Study of Lithium on Plasma Facing Components in IIAX (Ion Inter Action Surface eXperiment)

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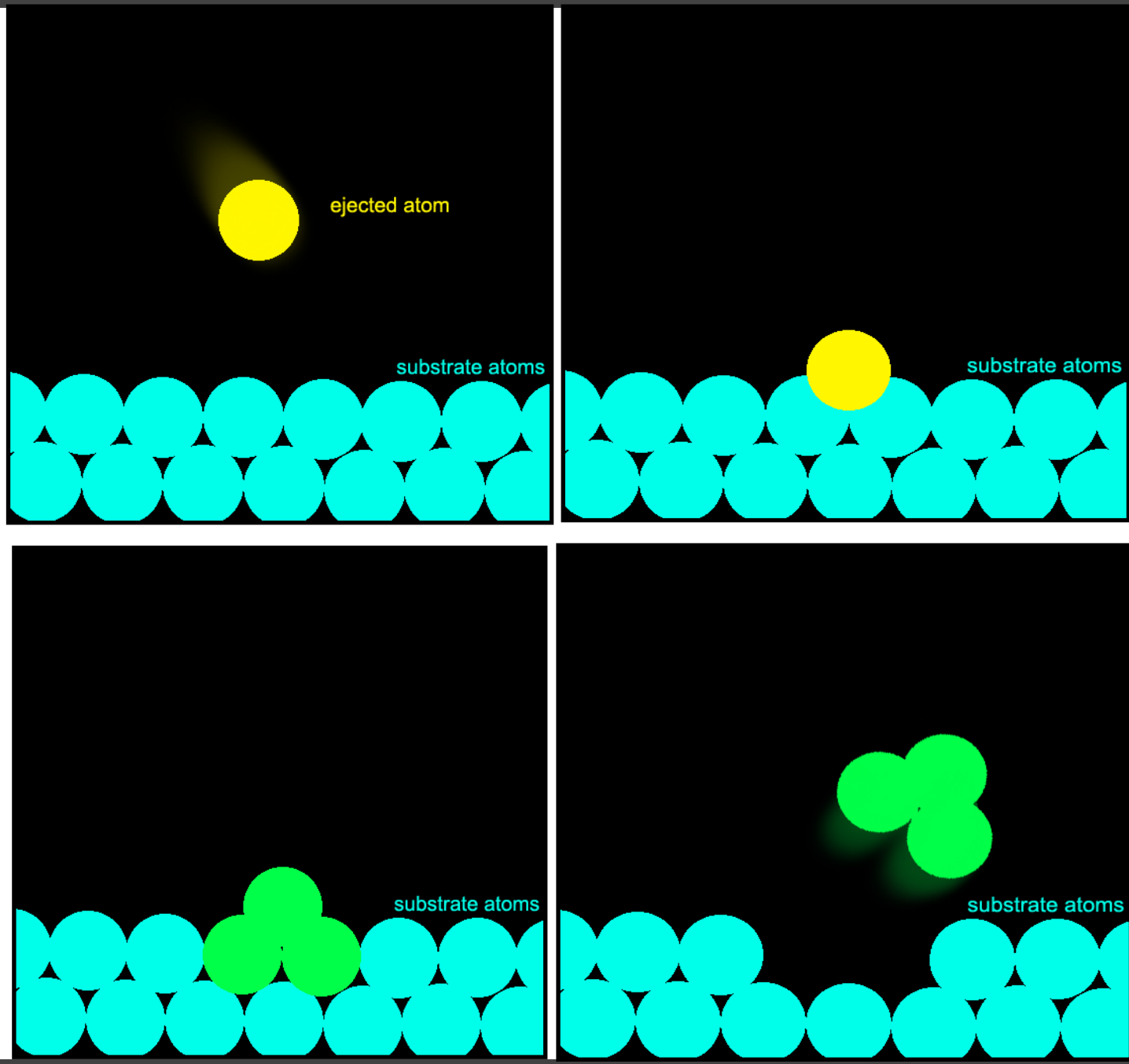
Outline

- Chemical Erosion Studies of Lithiated ATJ Graphite
- Wetting of Lithium on Boronized Plasma Facing Component Material
- Electrostatic Lithium Injector

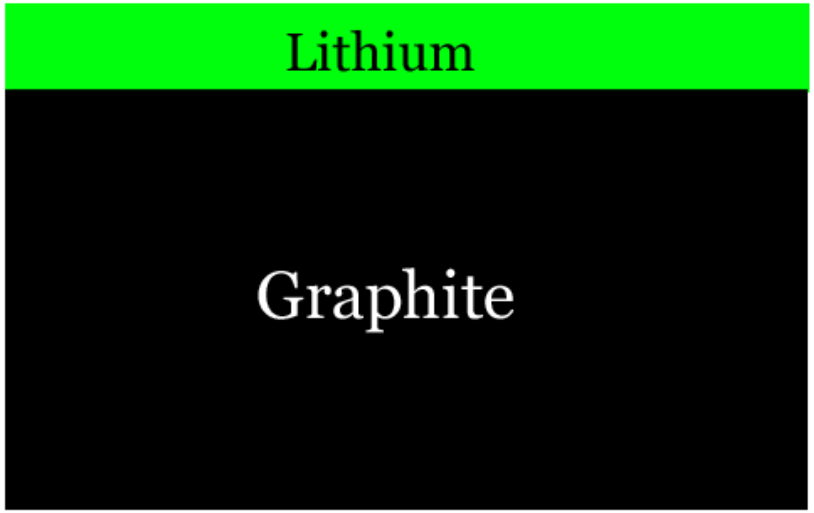
Objective : Chemical Sputtering Measurements

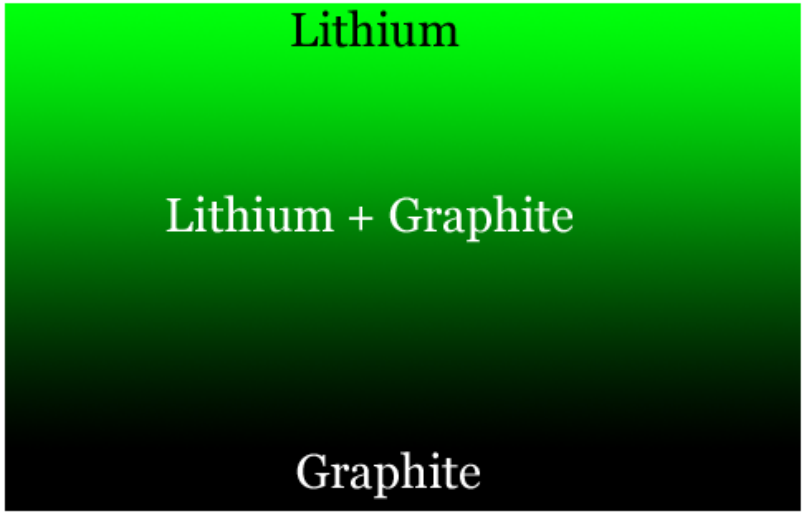
- Chemical sputtering – the combination of solid target particles with foreign particles resulting in a gaseous discharge.

- As with physical sputtering, it is believed that chemical sputtering on graphite is suppressed by Lithium deposition.



Lithiated Graphite Phenomenon

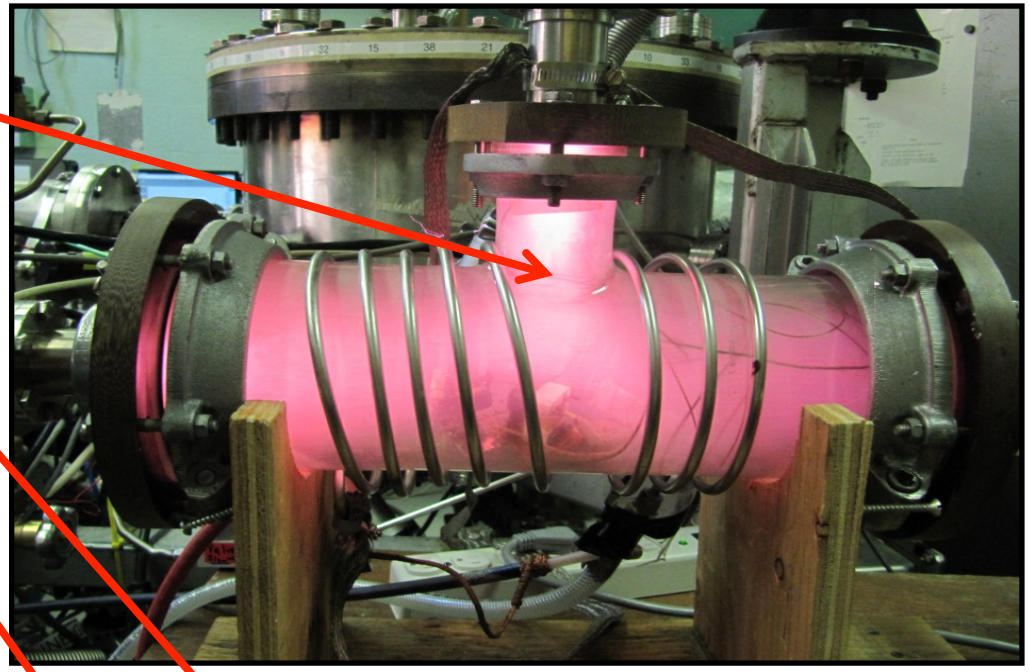


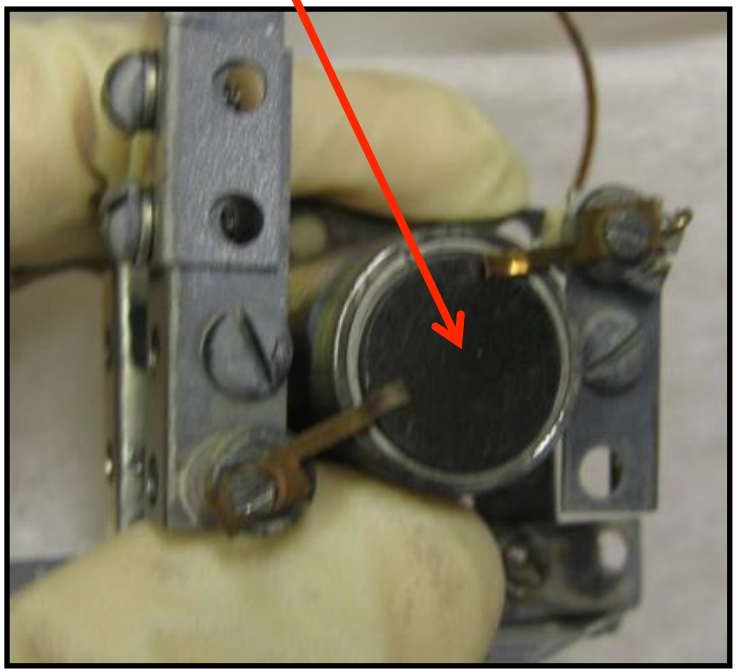


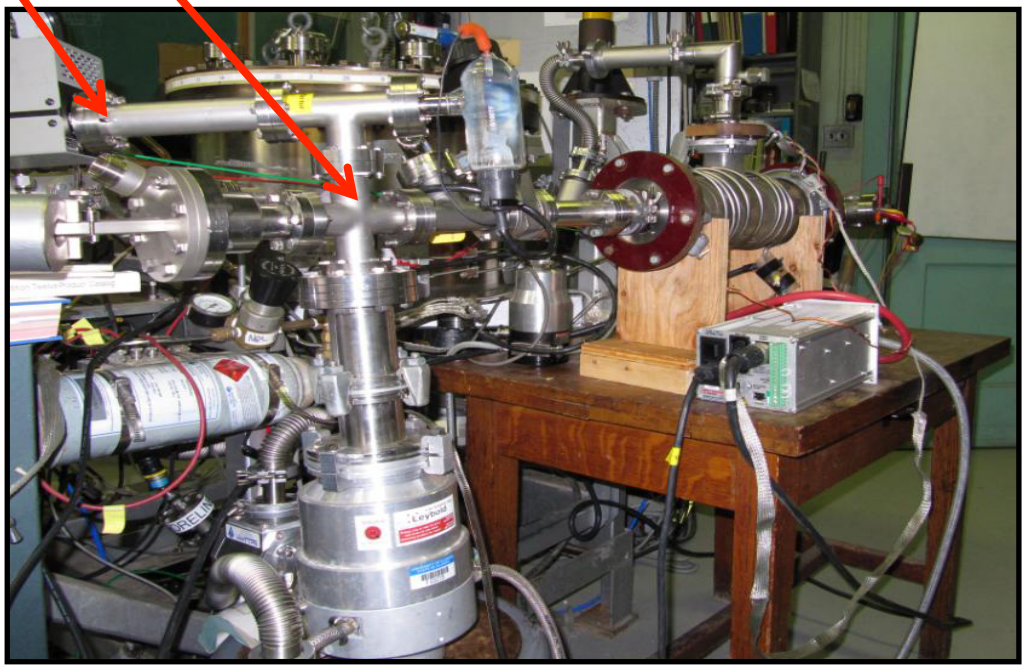
- Lithium is evaporated onto the surface of the graphite
- Instead of forming a layer on the surface, lithium atoms diffuse into the bulk region of the graphite forming a protective layer.
- There is some evidence that the lithium surface layer is replenished continuously.

Experimental Setup

- Surface interaction chamber at
  - P = 2×10^{-6} Torr
- Differential pumping chamber at
  - P = 1×10^{-7} Torr
- *Residual Gas analyzer (RGA)*
- *In-situ* Li evaporator
- Ultra high vacuum button heater
- 1” diameter ATJ graphite







List of Experiments Conducted

Experiments	Base Line	No Plasma	Plasma
WALL CASE (NO TARGET)	A	B	C
ATJ+WALL CASE	D	E	F
LITHIATED ATJ +WALL CASE	G	H	I

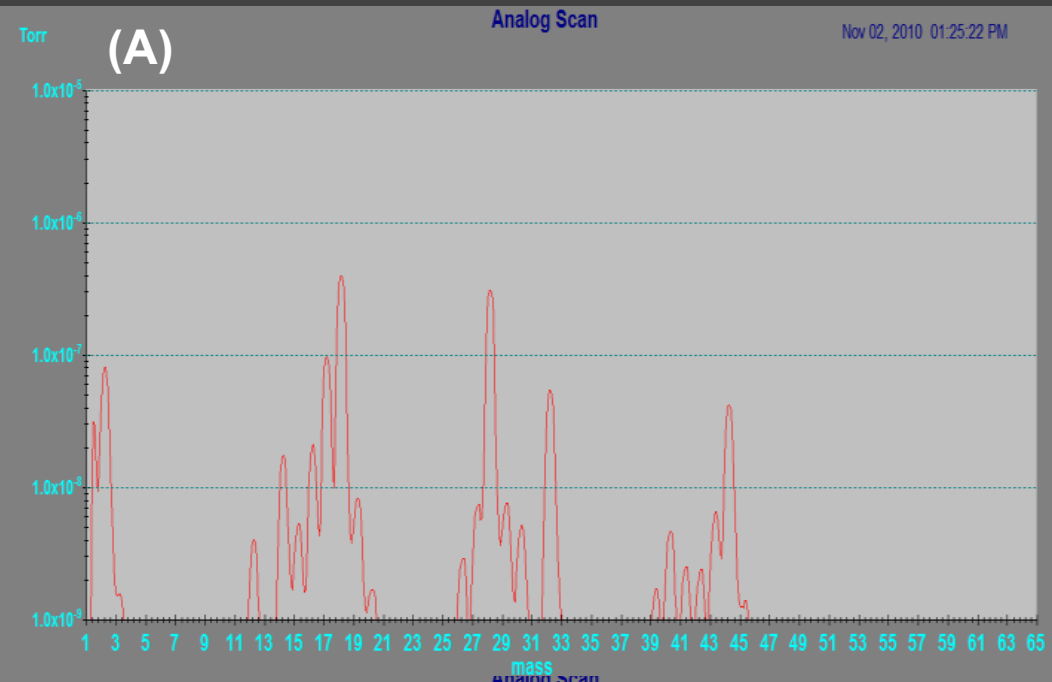
Plasma and gas used is D_2

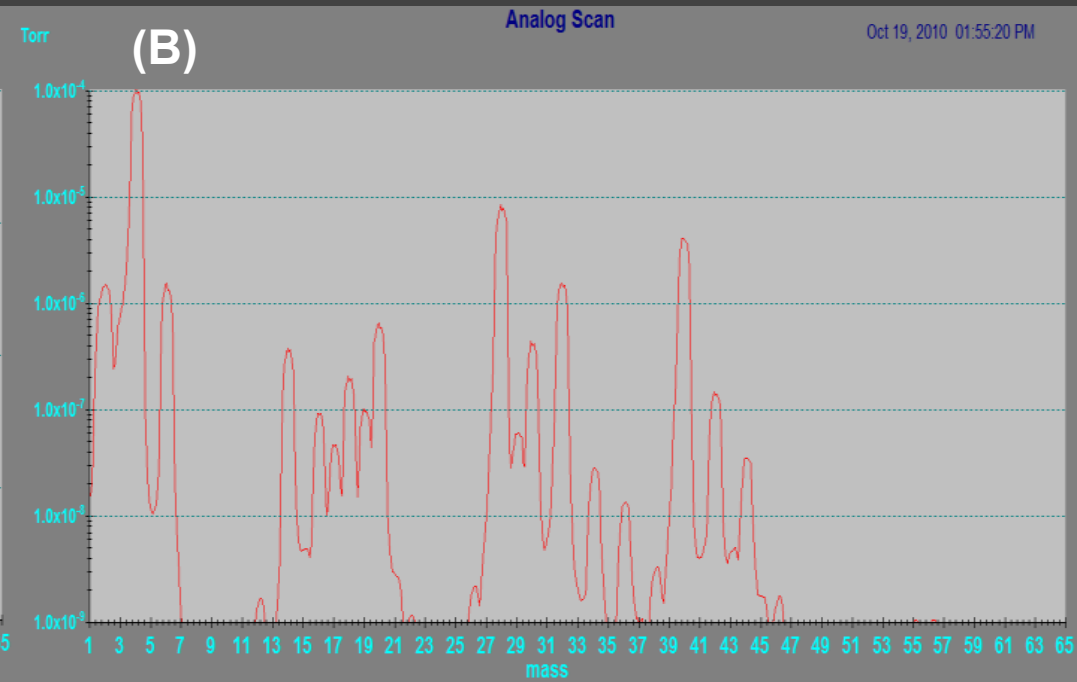
Target Species

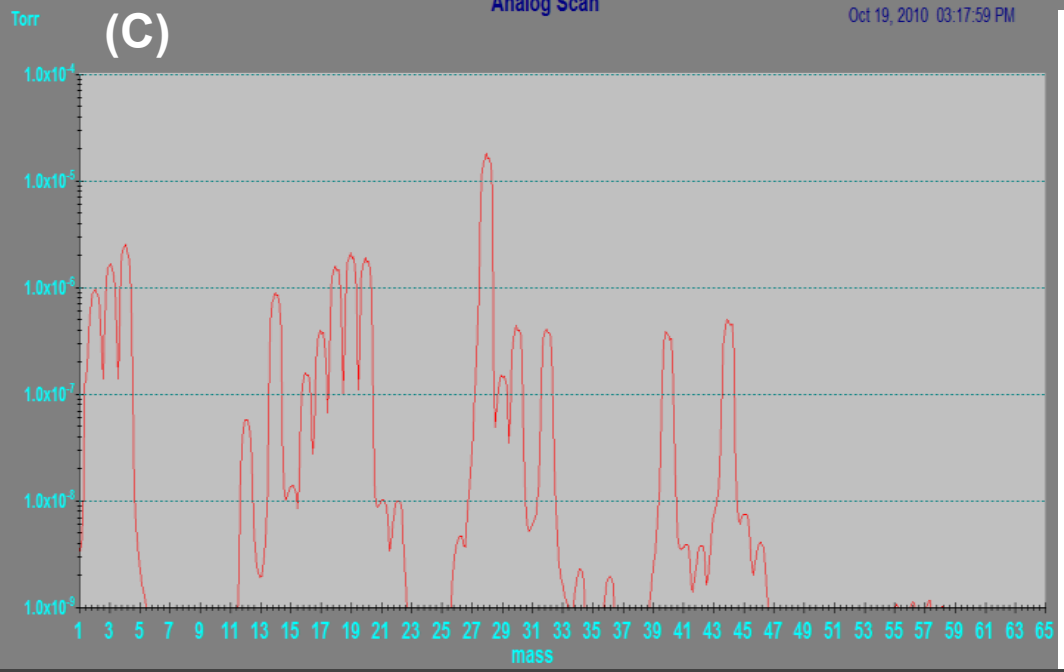
- For simplicity only single carbon chains are considered
  - These are more likely than higher order carbon chains
  - ***CD_4*** and ***CD_3H*** are the main ones analyzed.
    - Most dominant single carbon chain species

Mass Number	Possible Species
12	C
13	CH
14	N,CD
15	CDH
16	CD_2,O
17	CD_2H,OH
18	H_2O,CD_3,OD
19	CD_3H,HDO
20	CD_4,Ar^{++},D_2O

Results





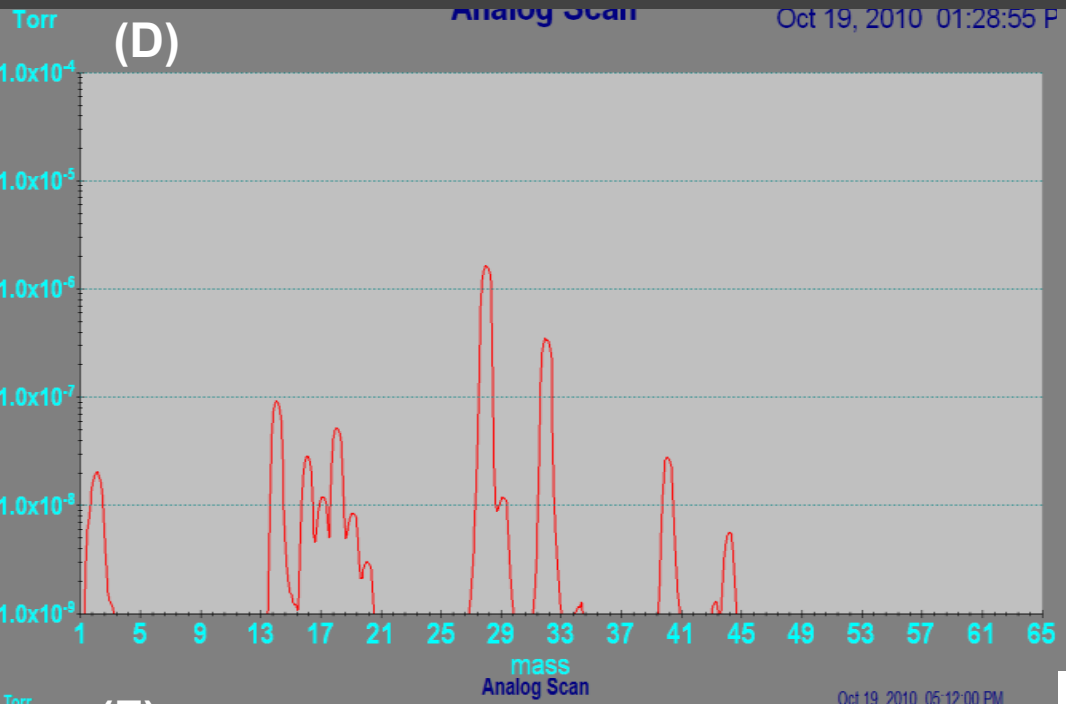


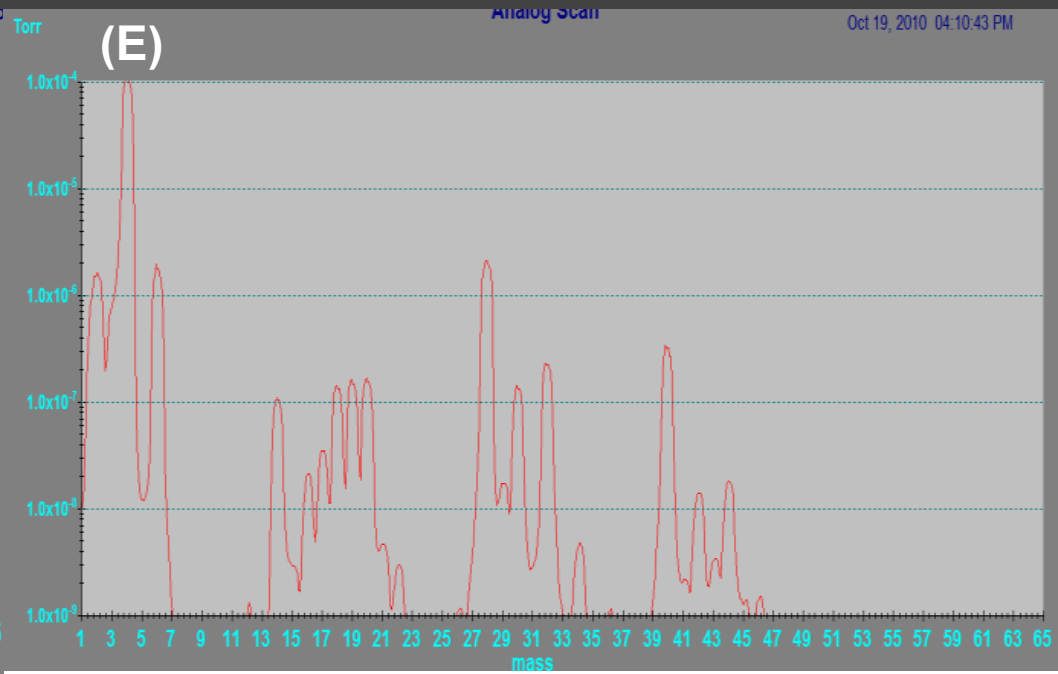
Upper Left: **Wall** no gas - Case (A)

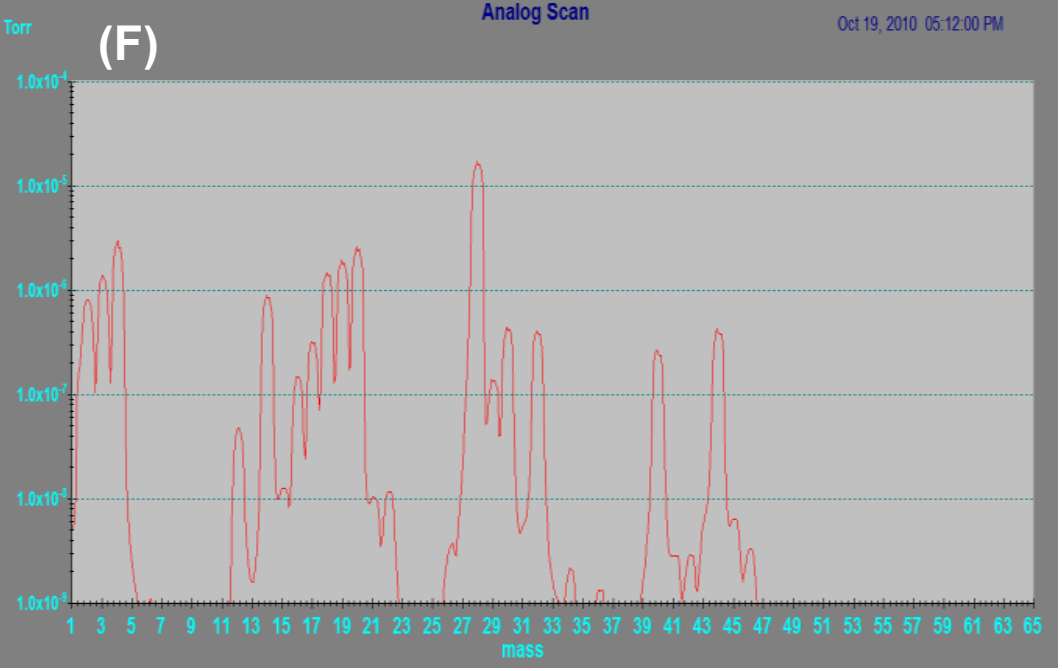
Above: **Wall** with **D_2 gas** – Case (B)

Left: **Wall** with **D_2 plasma** – Case (C)

Results





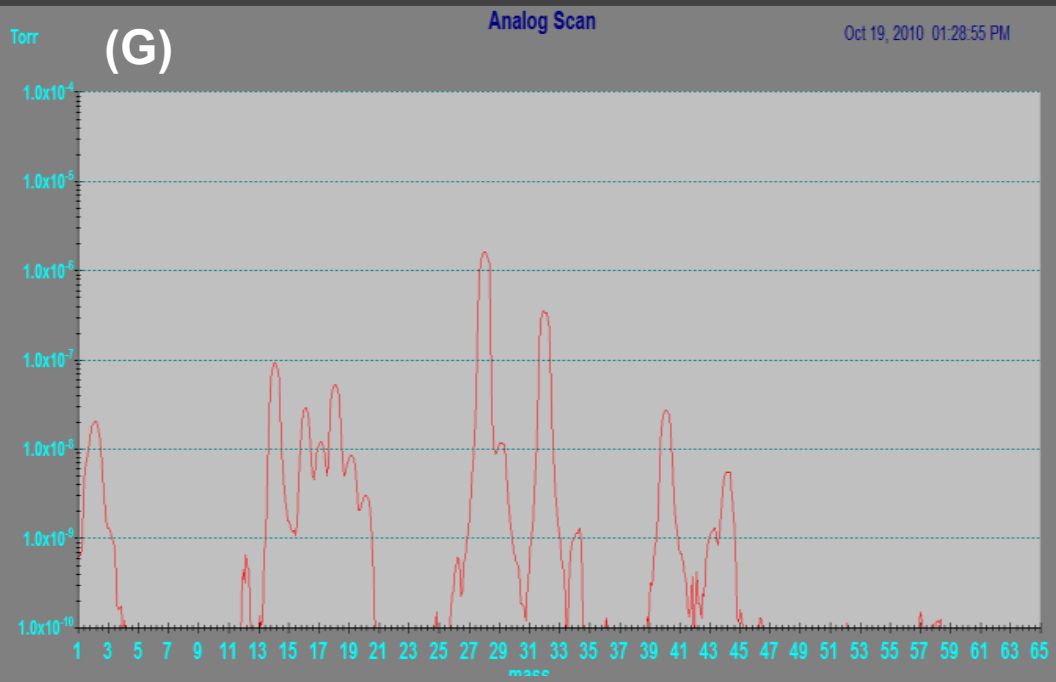


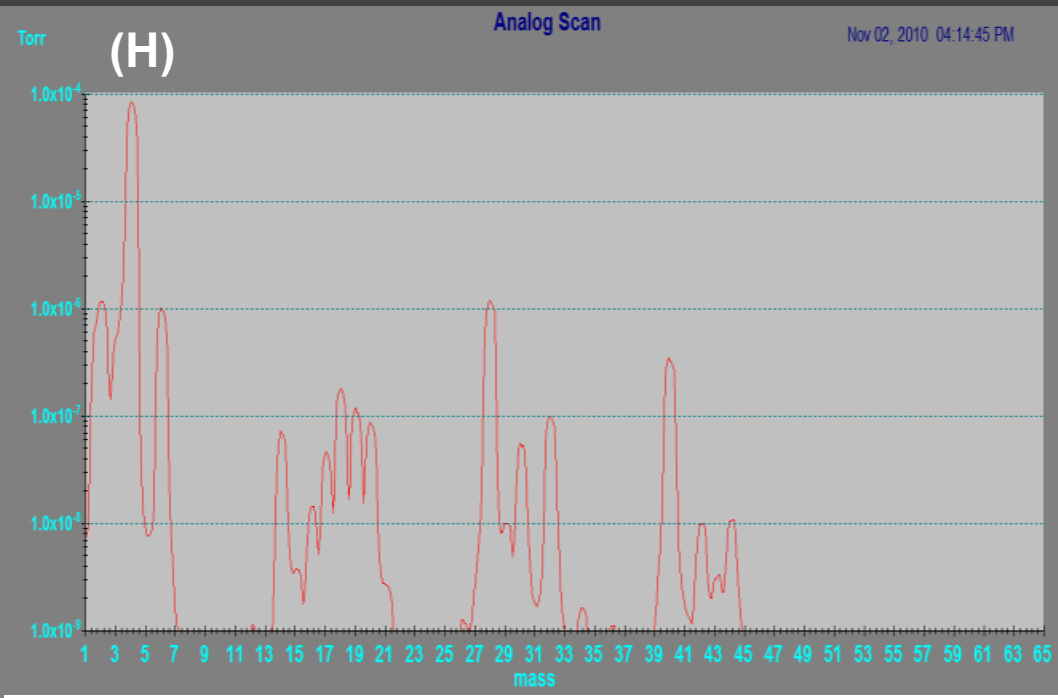
Upper Left: **ATJ + Wall** with no gas - Case (D)

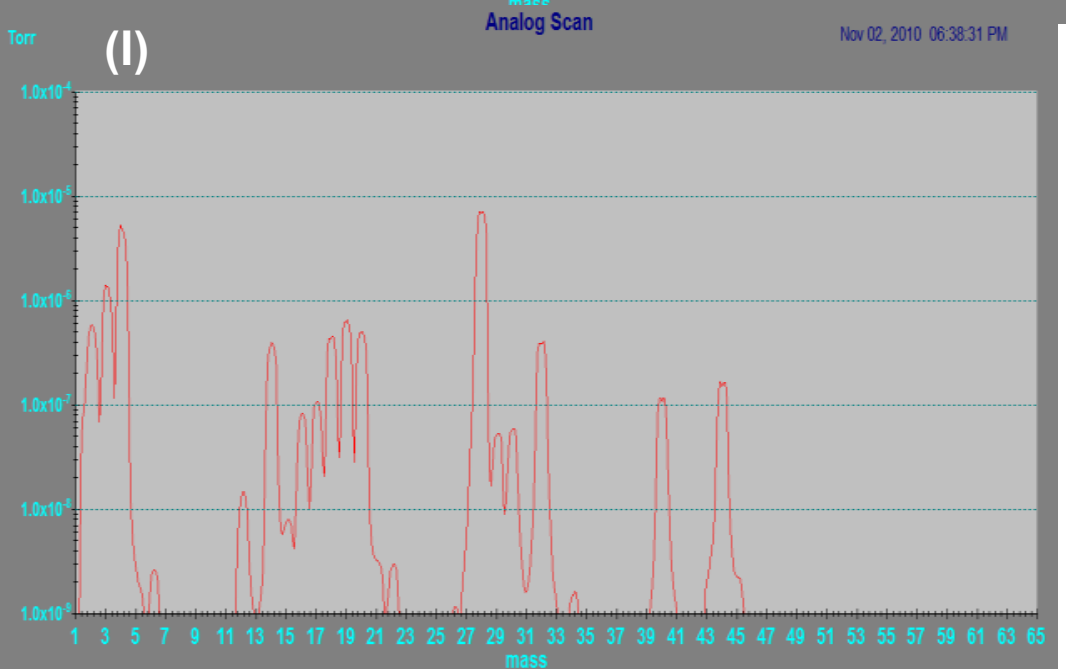
Above: **ATJ + Wall** with **D_2 gas**, no plasma – Case (E)

Left: **ATJ + Wall** case with **D_2 plasma** - Case (F)

Results







Upper Left: **ATJ + Wall + Lithium** - Case (G)

Above: **ATJ + Wall + Lithium** with **D_2 gas** – Case (H)

Left: **ATJ + Wall case + Lithium** with **D_2 plasma** – Case (I)

Data Analysis and Results

Mathematical Equations of the Analysis

$$S_{16} = 0.2040P_{CD_4} + 0.1020P_{CD_3H}$$

$$S_{17} = 0.444\,P_{CD_3H} + 0.1060\,P_{HDO} + 0.2120P_{H_2O}$$

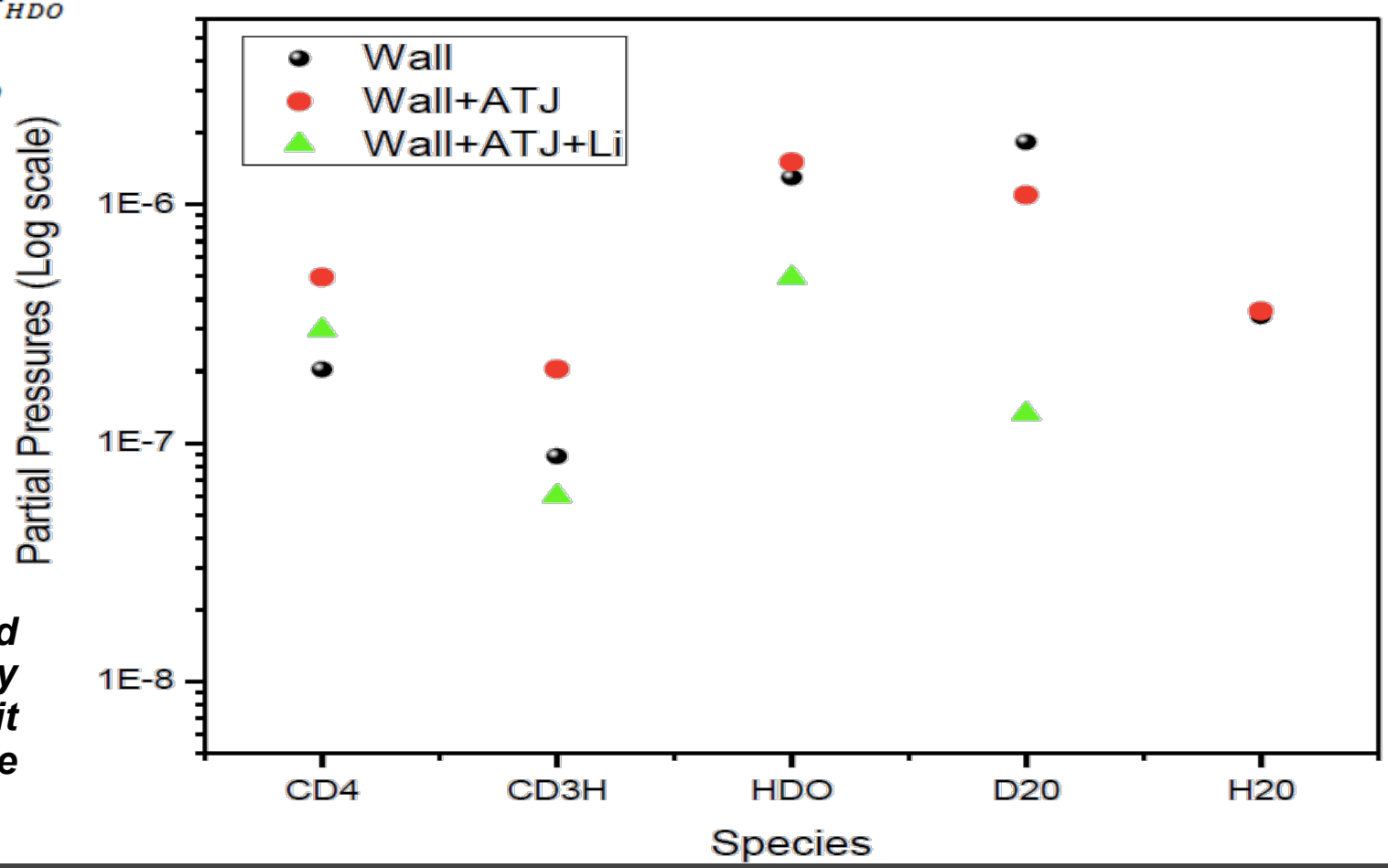
$$S_{18} = 0.8888P_{CD_4} + 0.444\,P_{CD_3H} + 0.1060P_{HDO} + 0.2120P_{D_2O} + 0.9999P_{H_2O}$$

$$S_{19} = 0.9999P_{CD_3H} + 0.9999P_{HDO}$$

$$S_{20} = 0.9999P_{CD_4} + 0.9999P_{D_2O}$$

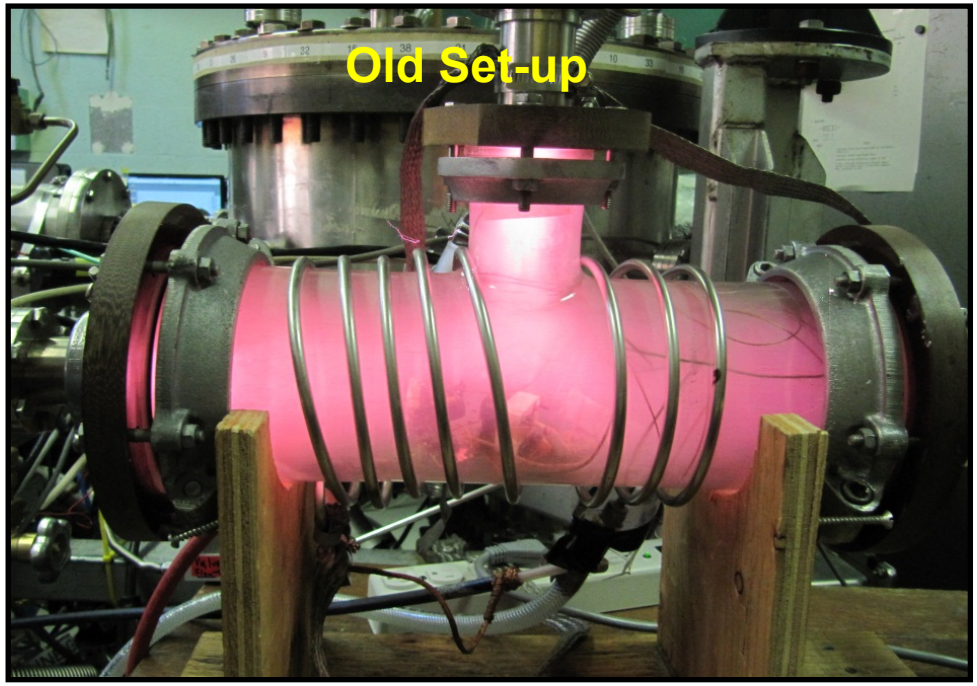
Mass Number	Possible Species
12	C
13	CH
14	N,CD
15	CDH
16	CD_2,O
17	CD_2H,OH
18	H_2O,CD_3,OD
19	CD_3H,HDO
20	CD_4,Ar^{++},D_2O

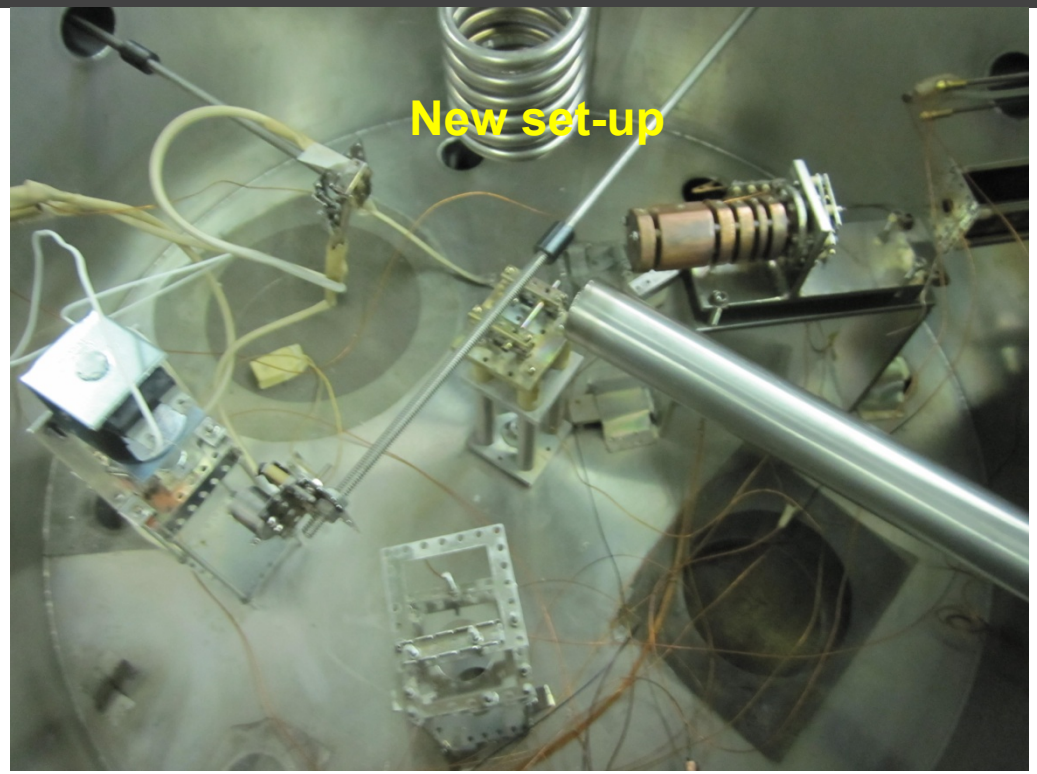
Analyzing plasma-gas contribution



For the analysis presented here, we consider only single carbon chains as it more likely than a double or triple carbon chain

Work in Progress





- Installed electromagnetic shutter for lithium evaporator
- Internal RF coil.
- Movable target set-up and faraday cup.
- Sniffer tube connected to the RGA
- This set-up could be used for plasma as well as beam experiments.
- Working on Chemical erosion measurements.
- Have tested RF plasma.

Results and Conclusions

- Lithium treatments show the suppression of all the considered products (CD_4, CD_3H,in the analysis.
- The chemical erosion studies conducted in a plasma chamber, as opposed to beam experiments, provide a better understanding of the phenomenon taking place in tokomaks.
- It was found that D_2O and HDO are present in relatively large amounts compared to the expected chemical erosion products. At present, we believe that these are due to the contribution from the walls (due to pre-existing lithium).
- D_2O and HDO are also suppressed. As lithium is evaporated in situ onto ATJ graphite target, some lithium is also deposited on the walls of the chamber and this fresh lithium is believed to be the reason for suppressing the contribution of D_2O and HDO coming from the walls.
- More detailed experiments are being planned for quantitative chemical erosion measurements.

Paper published: V. Surla, P. Raman, D. Burns, M.J. Neumann, D.N. Ruzic, Physical and chemical erosion studies of lithiated ATJ graphite, Journal of Nuclear Materials, In Press, Corrected Proof, Available online 1 February 2011, ISSN 0022-3115, DOI: 10.1016/j.jnucmat.2011.01.051.

Outline

- Chemical Erosion Studies of Lithiated ATJ Graphite
- Wetting of Lithium on Boronized Plasma Facing Component Materials
- Electrostatic Lithium Injector

Wetting of Lithium on Boronized Molybdenum

- Previous studies have shown that Boronization helps reduce particle recycling, achieves a higher density limit, higher plasma current, lower edge safety factor and wider operating region and promotes plasma recovery after events such as oxidation, shutdown and air leaks.

- Lithium treatments in the National Spherical Torus Experiment (NSTX) have shown dramatic improvements in plasma performance increasing the viability of lithium as plasma facing component (PFC) material

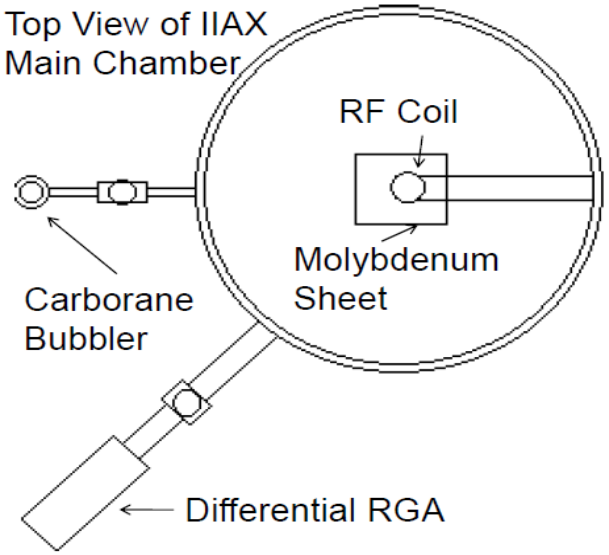
- Molybdenum was investigated as a target material because it is being installed as the inboard diverter material in NSTX.

- Wetting of Lithium on plain and boronized molybdenum has been carried out.

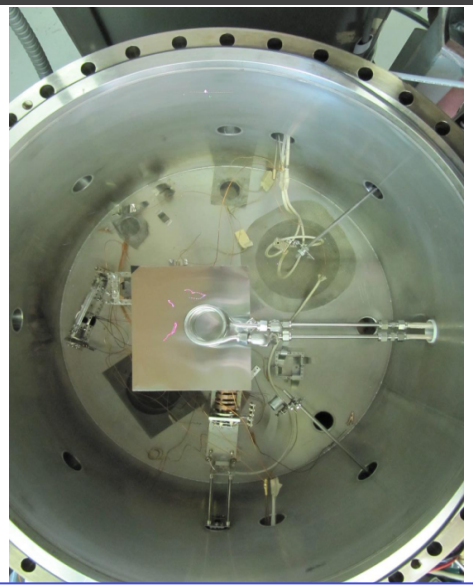
- Boronization is accomplished using carborane ($C_2B_{10}H_{12}$) powder.
  - Nontoxic and noncombustible, hence used for boronizing the molybdenum target [1]. External carborane bubbler chamber consisting of carborane evaporator cup is attached to the main chamber.

[1] J.H. Wu et al., Recent results of boronization on EAST and HT-7 superconducting tokamak, Journal of Nuclear Materials, DOI: 10.1016/j.jnucmat.2010.10.089.

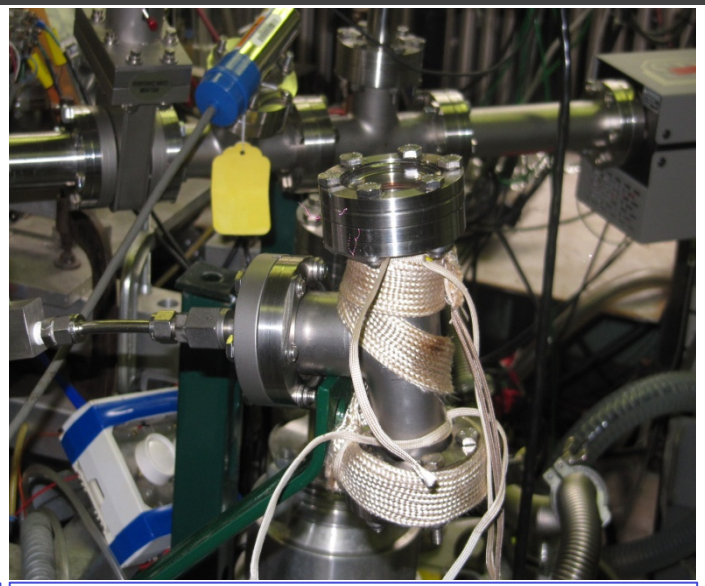
Experimental set-up



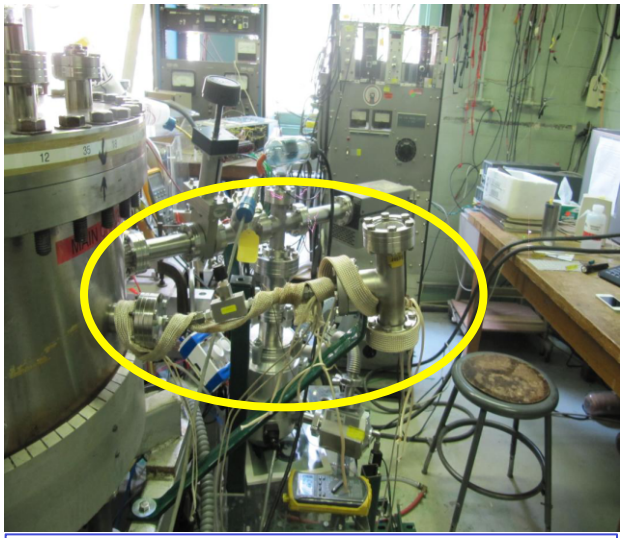
Top view of IIAX Main chamber



Main chamber: Internal RF antenna and Molybdenum sheet

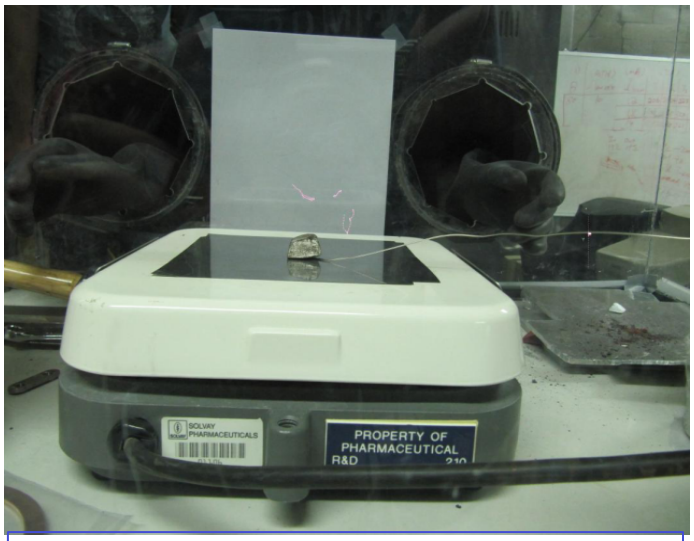


The bubbler used to evaporate carborane into the main chamber.



External Carborane bubbler: Window port, evaporation cup and heated gas inlet to the main chamber

Contact angle measurement setup



Heater setup inside Argon glove box

Experimental details

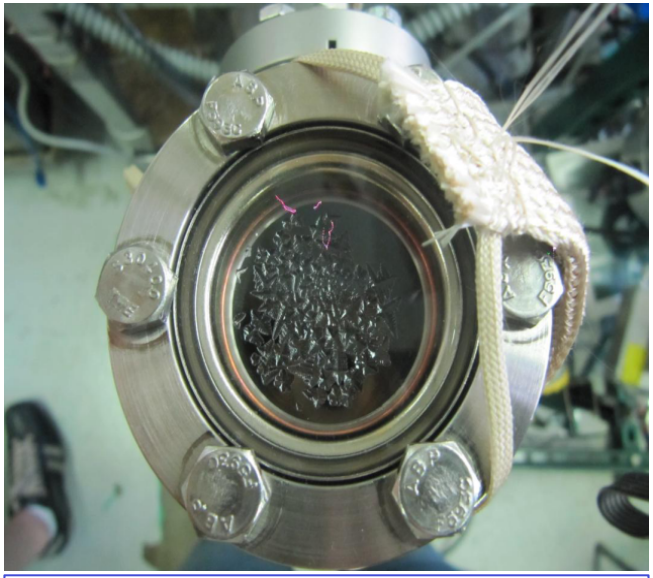
Experiment	Amount of Carborane	Molybdenum sheet dimensions
1	200 mg	16 cm x16 cm
2	400 mg	16 cm x 16 cm

Boronization Experiments

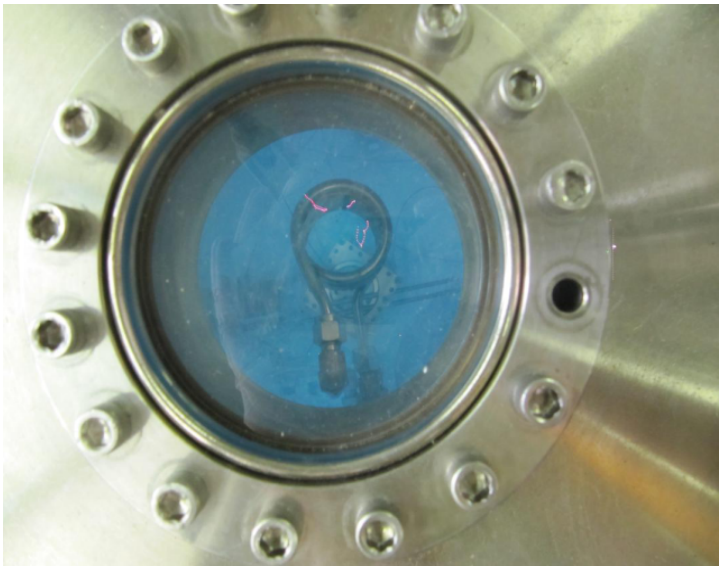
Experiment	Substrate
1	Plain Molybdenum
2	Boronized Molybdenum sheet 1 (200g Carborane)
3	Boronized Molybdenum sheet 2 (400g Carborane)

Contact Angle Experiments

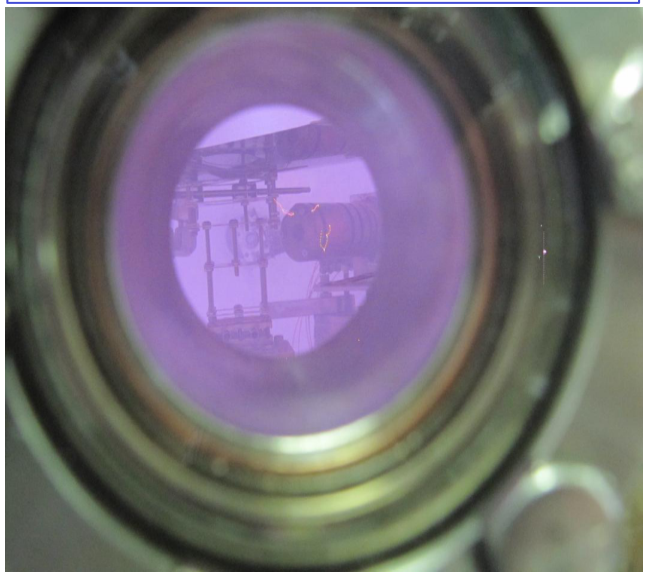
Evaporation Cup: Carborane heated to 120 C



Carborane condensation on to the window port

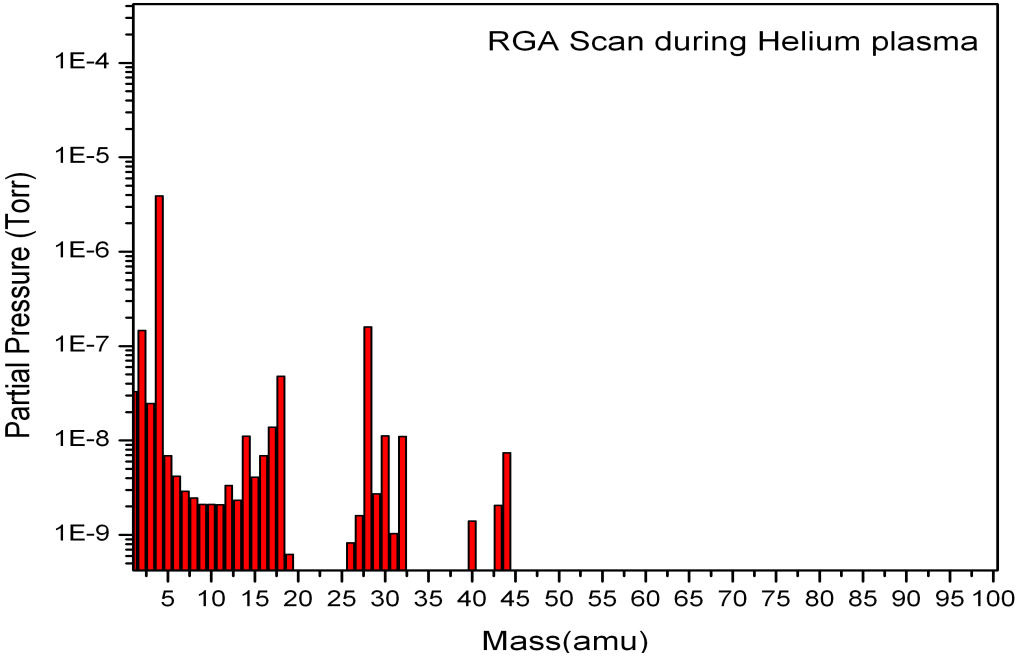


Helium Plasma

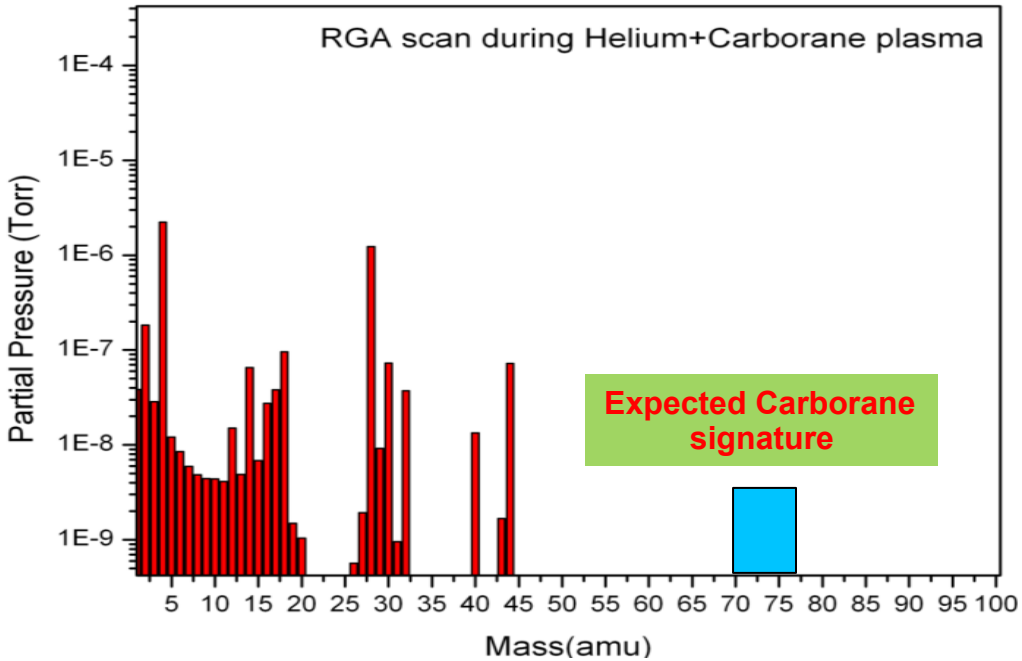


Plasma during boronization

Results



RGA spectrum during Helium plasma

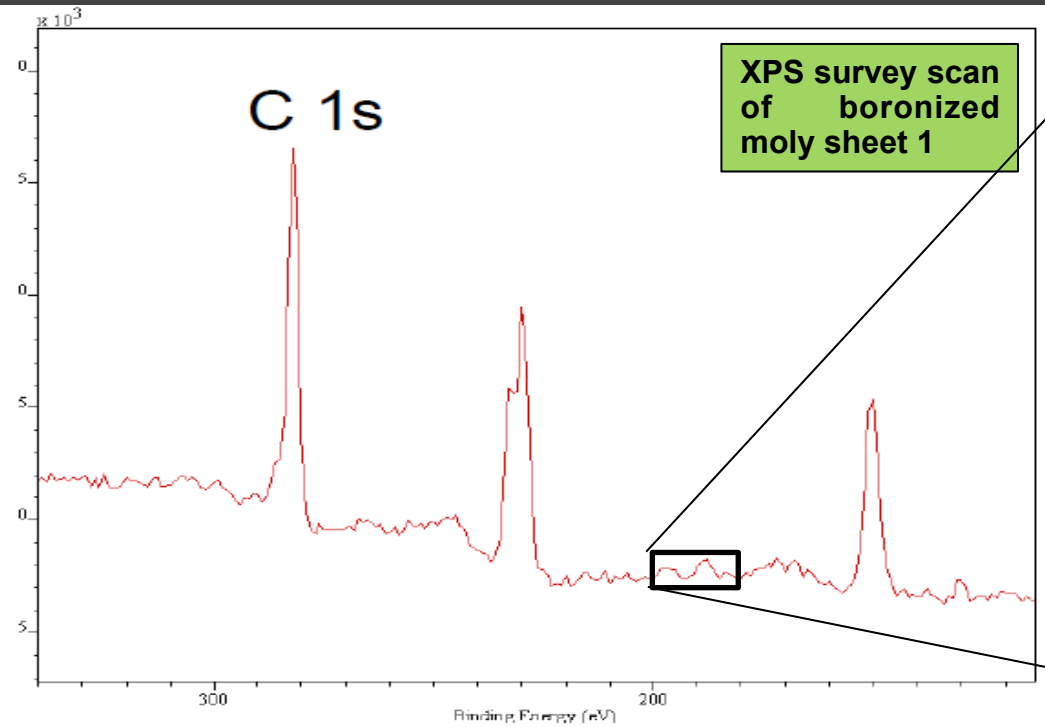


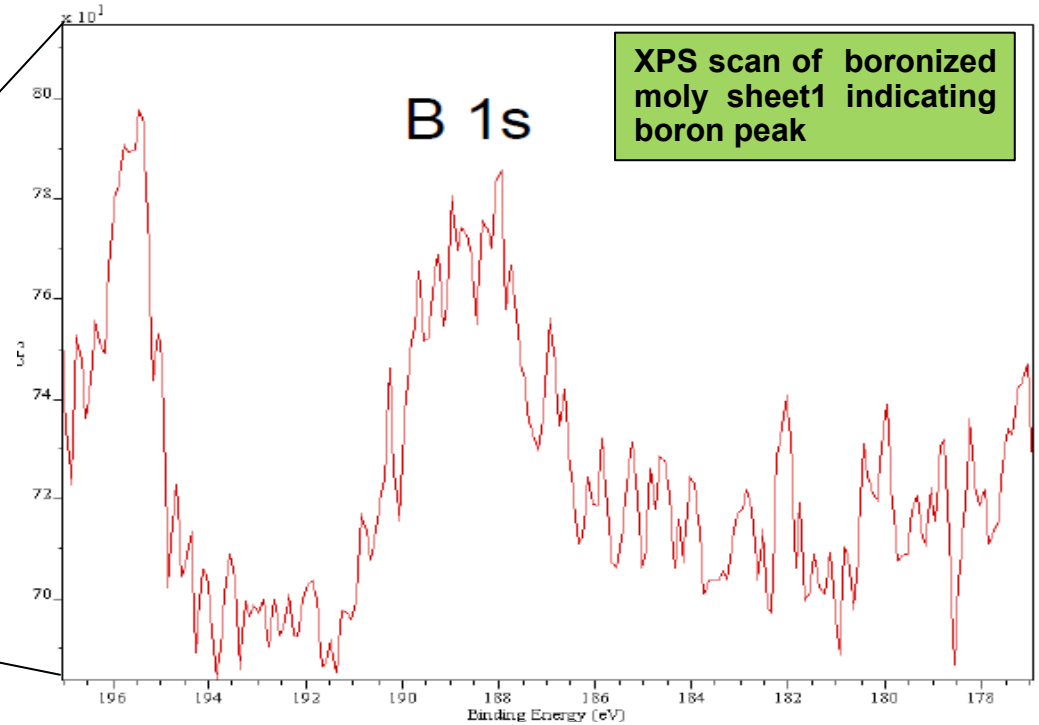
RGA spectrum during boronization

- Expected Carborane signature not found during boronization
  - Expect peak between 70 – 75 amu
  - Our T_e are much lower in Fusion glow discharges

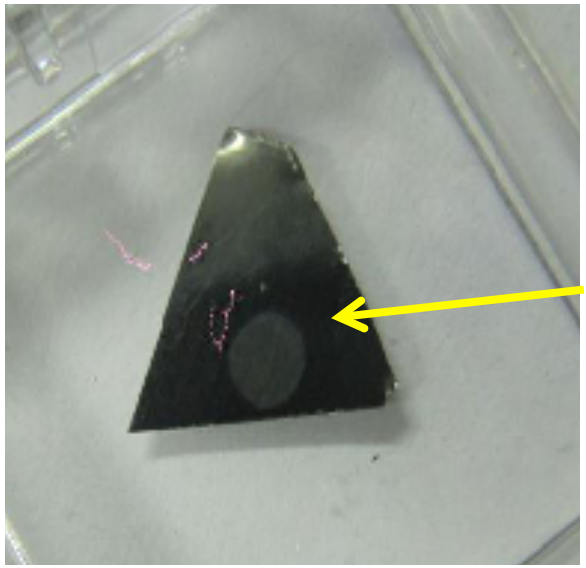
- How do we know we have Boronized?
  - Chemical analysis – XPS
  - Thickness estimated to be > 2 nm

Results: Boronized Sample



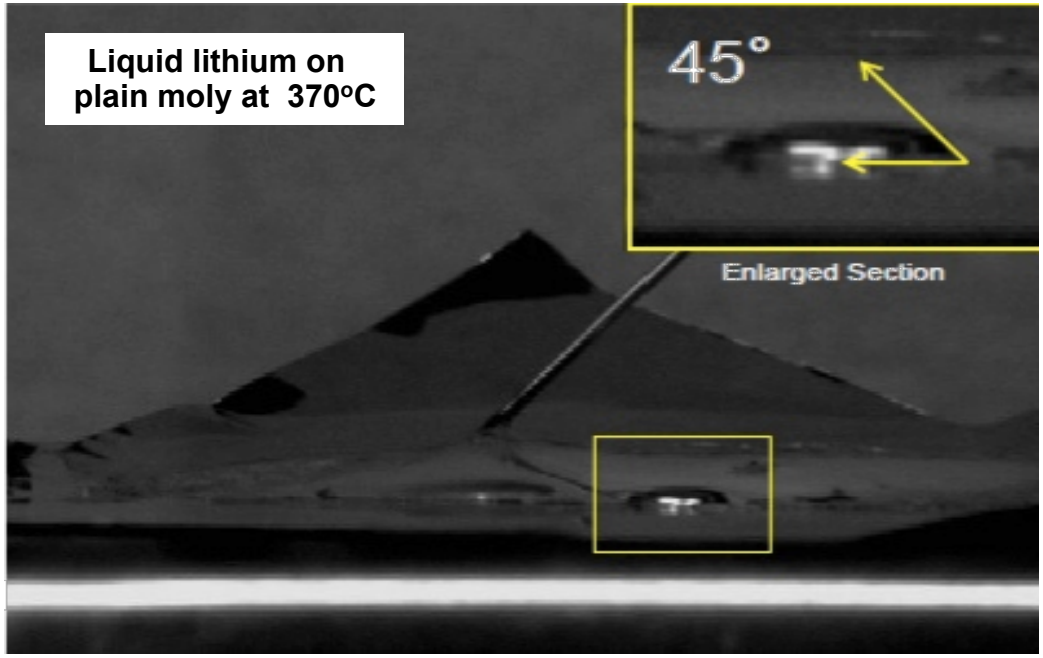


Substrate	Percentage of Boron	Percentage of Carbon
Boronized sample 1	9 %	91%
Boronized sample 2	9.5%	90.5%

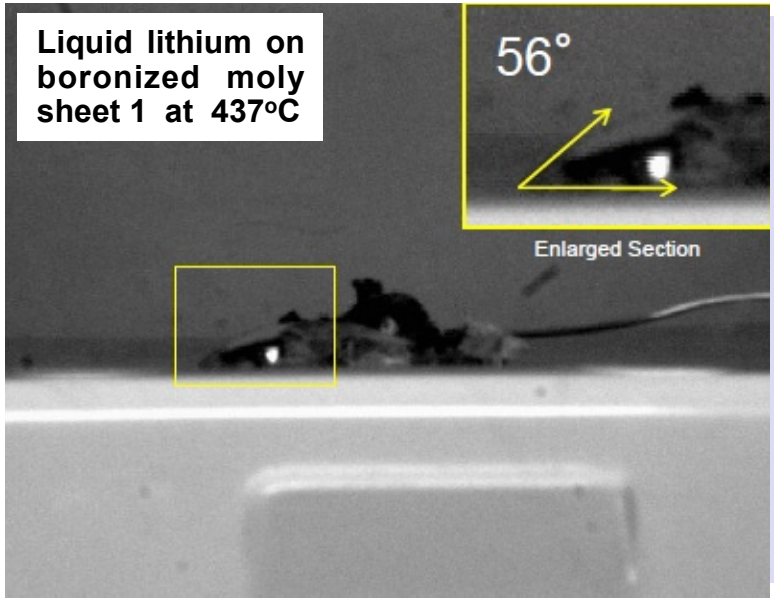


Visually can see the difference between plain and boronized molybdenum.

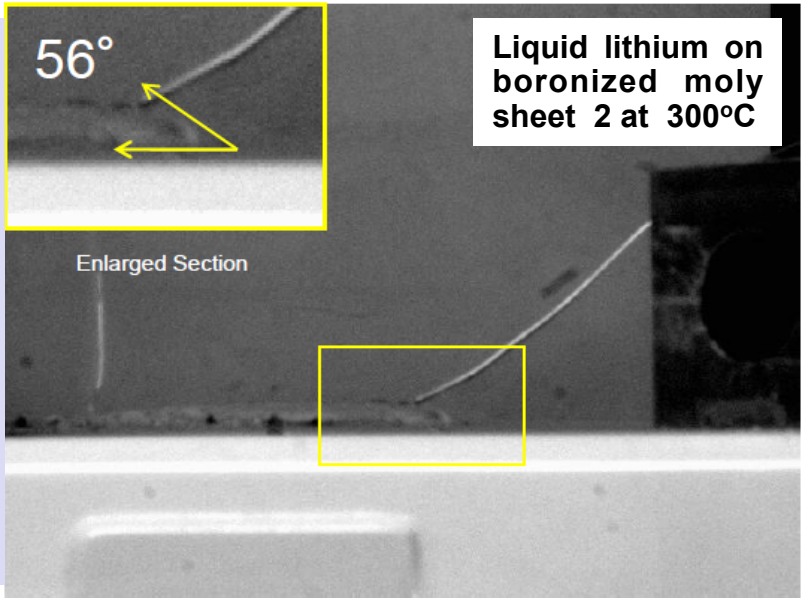
Contact Angle Measurements



- Contact angle for plain molybdenum 45°
- Borinized molybdenum contact angle 56°
  - Both the 200 mg and 400 mg samples measured the same contact angle
- Contact angle still les than 90°
  - **Lithium wets boronized molybdenum**



- **Moly sheet 1 run with 200 mg of carborane**
- **Moly sheet 2 run with 400 mg of carborane**
- **Contact angle for both boronized cases was 56° an increase on the non-boronized case of 11°.**
- **Evidence that Li wets boronized Moly**



Future Work

- Magnetron sputtering of boron target and lithium dropper arrangement is in progress to increase to the boron content of the deposited film and to get better contact angle measurements respectively.

- Ionization fraction measurement of lithium on mixed materials: When lithium is sputtered, it has been observed that 55-60% of the sputtered particles are ions [3]. This is ideal since the sheath returns them to the wall.
  - However, Li on top of other layers may act differently. To examine this phenomenon and assess erosion measurements of lithium on mixed materials (Li/Mo, Li/B/Mo), ionization fraction measurement of the sputtered lithium in the Ion surface InterAction eXperiment facility is currently is in progress.

[3] J.P. Allain and D.N. Ruzic, *Nucl. Fusion* **42** (2002), pp. 202–210.

Conclusion for Boronization

- Initial experimental results shows that Lithium wets boronized molybdenum.

- The contact angle increased by 11 degrees between the plain molybdenum and boronized molybdenum.

- Carborane is hard to ionize by a 1 – 3 eV electron temperature plasma. This is evident from the observation of no signature carborane peaks in the RGA spectrum and lower boron content in the deposited films

- Higher growth rate of boron film and higher degree of ionization and dissociation of carborane can be achieved in ~ 40 eV electron temperature plasma with relatively high density [2]. These conditions are typically produced in large fusion devices.

[2] O.I. Buzhinskij et al., Journal of Nuclear Materials 313–316 (2003), pp. 214–218.

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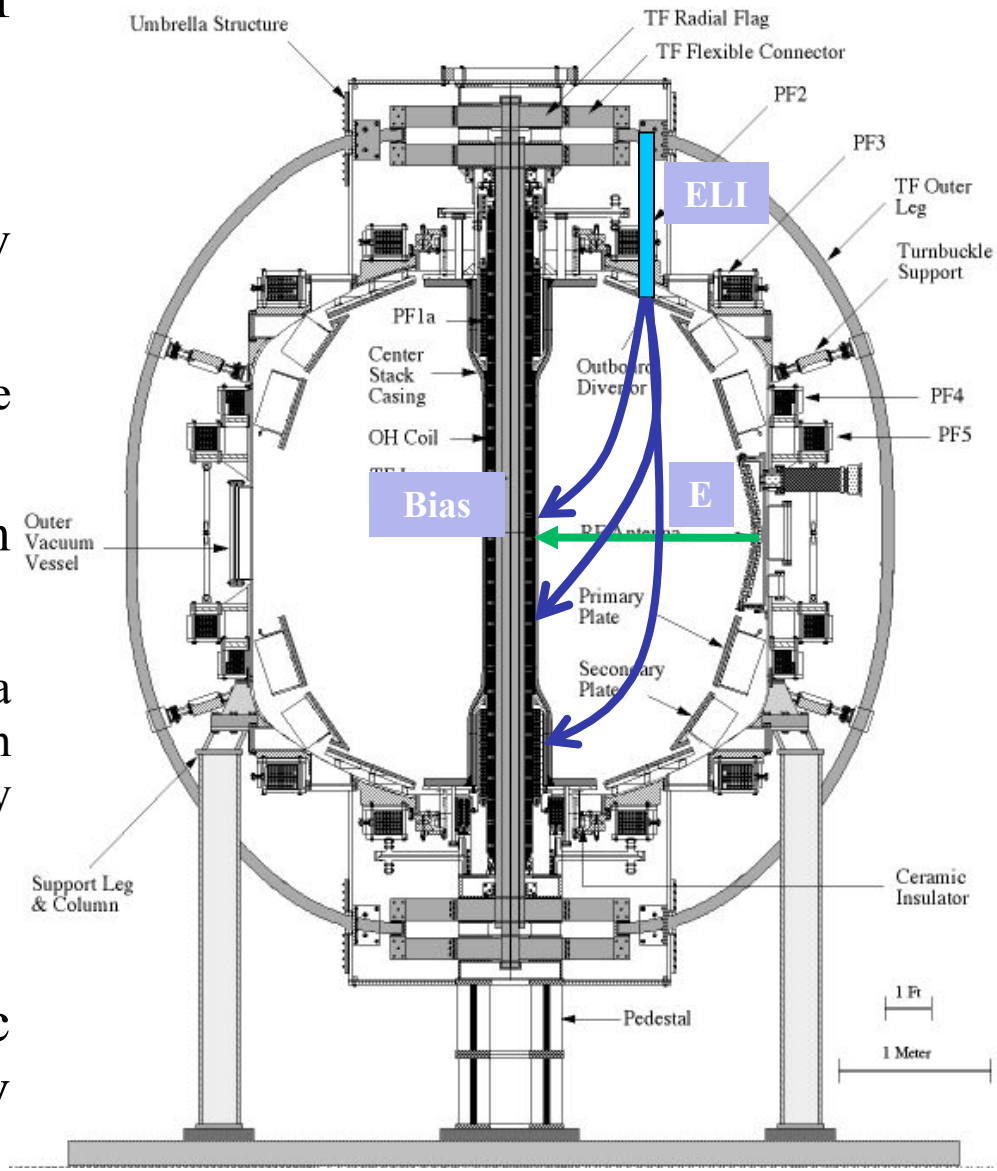
Electrostatic Lithium Injector (ELI)

- Coating of components in NSTX is of importance
  - LITER used to put Li into NSTX.
- One area of NSTX that is not reached by LITER which is line of sight.
  - Center-stack is thin, small target that cannot be uniformly coated.
- A possible solution to this problem may lie in the electrospray concept.
  - Electrospray is currently used to produce a charged aerosol, which with proper application of an electric field, could be used to selectively "paint" components of NSTX.
  - Run before or in between shots.
- ELI could be used to re-coat a specific area where lithium has been eroded away during a run.



Aerosol production and modeling

- The tube has a bias with a ground ring at the nozzle end.
  - As a drop forms, charges are drawn to it via the E-field produced between the tube and ground ring.

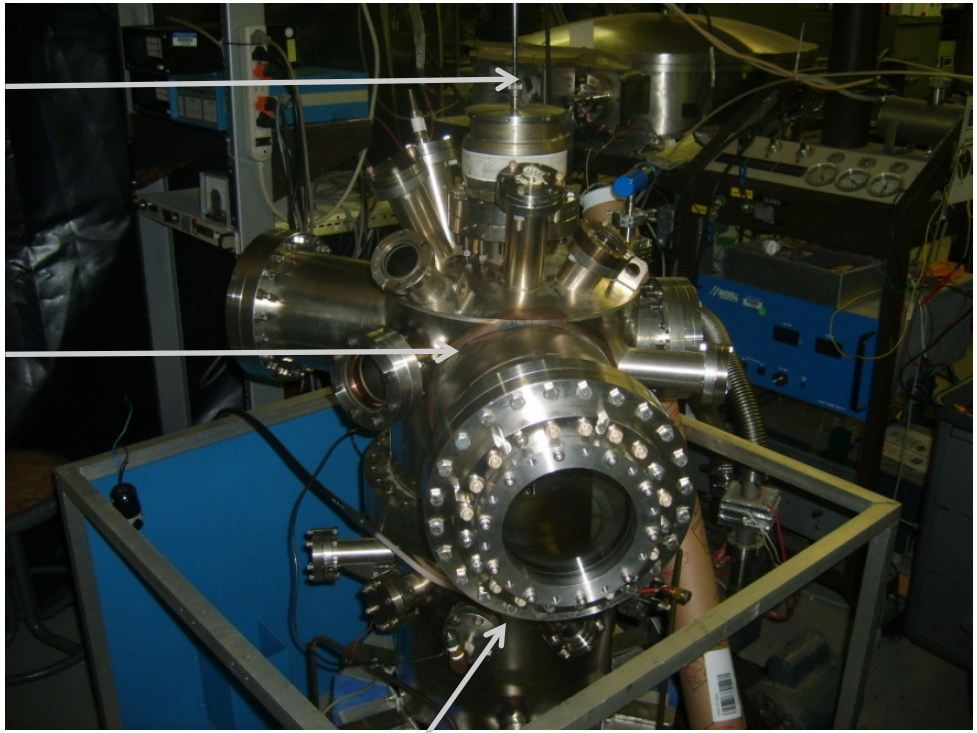
- As the droplet charges up the force due to the ion repulsion, F_i, is balanced by the force due to surface tension, F_t.
  - At some point $F_i > F_t$ and the droplet breaks apart into a charged aerosol mist

- Modeling work is being done to investigate the possibility of how to use time dependent electric fields to manipulate the trajectory of a charged lithium droplet within a tokamak to be able to use an electrostatic lithium injector for repair of PFCs and ELM control.

ELI Prototype

ELiI Tube

ELiI Chamber



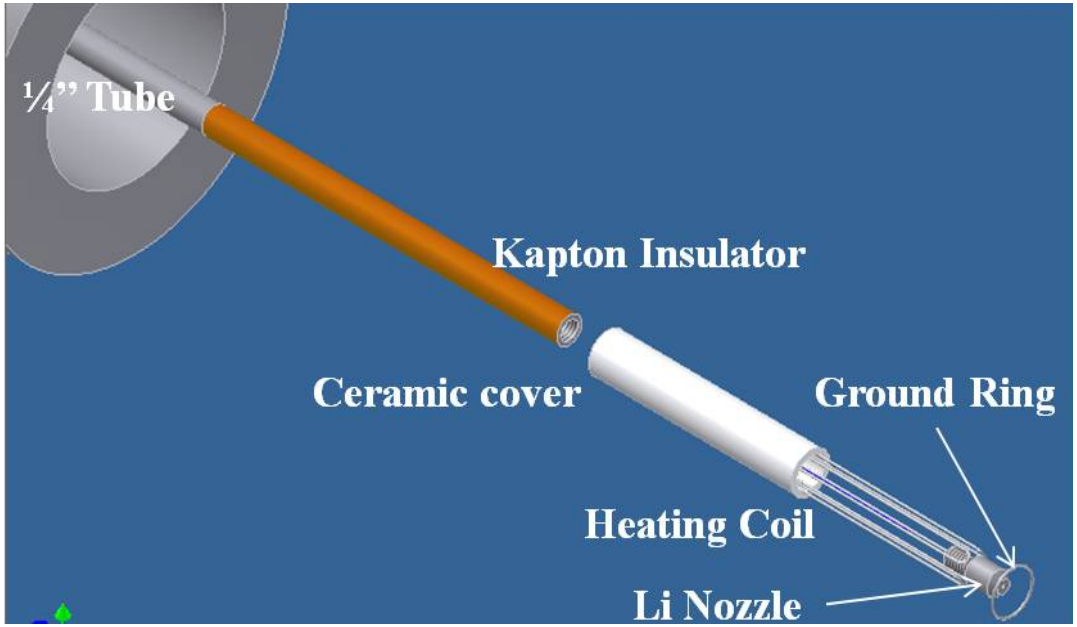
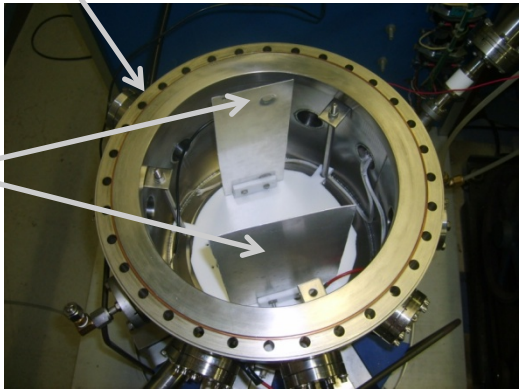


Plate Chamber

Bias Plates



Summary

- Chemical erosion studies on ATJ graphite
  - Using deuterium plasma
  - Results show that Li indeed does have a positive effect in suppressing chemical erosion.
- Lithium wetting of Boronized of molybdenum
  - Carborane used to boronize molybdenum
  - slightly changes the lithium contact angle: 45° – 56°
  - but lithium still wets boronized molybdenum
  - Future studies using molybdenum with magnetron sputtered boron
- A new concept of using and electrospray to produce a aerosol of lithium to coat the inside of NSTX.