Initial results from the NSTX Deposition Monitor

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- Experimental setup
- Deposition during individual discharges
- 4-week record
- Elemental / chemical analysis

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Port Townsend, WA, July 28-31, 2003

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Workshop Agenda

NSTX Deposition Monitor

- Principle: quartz crystal oscillates at ~ 6 MHz, exact frequency depends on mass and on temperature.
- Deposition inferred from change in frequency (measured to ~1Å, ~1Hz)
- Detector is located in 4” tube outside main plasma chamber

  ~ 80 cm from last closed flux surface
- Detector configuration mimics typical diagnostic windows and mirrors,

  - samples neutrals deposited on NSTX wall away from plasma.
- Gate valve permits sample retrieval for surface analysis without machine vent
- Instrument operational since January ‘03, initial results presented.

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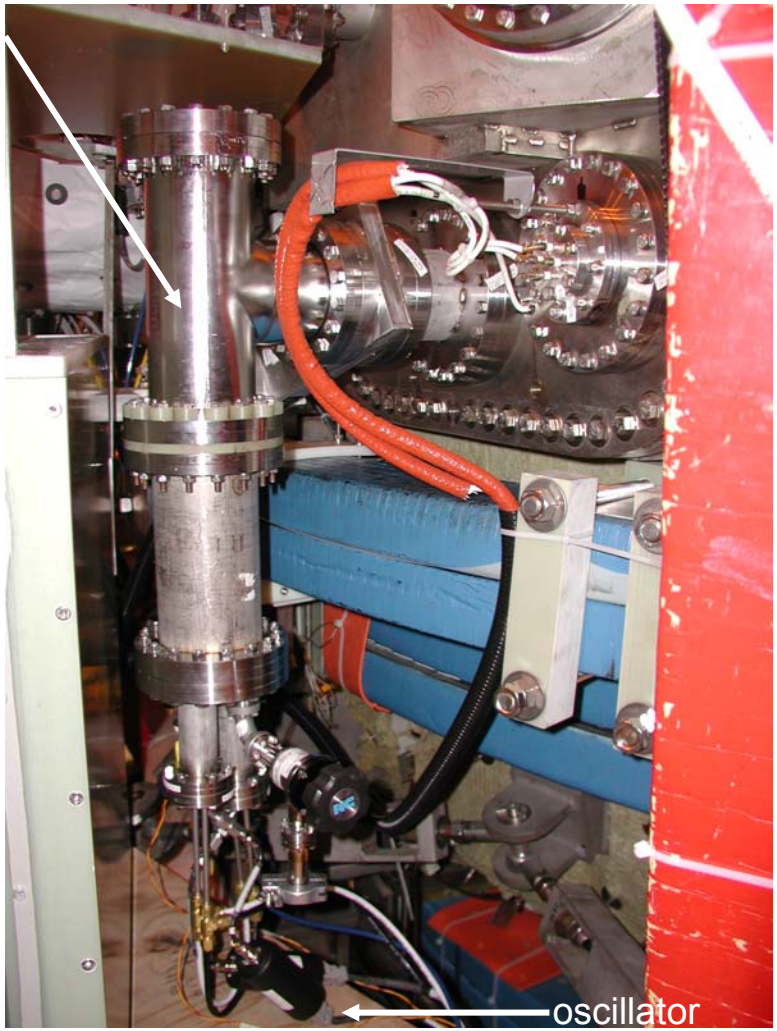
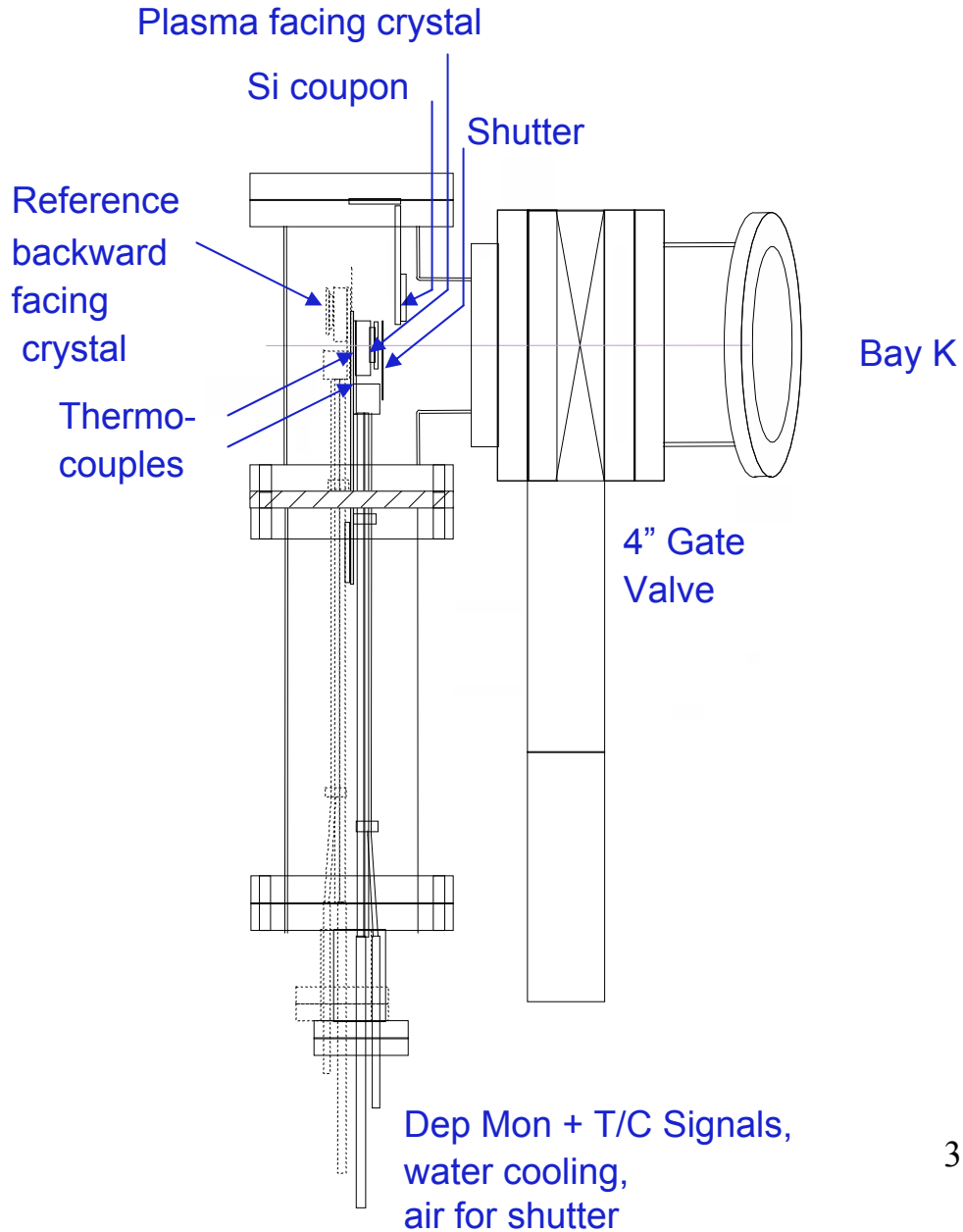
NSTX Bay K Deposition Monitor

detector
inside



crystal @ R= 231 cm
~ 80 cm from last closed flux surface 33 cm below midplane



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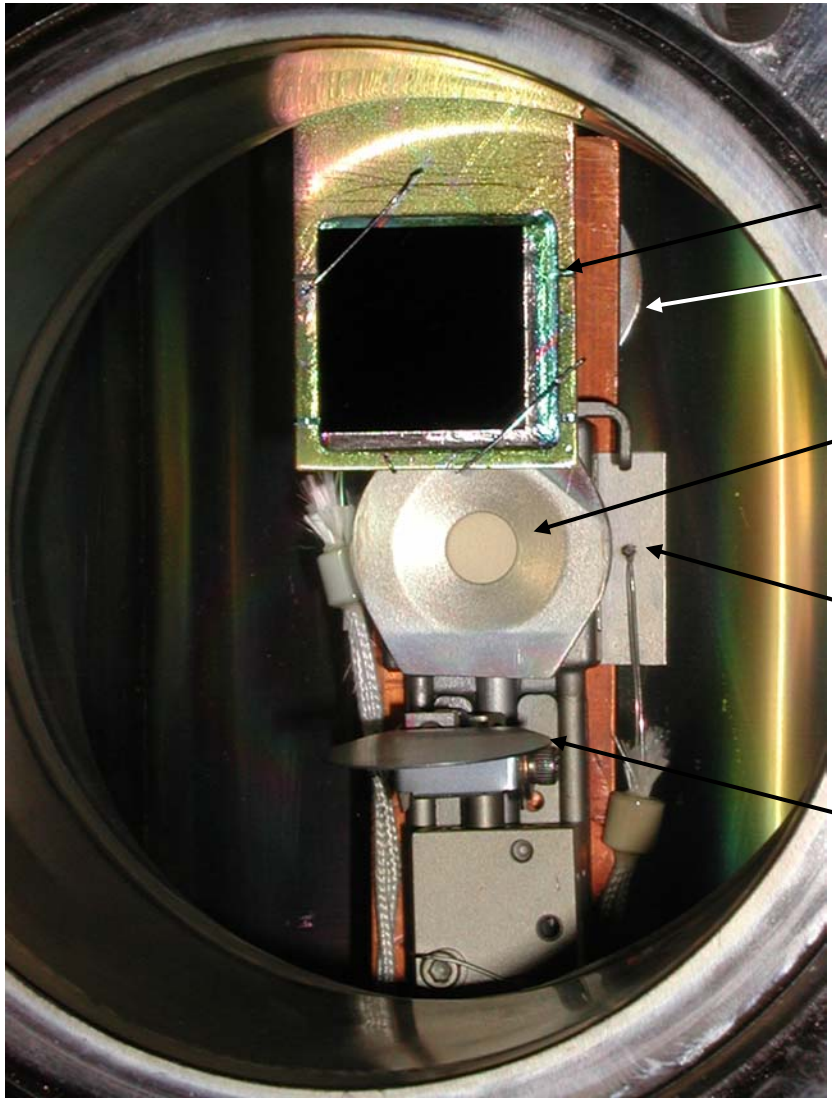
Deposition Monitor

Specification:

- Detector resolution ≈1 Å (≈ one monolayer).
- Accuracy 0.5%
- Bakeable to 450°C - water cooled during deposition monitoring.
- Built in shutter on plasma facing detector.
- Thermocouples on both detectors record temperature.
- Assume density 2.37 g/cm$_3$ (B_4C) and 100% impedance match
- Thickness calibration planned for 'tokamakium'



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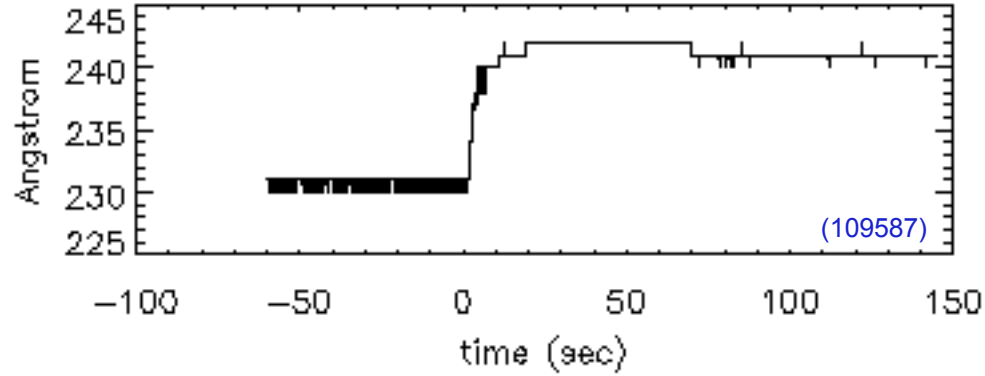
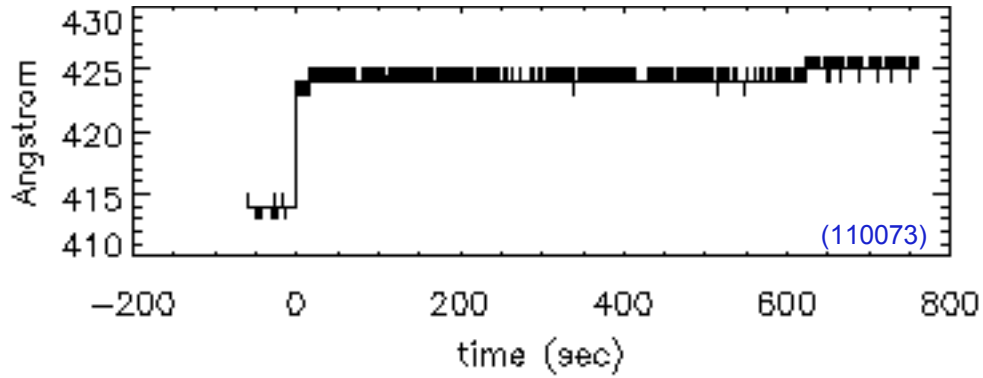
Deposition

11 Å deposition during CHI XP
(assume ρ=2.37)

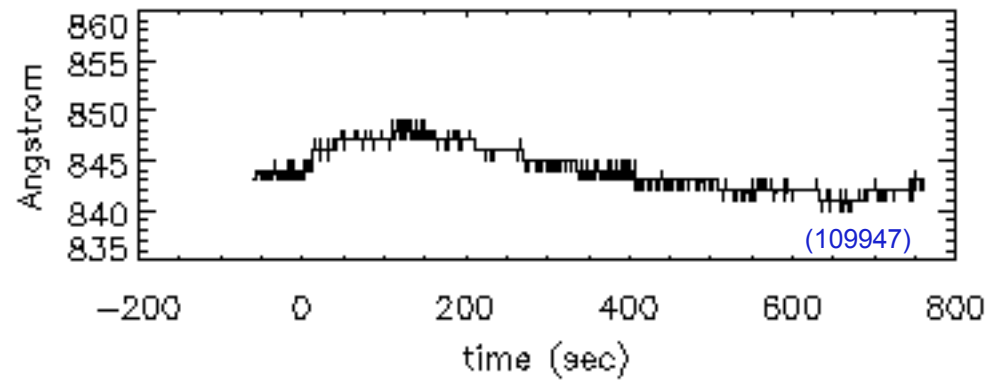


and during XP228



But more typically, 1 shot deposition hard to resolve

(oscillation due to water cooler cycling, improvements in progress)



Fortunately absolute measurement of 6 MHz crystal oscillation frequency easily reveals long term trend.

(Reference point: 1 Å hydrocarbon layer ~ same D content as NSTX plasma.
100 - 200 Å layer seriously degrades mirror reflectivity)

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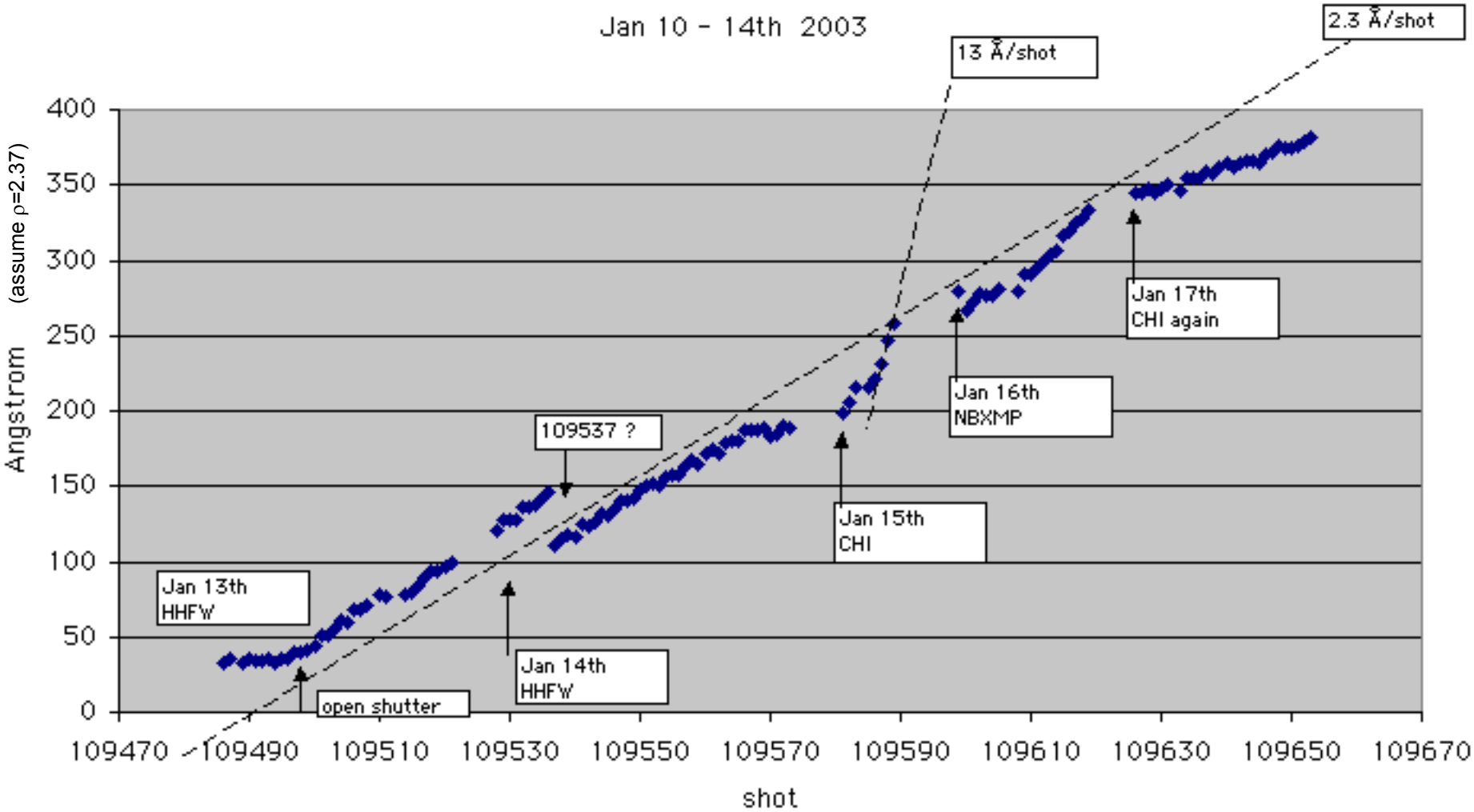
Shot-by-shot data from four run weeks with a variety of plasma conditions
Week 1:



Each data point represents one discharge

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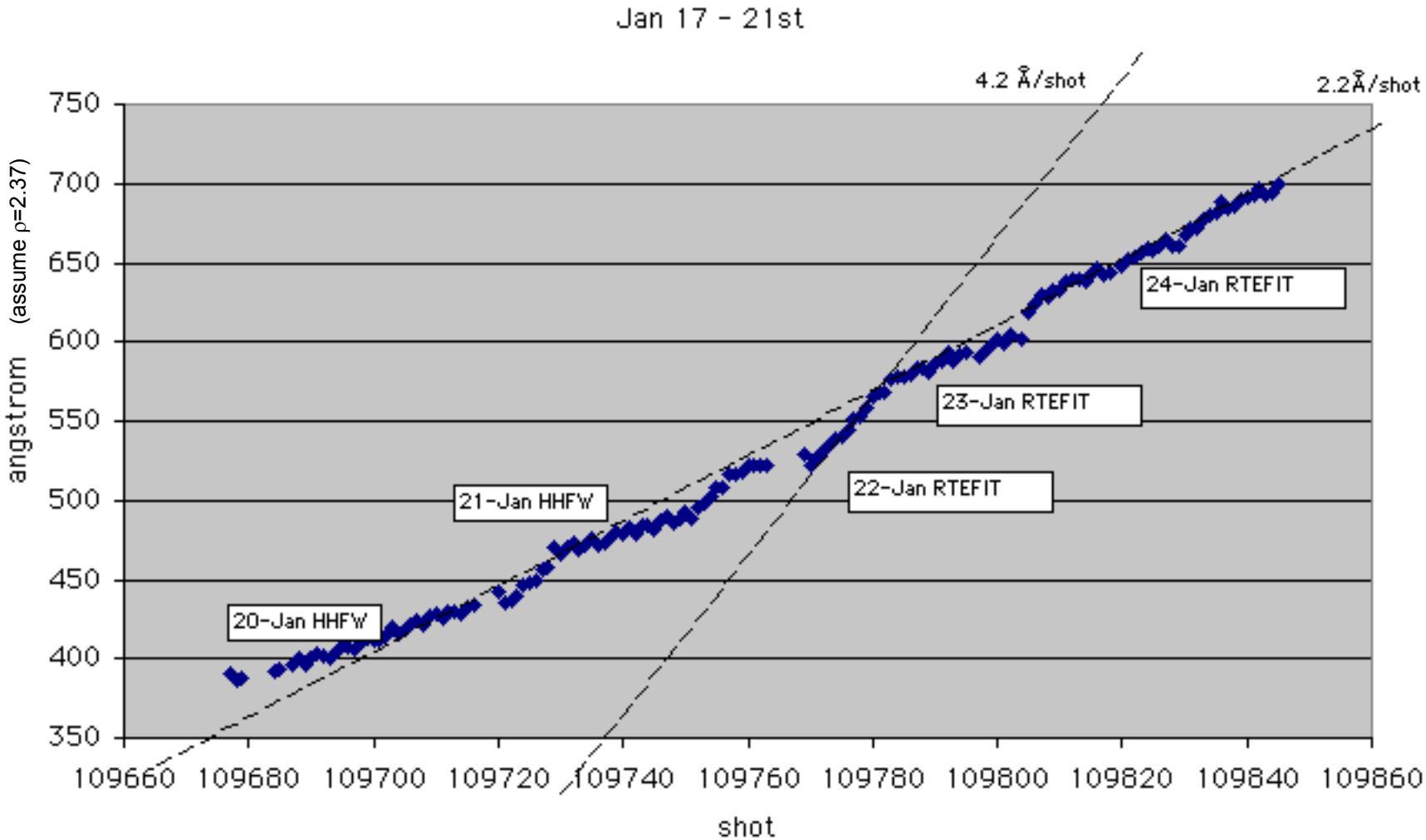
Long term trends are clear:



HHFW: High Harmonic Fast Wave experiments
RTEFIT: real time EFIT control experiments

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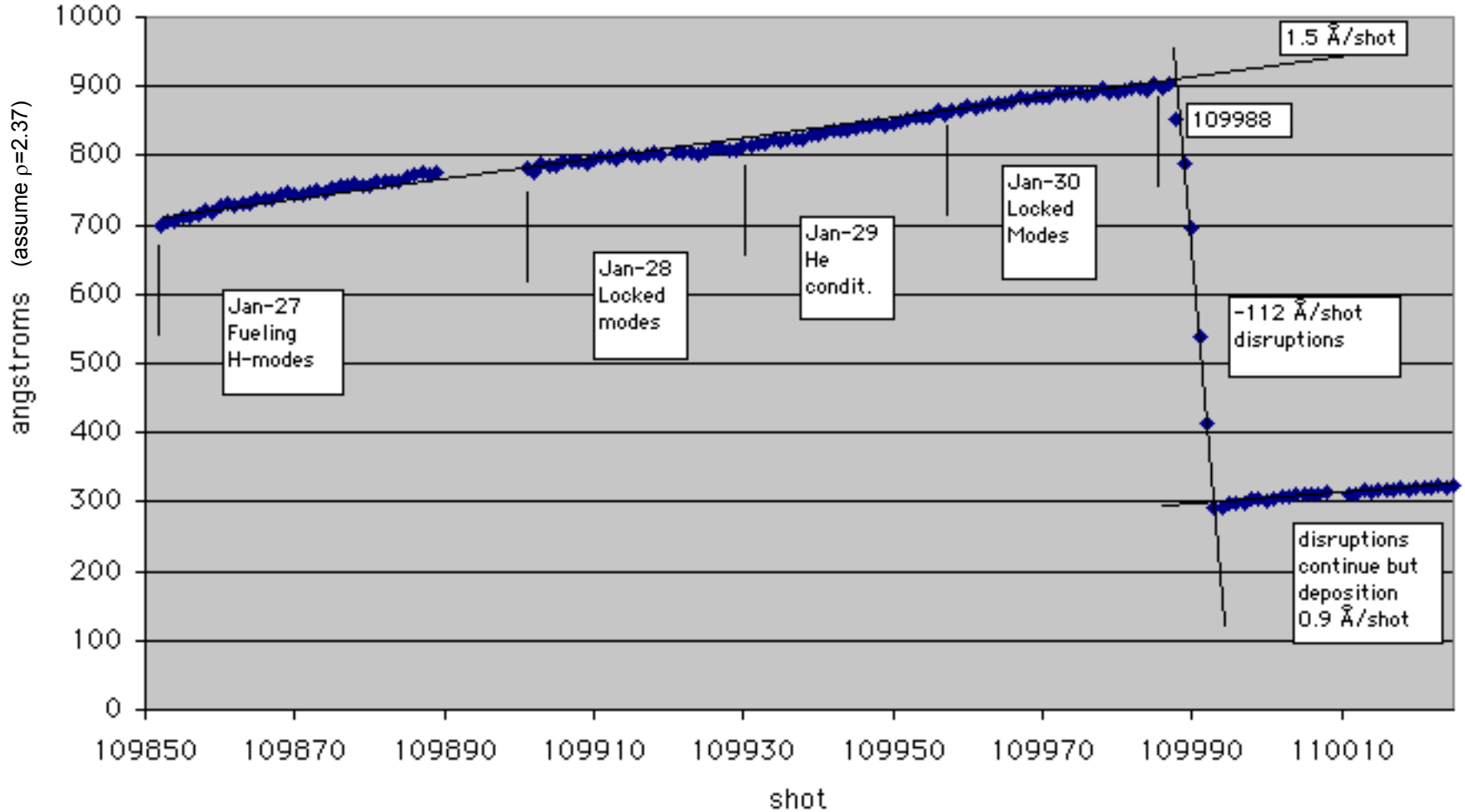
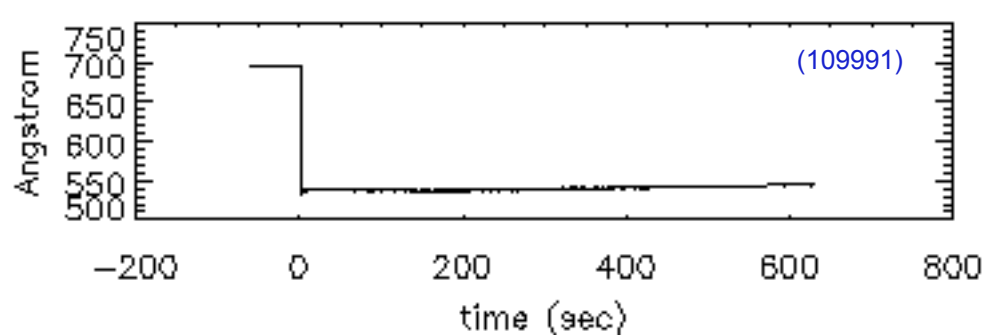
Erosion apparent during high beta disruptions





Erosion 109988 - 109993 presumably due to "disruptive discharge cleaning"

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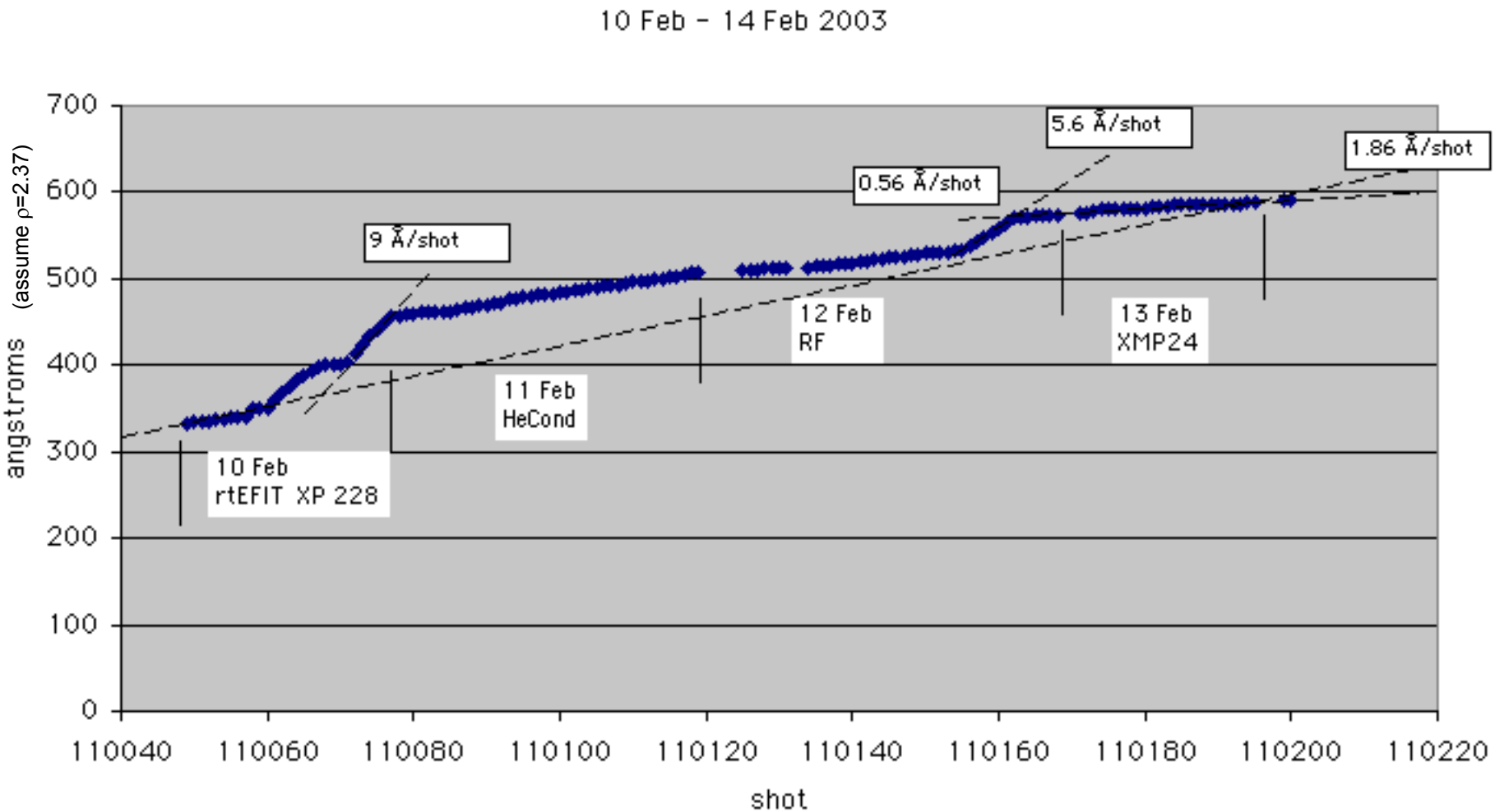
High deposition during XP228



XP228 is exploration of long pulse high triangularity plasmas

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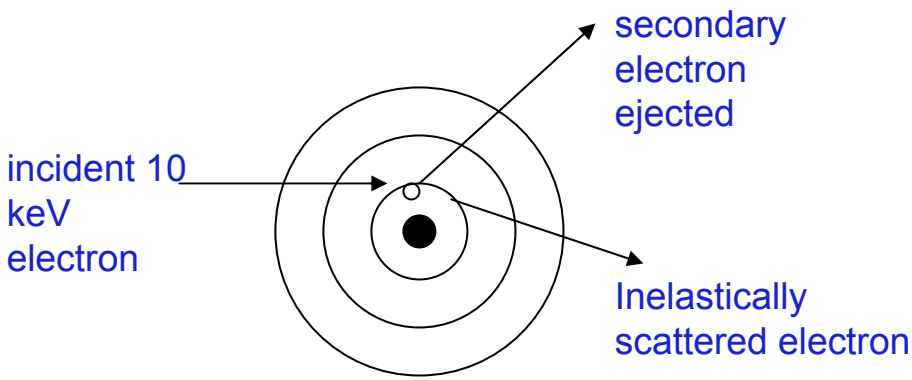
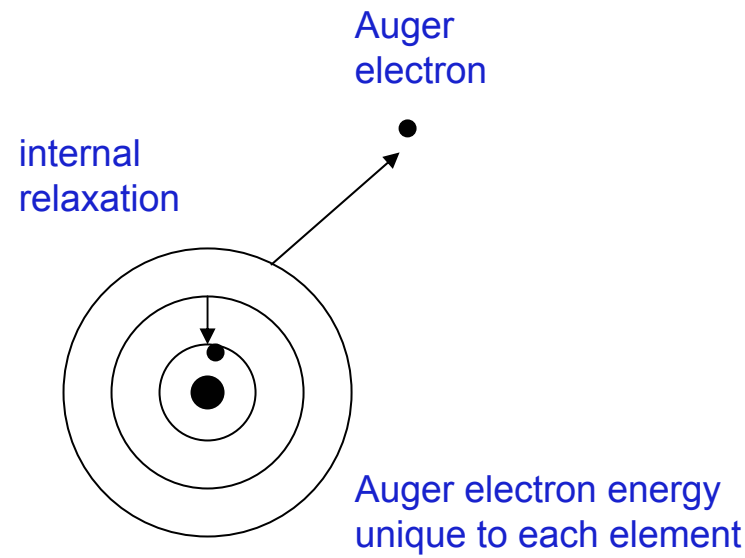
Surface Analysis*

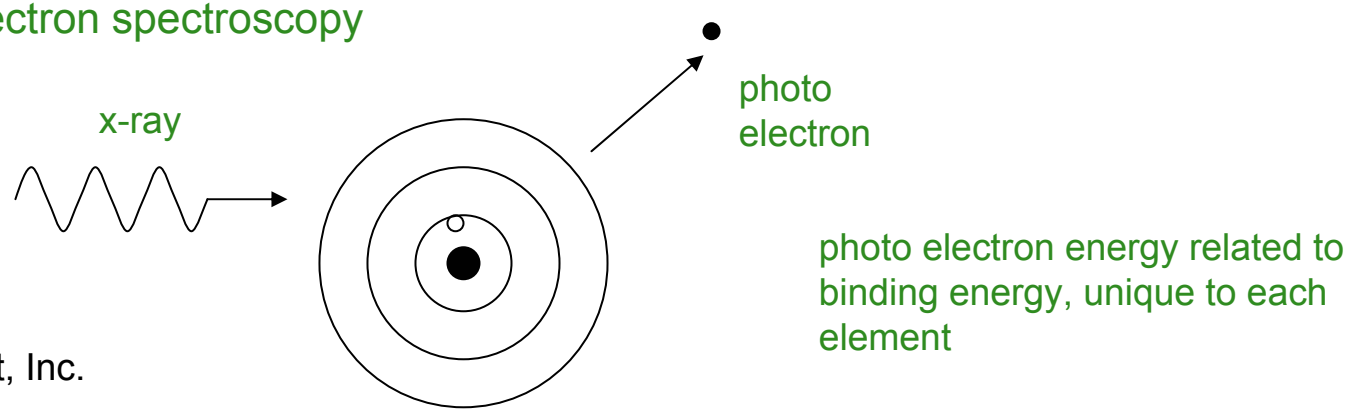
Techniques used so far:
SEM: scanning electron microscopy
AES: Auger electron spectroscopy





3 keV Ar+ beam sputters surface to give depth profile.

XPS: X-ray photoelectron spectroscopy



*Analysis by Evans East, Inc.

Note: AES, XPS does not detect hydrogen or helium.

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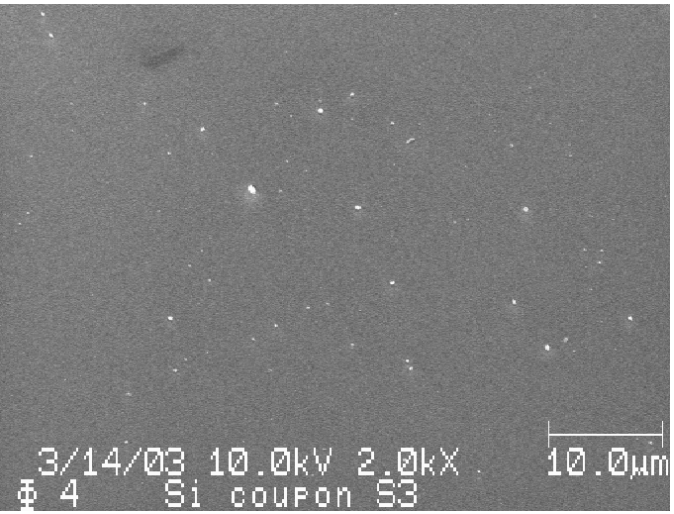
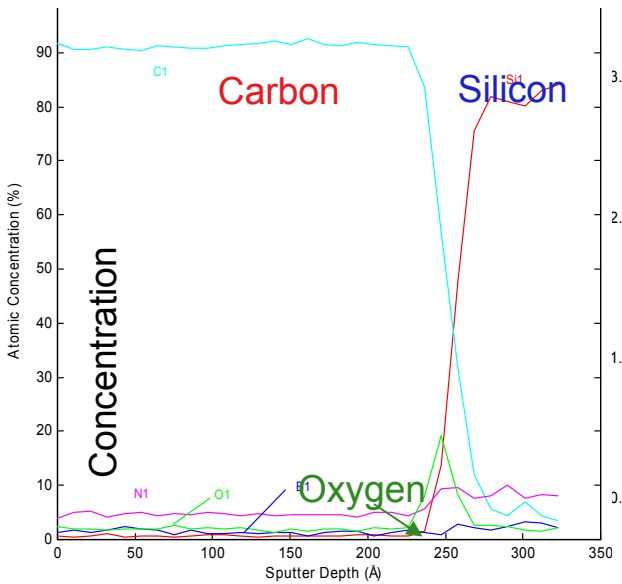
Surface Analysis of Si witness coupon exposed to plasmas

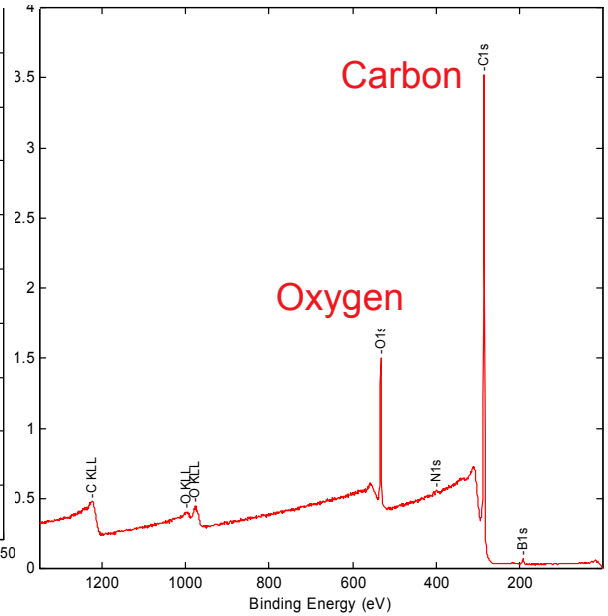
SEM



AES



XPS



Depth into sample 0 - 350 Å

Binding Energy (eV)

AES shows depth of layer to be 250 Å (assuming sputter rate same as diamond-like carbon.)

XPS shows the surface of the samples contained mostly carbon (as C-(C,H) and C-O) and oxygen. Si coupon S3 also contained boron (as B^{3+} and possibly B-N) and nitrogen (as C-N and NO_x).

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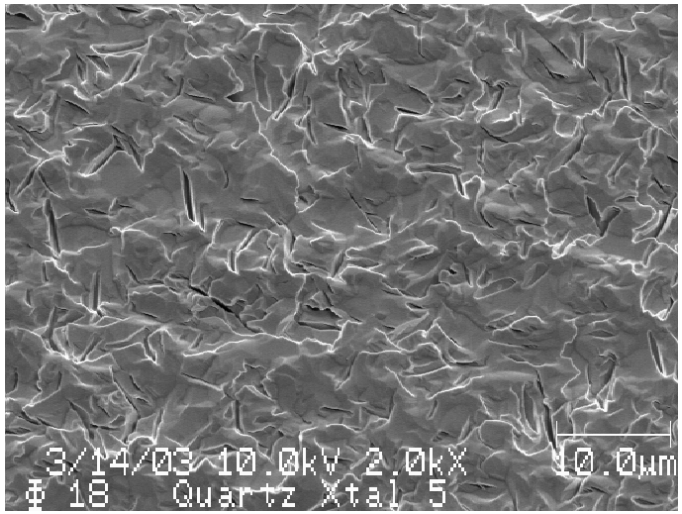
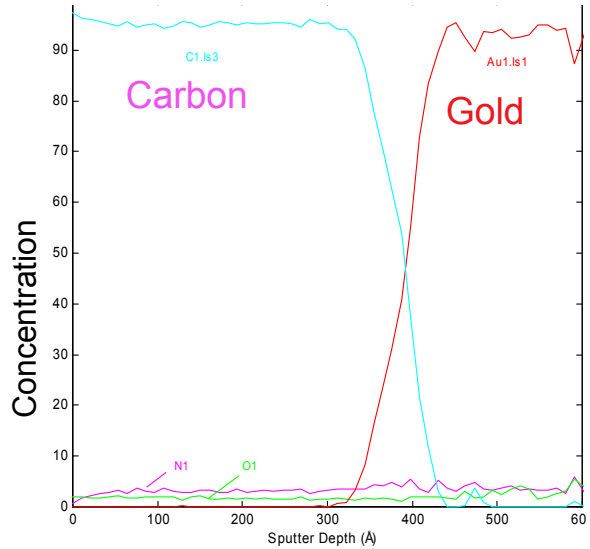
Surface Analysis of plasma facing quartz crystal

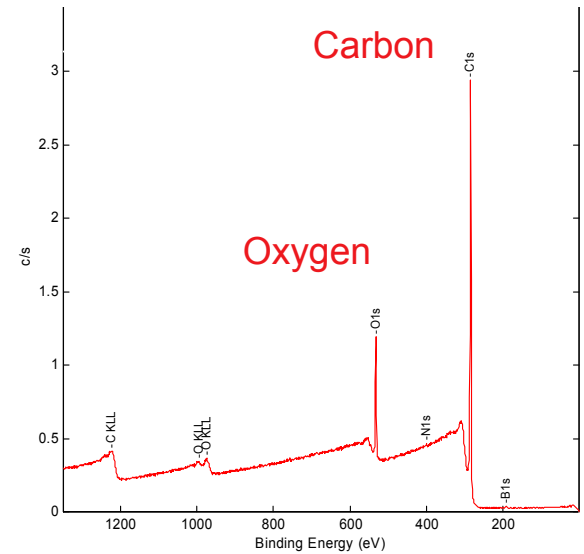
SEM



AES



XPS



Depth into sample 0 - 600 Å

Binding Energy (eV)

AES shows depth of layer to be 400 Å (assuming sputter rate same as diamond-like carbon.)

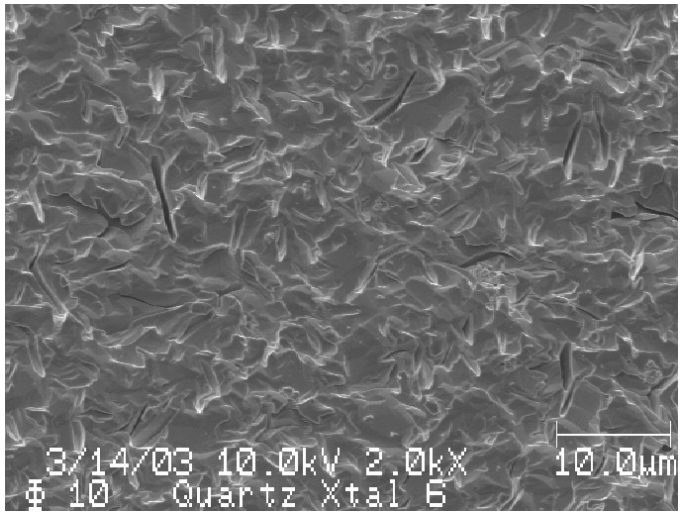
Cumulative thickness indicated by deposition monitor 600 Å (assuming density ρ=2.37)

- RBS planned to calibrate thickness (Wampler SNL).

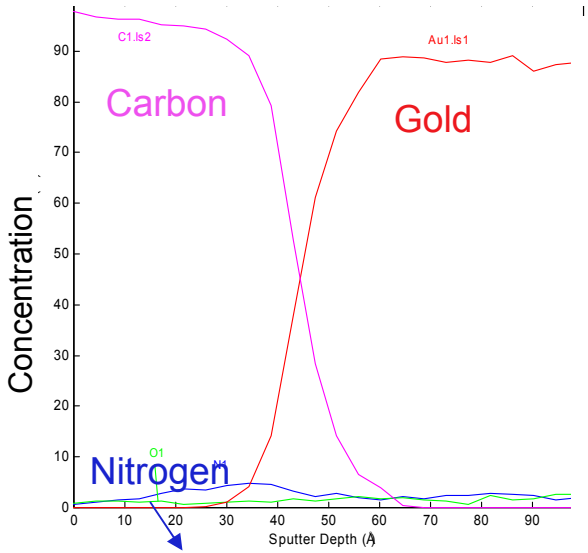
XPS shows the surface of the samples contained mostly carbon (as C-(C,H) and C-O) and oxygen. Quartz Xtal 5 (plasma facing) also contained boron (as B^{3+} and possibly B-N) and nitrogen (as C-N and NO_x).

Surface Analysis of backward facing quartz crystal

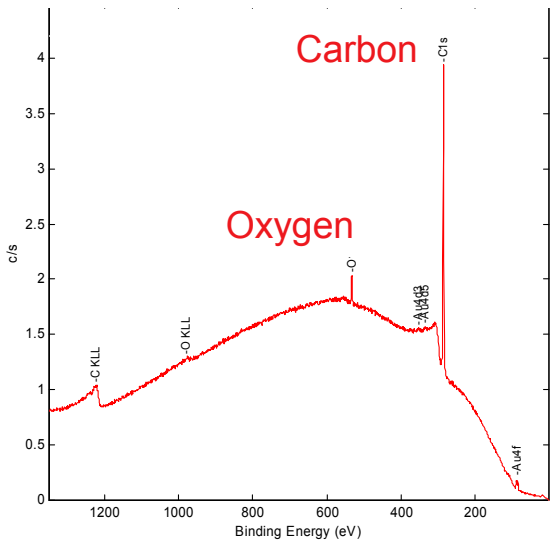
SEM



AES



XPS



Depth into sample 0 - 100 Å

Binding Energy (eV)

AES shows depth of layer to be 45 Å (assuming sputter rate same as diamond-like carbon.)

The surface of all three samples contained mostly carbon (as C-(C,H) and C-O) and oxygen.

Sample Quartz Xtal 6 (backward facing) also contained nitrogen (as C-N and R_4-N^+) and gold (as Au°).

The general lineshape indicates the presence of an overlayer on a gold substrate.

The lineshape to the left of the carbon peak indicates that carbon is in this overlayer.

Difference between plasma facing and backward facing deposition related to sticking coefficients.

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Elemental analysis

Concentration of Elements Detected (in atom%, normalized to 100% excl. H)

Sample	Filename	O	N	C	B	Au
Si coupon S3	030433101	12.3	0.6	85.1	2.0	nd
Quartz Xtal 5	030433111	11.3	0.3	87.4	0.9	nd
Quartz Xtal 6	030433121	4.5	0.4	94.8	nd	0.3

Note: AES, XPS does not detect hydrogen or helium (RBS planned at Sandia).
nd = not detectable (< 0.1 atom %)

- The surface of all three samples contained mostly carbon (as C-(C,H) and C-O) and oxygen.
- Samples Si coupon S3 and Quartz Xtal 5 (plasma facing) also contained boron (as B^{3+} and possibly B-N) and nitrogen (as C-N and NO_x).
- Sample Quartz Xtal 6 (facing away) also contained nitrogen (as C-N and R_4-N^+) and gold (as Au°).
- Ratio between plasma facing and facing away from plasma layer thickness is 9 : 1

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Ion Beam analysis*

Sample:	D (IBA) atoms/cm^2	C (IBA) atoms/ cm^2	D/C ratio	D+C mass (IBA) µg/cm2	Total mass (micro-balance) µg/cm2	Thickness of D+C (IBA) (nm)	Thickness (micro-balance) (nm)
Si #3 (plasmas)	0.58 x10^{17}	3.8 x10^{17}	0.153	7.55	*	*31.8*	*
Quartz #5 (front facing, plasmas)	0.59 x10^{17}	5.7 x10^{17}	0.104	11.4	14.8	*47.9*	*59.8*
Quartz #6 (back facing, plasmas)	0.044 x10^{17}	0.60 x10^{17}	0.073	1.21	*	*5.0*	*

- Ion Beam detects deuterium and carbon but not hydrogen or oxygen. Combining XPS measurements of % oxygen and % carbon with absolute carbon density from IBA:
- Mass of D+C+O = 13.5 µg/cm2
- Total mass measured by quartz microbalance = 14.8 µg/cm2
- Excellent agreement and within 10% experimental uncertainty.

*W. Wampler, SNL

Summary :

- Measurements reveal discharge-by-discharge growth of deposition and the erosion of coating by disruptions.
- Coating rate for NSTX typically 1-2 Å per ~ 0.5 sec discharge but can range over 0.5-13Å per discharge
  - implications for wall conditions, boronization frequency and window / mirror coating (ITER diagnostic issue).
- Specific, unique correlation to other diagnostics not yet found.
- Surface analysis (SEM, AES, XPS...) of samples show mostly carbon, some oxygen with traces of metals.
- Good agreement between independent measurements of deposited mass by ion beam analysis and quartz microbalance.
- Long term - put another deposition monitor inside vessel closer to plasma.