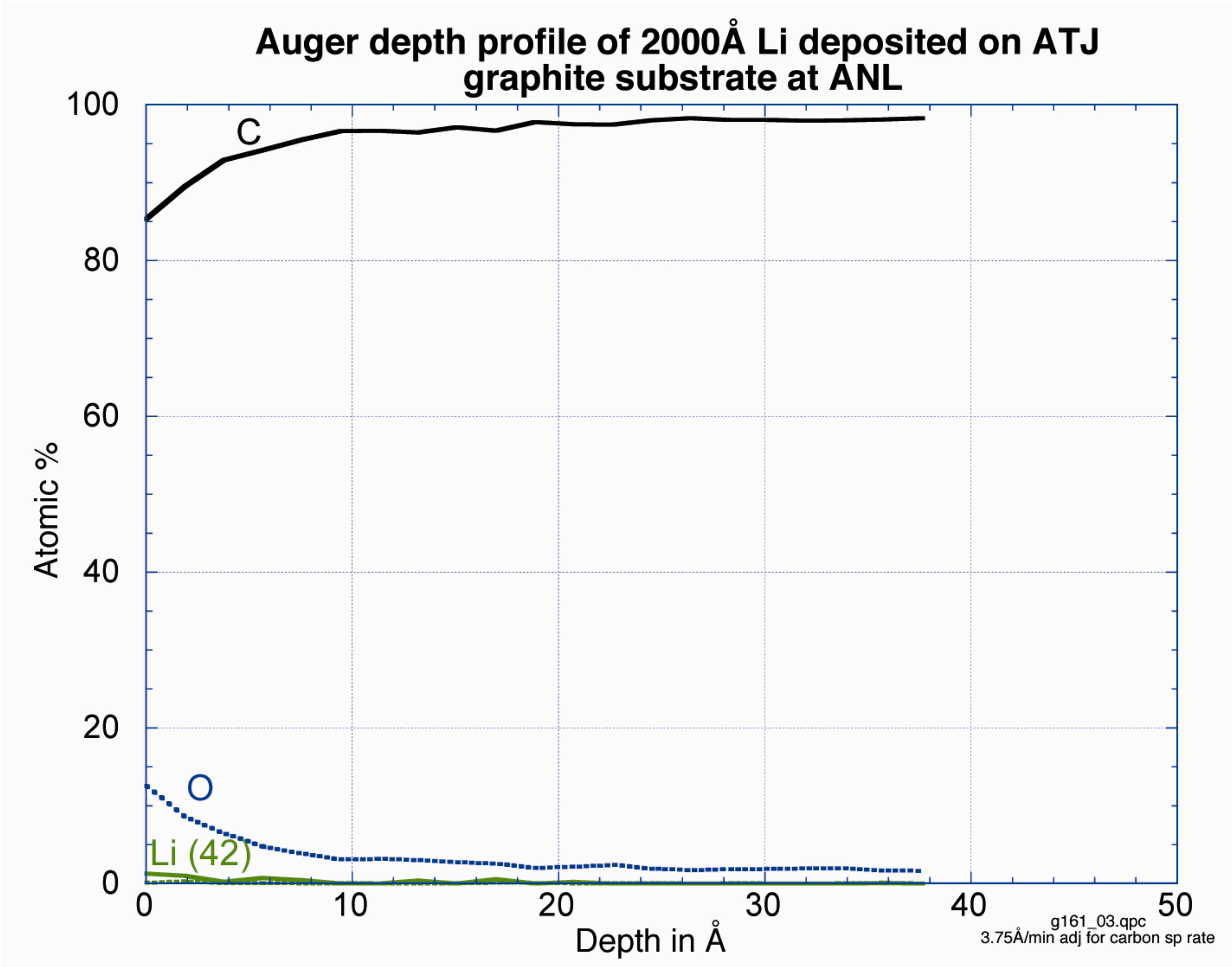
- **Experiment designed to provide detailed top atomic layer analysis of thin films and materials (low-energy ion scattering spectroscopy and Auger electron spectroscopy)**
- **The system has two sections:**
  - A low-energy (0.1 – 3 keV) ion source differentially pumped chamber
  - UHV analysis chamber (P ~ 10^{-10} Torr)
- **ATJ graphite samples with a ~ 200-300 nm lithium thin-film was transferred under argon atmosphere from Argonne**
- **In addition, a 50 µm lithium foil was heated on ATJ graphite sample up to ~ 500 ºC (no liquid Li layer observed).**
- **ISS, AES and SEM analysis of samples.**

Energy spectrum from AES



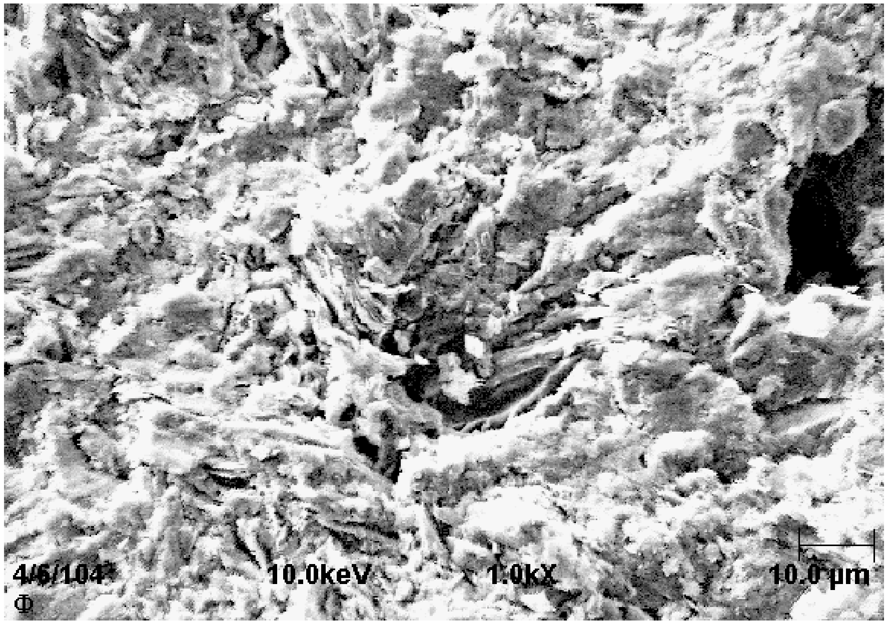
50 um sample

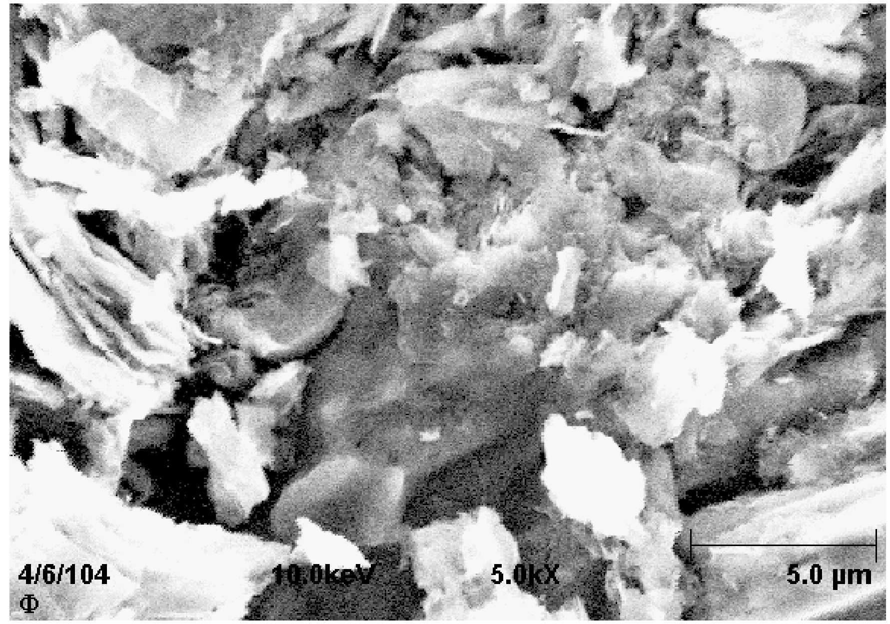

Auger depth profile of Li foil heated on ATJ graphite substrate at SNL, near center of sample
100
80
60
40
20
0
Atomic %
carbide
Li (42)
O
Li (37)
Li (52)
S
0
50
100
150
200
Depth in Å
g156_15.qpc
3.75Å/min adj for carbon sp rate



Auger depth profile of 2000Å Li deposited on ATJ graphite substrate at ANL
100
80
60
40
20
0
Atomic %
C
O
Li (42)
0
10
20
30
40
50
Depth in Å
g161_03.qpc
3.75Å/min adj for carbon sp rate

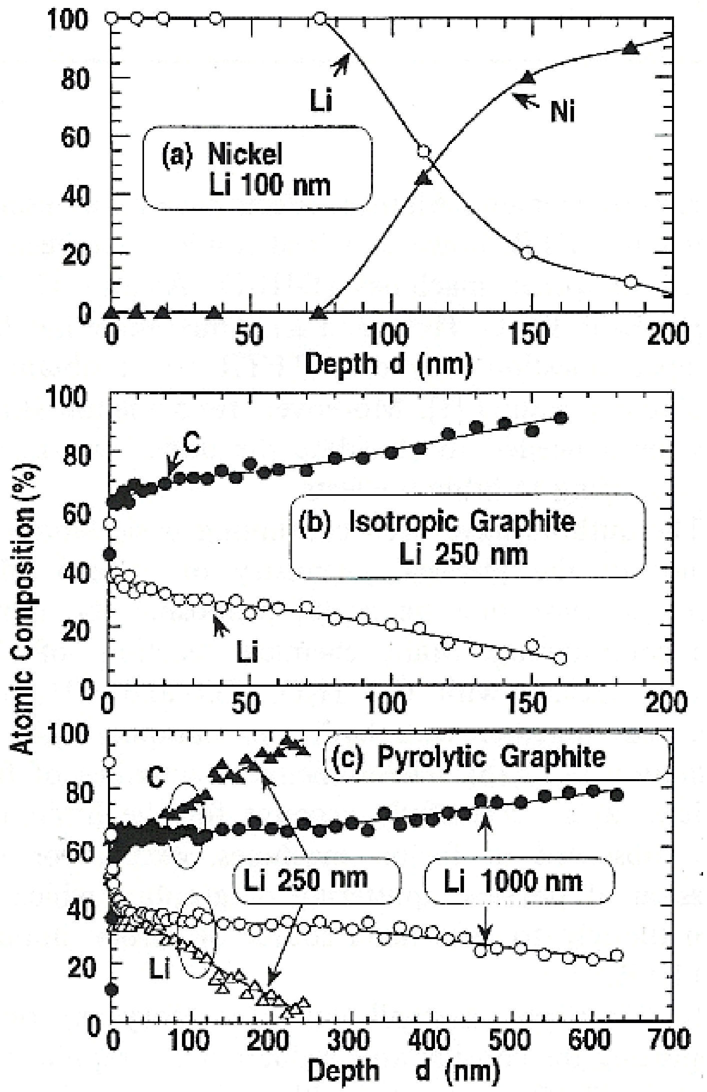
SEM data on samples





- **Scanning electron microscopy (SEM) images illustrate a very rough surface morphology with flakes resembling "lithium oxide" surface structure.**
- **Images above are for Li thin-film coated ATJ graphite samples from ANL.**
- **Surface topography may have affected sputter yield measurements shown earlier.**

AES data of Li in graphite (H. Sugai)



- Deposition of 100 nm Li on nickel substrates shows no diffusion to bulk
- Under similar vacuum conditions (i.e. oxygen, water) lithium depositions on both isotropic and HOPG graphite show rapid and extensive diffusion towards the bulk
- RBS data show diffusion of lithium down to 20-30 µm in some cases
- Inability to obtain a 100% lithium thin-film on graphite substrates

Conclusions from IMPACT and ARIES results

- **Total physical sputtering (C and Li) reduction by close to an order of magnitude. The reduction is consistent, but the magnitude is not based on previous Li/graphite studies (Sugai, et al.)**
- **AES data on 200 nm Li coating and 50 μm Li foil on graphite show strong transport of lithium to the graphite bulk. Consistent with previous Li/graphite studies (Sugai, et al.)**
- **SEM images of a rough surface morphology could explain sputter yield results, however further studies needed**
- **Polished and annealed ATJ graphite samples will be utilized for future Li evaporation at Argonne**

Implications to Module A design in NSTX

- **Lithium deposited on bare graphite may not exist as a distinct liquid layer**
- **Deuterium uptake will be dictated by minimizing impurities on graphite surface and maximizing lithium thin-film coverage over the ~ 2-sec NSTX discharge time**
- **Previous lab studies and successful TFTR “Li supershot” results required ohmic discharge cleaning to remove implanted deuterium**
- **Boron treatment on graphite tiles in NSTX may form an “active” diffusion barrier for lithium thin-film deposition. Experiments underway at ANL and SNLL**

Future Work and Plans

- Continue He and D bombardment of “polished” and annealed ATJ graphite, W and Mo substrates in *IMPACT* as a function of: incident energy, angle, flux and surface temperature.
- In ARIES the effect of boron on lithium intercalation into graphite will be studied
- In-situ metrology (ISS, AES) will be used to study surface kinetics of lithium/graphite system in *IMPACT*.
- Assess role of lithium intercalation in graphite on secondary sputtered ion fraction of lithium atoms (~ 2/3 of sputtered lithium in the ionized state).

Future Work and Plans (cont.)

- In July, Allain will spend two weeks at SNLL and work Li/graphite surface kinetics studies with Bastasz.
- Graphite samples currently being prepared with boron thin-films for use at SNLL and ANL experimental facilities.
- ARIES measurements will also conduct work with lithium-coated W and Mo samples.
- Continued modeling with new DYN-ITMC code at Argonne.

Acknowledgements

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