Tritium retention in plasma-facing components

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Outline of the talk

- Motivation and challenge

- Methodology and research approach

- Progress and future plans
ITER first wall/divertor

- Plasma-facing component (PFC)
  - Beryllium at first wall to minimize $Z_{\text{eff}}$
  - Tungsten at divertor for high $q_{\text{heat}} > 10$ MW/m$^2$

$P_{\text{bremsstrahlung}} \sim (Z_{\text{eff}})^2$

Beryllium first wall
(low Z $\rightarrow$ low radiation loss)

Tungsten divertor
(high melting point, low sputtering yield and high thermal conductivity)
Material challenges in plasma-facing components

- **Materials under extreme condition:**
  - Intense plasma particle/heat flux
  - 14 MeV neutron flux

- **Safety concern for tritium inventory:**
  - 4 kg: ITER site limit (~ 1,500,000 TBq)*
    - 2~3 kg: in tritium plant?
    - 1 kg: in hot cell
    - 1 kg: in vacuum vessel including piping system
      - (120 -150) g in cryo-pumps
      - (850 – 880) g in plasma-facing components
  - ITER: Be co-deposit is predominating factor.
  - DEMO: Neutron-irradiated W will be predominant factor.

* Fushikuma Daiichi released ~ 540,000 TBq in 2011
Outline of the talk

• Motivation and challenges

• Methodology and research approach

• Progress and future plans
Research approach for tritium retention study

• Challenges:
  – Unavailability of high-flux 14 MeV neutron source.
  – Unavailability of simultaneous neutron and plasma irradiation capability

• Research approach:
  – Use of available fission reactor (e.g. High Flux Isotope Reactor/HFIR).
    • Relatively high-flux fast neutron (>0.1 MeV) available to simulate neutron damage
    • High-flux thermal neutron accelerates solid transmutation and increases activation
  – Use of thermal neutron-shielding (e.g. cadmium or gadolinium)
    • To minimize thermal neutron and simulate fast fusion neutron spectrum.
  – Use of linear plasma device to study sequential neutron-plasma irradiation.
    • TPE can handle tritium neutron-irradiated material.

  – Neutron-irradiation with thermal neutron-shielding at RB* position in HFIR, ORNL
    • 500, 800 and 1100 °C irradiation temperature up to 1.0 dpa
  – Deuterium plasma exposure in TPE, INL
    • 500, 800 and 1100 °C plasma exposure temperature
    • > $10^{22}$ m$^{-2}$s$^{-1}$ D ion flux up to $10^{27}$ m$^{-2}$ D ion fluence
  – Post irradiation examination (PIE) at INL, SNL-NM, ORNL.
    • Nuclear reaction analysis (NRA) for D depth profiling
    • Thermal desorption spectroscopy for total D retention
    • XPS, SAM, PAS, GD-OES for surface/bulk characterization.

M. Shimada (INL) | 38th TFGM | May 9, 2017 | PNNL
Neutron energy spectrum with Gd thermal neutron shielding

Joyo HP

From 2017 PHENIX SCM

M. Shimada (INL) | 38th TFGM | May 9, 2017 | PNNL
Capsule design, specimen matrix and irradiation schedule

• **RB19J capsule design**
  – Cost sharing with JAEA-ORNL F82H irradiation program (300 °C zone)
  – 500, 800, 1100 °C temperature zone for PHENIX tungsten irradiation

• **Specimen matrix for PHENIX Task 3: “Tritium behavior in irradiated tungsten”**
  – D6TQ : 6 mm OD, 0.25mm thick discs  : ~ x10 each temp. zone
  – D6TH : 6 mm OD, 0.5 mm thick discs (standard)  : ~ x80 each temp. zone
  – D6T1 : 6 mm OD, 1.0 mm thick discs  : ~ x10 each temp. zone
  – D10TQ : 10 mm OD, 0.25 mm thick discs  : ~ x15 each temp. zone
  – D10TH : 10 mm OD, 0.5 mm thick discs  : ~ x10 each temp. zone

• **RB19J irradiation completed**
  – Started: Cycle 466 (June, 2016)
  – Ended: Cycle 469 (December, 2016)
TPE - Tritium Plasma Experiment

- TPE is a unique linear plasma device in four elements:
  - Tritium plasma (< 500 Ci per discharge),
  - Divertor-relevant high-flux plasma (>10^{22} \text{ m}^{-2}\text{s}^{-1})
  - Moderately radioactive (< 1 mSr/hr @ 30 cm) materials handling
  - Beryllium handling

- Utilizes two containments other than its SS vacuum vessel
  - Ventilated enclosure (as a high contamination area/HCA boundary)
  - Ventilated PermaCon room (as a contamination area/CA boundary for T)

- TPE is the only high-flux linear plasma device to investigate tritium retention using tritium and neutron-irradiated material.
Outline of the talk

• Motivation and challenges

• Methodology and research approach

• Progress and future plans
Tungsten specimens irradiated under TITAN and PHENIX

- **Material and heat treatment:**
  - SRW: Stress-relieved (1 h @ 900 °C in H₂ + 0.5 h @ 900 °C in vacuum) 99.99 wt.% (A.L.M.T., Japan)
  - RxW: Recrystallized (1 h @ 1500 °C + 0.5 h @ 1300 °C in vacuum) 99.99 wt.% (A.L.M.T., Japan)
  - SCW: Single-crystal (100) 99.99 wt.% (Goodfellow, USA)

- **Specimen size:**
  - Disc: 6 mm dia. 0.2 mm thick (D6TQ) and 6 mm dia. 0.5 mm thick (D6TH)
  - Square: 4 x 4 x 0.5 mm³ square (TS) from 16 x 4 x 0.5 mm³ tensile bar specimen

<table>
<thead>
<tr>
<th>project</th>
<th>capsule ID</th>
<th>Damage [dpa] ²⁴</th>
<th>Irradiation temp.[°C ]</th>
<th>sample size</th>
<th># of sample Irradiated for Task 2-1</th>
<th>shipped to INL?</th>
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<th>PIE completed?</th>
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<td>0.025</td>
<td>80</td>
<td>D6TQ</td>
<td>6 (SRW)</td>
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<td>4 (SRW) *¹</td>
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<td>D6TH</td>
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<td>D6TH</td>
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Note: *¹ 2 out of 6 were broken during shipment, *² 10 out of 12 were broken in reactor, *³ all broken or missing
*⁴ 1 dpa ~ 5.0E25 n/m² (E>0.1 MeV)
Tungsten specimens exposed to TPE plasma for PMI to date

- Post-Irradiation Examination (PIE) sequence:
  - TPE/TDS:
    - TDS (10°C/min to 900°C) performed within 24 hours after the TPE exposure (0.5x10^{26} m^{-2})
  - TPE/NRA/TPE/NRA/TDS:
    - TDS (10°C/min to 900°C) performed after the 1st TPE exposure (0.5x10^{26} m^{-2}), 1st NRA, 2nd TPE exposure (0.5x10^{26} m^{-2}), and 2nd NRA. The time interval between 1st TPE exposure and TDS were 400-600 days.

<table>
<thead>
<tr>
<th>capsule ID</th>
<th>Damage [dpa]</th>
<th>HFIR irradiation temp. [°C]</th>
<th>sample size</th>
<th>sample ID</th>
<th>D/T/He plasma</th>
<th>TPE exposure temp. [°C]</th>
<th>Ion flux [10^{22} m^{-2} s^{-1}]</th>
<th>Ion fluence [10^{26} m^{-2} s^{-1}]</th>
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<td>0.4</td>
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<td>Y103</td>
<td>D</td>
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<td>D</td>
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<td>Y107</td>
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<td>W26B</td>
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<td>W53B</td>
<td>D</td>
<td>400</td>
<td>0.5</td>
<td>0.5</td>
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<td>TB-650-2</td>
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<td>690</td>
<td>TS</td>
<td>W55A</td>
<td>D</td>
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<td>0.8</td>
<td>0.5</td>
<td>TPE/TDS</td>
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<td>W55B</td>
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<td>0.5</td>
<td>TPE/NRA</td>
<td>[3]</td>
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</table>

PIE results from TITAN SRW specimens

- Deep migration of D at elevated temp. [1]
  - Specimens:
    - Capsule: T9A1 and T9A2
    - Dose: 0.025 and 0.3 dpa
    - HFIR irradiation temp.: 80 °C at HFIR
    - TPE exposed temp.: 100, 200, and 500°C
  - NRA results showed that the max. D concentration up to 0.8 at% D/W in 0.3 dpa
  - The large discrepancy between the TDS and NRA, indicating that D was trapped in bulk
    - > 50 µm depth for 0.025 dpa exposed at 500°C
    - > 35 µm depth for 0.3 dpa exposed at 500°C

- Defect annealing of n-irradiated SRW [2]
  - Specimens were re-exposed to TPE after TDS
    - TPE condition: 0.5x10^{26} m^{-2} D plasma at 200/500 °C
    - TDS condition: 10°C/min to 900°C
  - D retention decreases approximately 70% for sample exposed at 500 °C after each annealing,
  - HFIR induced radiation damages were NOT annealed out completely even after the 3rd annealing at 900 °C for 0.5 h (1.5h total)
  - D desorption peak shifts from 800 °C to 600 °C, indicating the change in de-trapping energy and microstructure.

PIE results from PHENIX SCW specimens

- D retention in n-irradiated SCW at DEMO-relevant elevated temp. (1\textsuperscript{st} campaign)
  - Specimens:
    - Capsule: TB-300-2, TB-500-2, and TB-650-3
    - Dose: 0.1 dpa
    - HFIR irradiation temp.: 360, 690, and 760°C
    - TPE exposed temp.: 400, 600, and 700°C
  - TDS (10°C/min to 900°C) performed within 24 hours after the TPE exposure (0.5x10\textsuperscript{26} m\textsuperscript{-2})
  - 1\textsuperscript{st} campaign (TPE/TDS study) showed the competing dynamics of trapping and D diffusion in the bulk in m-irradiated SCW.
    - Diffusion became the dominant transport mechanism, with trapping, or loss of trapping sites from material annealing, playing a reduced role in the retention process.
  - 2\textsuperscript{nd} campaign is to measure D depth profile on similar specimens by NRA at SNL-MN (next slide)

Figure: TDS spectra (left) and total retention (right) for neutron-irradiated single crystal W samples (100 eV D with 0.5x10\textsuperscript{26} D m\textsuperscript{-2})

Depth profile (linear) from HFIR irradiated SCW

- Nuclear reaction analysis (NRA) by Bill Wampler (SNL-MN)

- W53B: \((T_{HFIR}, T_{TPE}) = (360, 400)\)
  - 0.3 \% D/W near surface < 2 µm and 0.15 \% D/W near surface > 2 µm

- W55B: \((T_{HFIR}, T_{TPE}) = (690, 600)\)
  - D desorption near surface < 2 µm and 0.015 \% D/W near surface > 2µm

- W26B: \((T_{HFIR}, T_{TPE}) = (760, 700)\)
  - D desorption near surface < 2 µm and 0.005 \% D/W near surface > 2µm
Depth profile (log) from HFIR irradiated SCW

• Nuclear reaction analysis (NRA) by Bill Wampler (SNL-MN)

• W53B: \((T_{HFIR}, T_{TPE})=(360, 400)\)
  – 0.3 % D/W near surface < 2 µm and 0.15 % D/W near surface > 2 µm

• W55B: \((T_{HFIR}, T_{TPE})=(690, 600)\)
  – D desorption near surface < 2 µm and 0.015 % D/W near surface > 2µm

• W26B: \((T_{HFIR}, T_{TPE})=(760, 700)\)
  – D desorption near surface < 2 µm and 0.005 % D/W near surface > 2µm
# Tungsten specimens irradiated under PHENIX

- **Material and heat treatment:**
  - SRW: Stress-relieved (1 h@900 °C in H₂ + 0.5 h@900 °C in vacuum) 99.99 wt.% (A.L.M.T., Japan)
  - SCW: Single-crystal [110] (0.5 h@900 °C in vacuum) 99.99 wt.%
  - W-3Re: 3%Re/W (prepared by Tohoku U.) and W-5Re: 5%Re/W (prepared by U. of Toyama)
  - UFG-W: Ultra fine grained (1.1% TiC)

- **Specimen size:**
  - 6 mm dia. disc: 0.25 mm thick (D6TQ), 0.5 mm thick (D6TH), 1.0 mm thick (D6T1)
  - 10 mm dia. disc: 0.25 mm thick (D10TQ), 0.5 mm thick (D10TH)

<table>
<thead>
<tr>
<th>material</th>
<th>sample size</th>
<th># of sample for PHENIX Task 3</th>
<th>PIE plan</th>
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<td></td>
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<td>500 °C</td>
<td>800 °C</td>
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<tr>
<td>SRW</td>
<td>D6TQ</td>
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Note: All specimens were irradiated at HFIR’s RB* with Gd thermal neutron shielding for 4 cycles.
Neutron-irradiated W specimen exposed to TPE

- Displacement damage vs. irradiation temperature
  - TITAN ('07-'12) focused on low temperature (<500°C) high dose (<< 1 dpa) in pure W
  - PHENIX ('13-'18) aims at high temperature (>500°C) high dose (> 1 dpa) in W and W alloy
PIE plans in PHENIX Task 3

1) PIE at ORNL (sharing data from Task 2)
   - Positron Annihilation Spectroscopy coincidence lifetime (PAS-CL) for defect characterization
   - Transmission Electron Spectroscopy (TEM) for defect characterization

2) PIE at STAR, INL
   - Plasma-driven D/T/He implantation with TPE for D/T/He implantation
   - Gas-driven T permeation with TGAP for T permeation
   - Deuterium gas exposure with SGAS for D absorption
   - PAS Doppler broadening measurement (PAS-DB) for defect characterization
   - Thermal desorption spectroscopy (TDS) for total D/T/He retention measure’t
   - X-ray photoelectron spectroscopy (XPS) for surface chemical state
   - Scanning Auger Microscopy (SAM) for surface elemental composition
   - Glow discharge optical emission spectroscopy (GD-OES) for depth profiling

3) Nuclear reaction analysis (NRA) at SNL-MN (Bill Wampler)

4) Supporting PIE at JA Oarai Center
   - PAS-DB, PAS-CL, Compact Divertor Plasma Simulator (CDPS), TDS etc.
PIE plans in TPE under PHENIX Task 3

**TPE experimental condition:**
- $n_{e,\text{max}}$: $0.5 \times 10^{19}$ m$^{-3}$
- $\Gamma_{\text{max}}$: $0.5 \times 10^{23}$ m$^{-2}$ s$^{-1}$
- $q_{\text{heat}}$: $< 1.0$ MW m$^{-2}$
- $T_{\text{sample}}$: $< 700$ °C
- $E_{\text{ion}}$: up to 600 eV
- Plasma species: He, D, T

$\Rightarrow > 1.0 \times 10^{19}$ m$^{-3}$ (plan $^*1$)
$\Rightarrow > 1.0 \times 10^{23}$ m$^{-2}$ s$^{-1}$ (plan $^*1$)
$\Rightarrow > 1.0$ MW m$^{-2}$ (plan $^*1$)
$\Rightarrow$ up to 1200 °C (plan $^*2$)

**PIE plans $^*3$**
- D6TH specimens (Task 3 standard specimen)
  - Temperature (500-1100 °C), ion flux ($1 \times 10^{22}$-$1 \times 10^{23}$ m$^{-2}$s$^{-1}$), ion fluence ($1 \times 10^{26}$-$1 \times 10^{28}$ m$^{-2}$) dependence on plasma-driven D/T/He retention
- D6TQ, D6TH, and D6T1 specimens
  - Thickness dependence on plasma-driven D/T/He retention
  - Thickness dependence on gas-driven D retention
- D10TQ and D10TH specimens
  - Thickness dependence in gas-driven T permeation

**NOTE:**

$^*1$) New source holder was designed based on UCSD PISCES-A, and is being fabricated at INL.
$^*2$) New sample holder is being designed based on UCSD PISCES-A.
$^*3$) Actual test matrix will be determined based on the number of intact HFIR irradiated samples after its shipment to INL.
Summary

- Tritium retention in plasma facing components (PFCs) is one of the key safety issues in magnetic fusion energy.
- Tritium retention is neutron-irradiated tungsten and tungsten alloys will be predominant factor in DEMO and future fusion reactor.
- Under US-Japan PHENIX program, the combination of thermal neutron-shielded irradiation at HFIR, ORNL and high-flux plasma exposure at TPE, INL is used to investigate tritium retention in neutron-irradiated tungsten.
- RB19J irradiation successfully ended in December 2016 with 4 cycles
  - Irradiation temperature: 500, 800, 1100 °C
  - Radiation damage: up to 1.0 dpa
- RB19J neutron-irradiated tungsten samples will be shipped to INL in FY18
- Plan to perform PIE of hundreds of neutron-irradiated samples by March 2019.
- PIE from RB19J neutron-irradiated tungsten samples will provide valuable database for in-vessel tritium source (i.e. tritium retention) assessment in future DEMO licensing.
Future Directions in FSE Research

- Strategy based on FES guidance and 2013 FES Peer Review Comments
  - Materials Research: Fusion materials, including tungsten irradiated, will be studied at high temperature and heat flux to measure tritium retention and permeation. Dust explosion measurements for fusion materials will continue in support of licensing and computer code development activities. New material diagnostics.

- Code Development: for the near term, a newer version of MELCOR for ITER will be completed that includes tritium transport and dust explosion models. Long-term: Multi-dimensional safety code capabilities need to be developed that take advantage of parallel computing (example RELAP 7).

- Risk and Licensing: FSP's evolving failure rate database will be expanded to include maintenance data from existing tokamaks. Risk-informed safety analysis methods (example RISMC Toolkit) will be studied for application to an FNSF. Continue ASME codes and standards and licensing framework development.

- Collaborations: Participation in existing collaborations to leverage other institution's capabilities and reduce duplication of effort. STAR will move towards being more effective FES User Facility.
Supporting slides
Material challenges in nuclear fusion (2/2)

PMI

\[ > 10^{23} \text{ m}^{-2}\text{s}^{-1}, > 10 \text{ MWm}^{-2} \]

- Erosion & re-deposition
- High heat flux (steady state & transient)
- Surface morphology (tungsten fuzz, \(^{1}H/^{4}He\) bubble, etc...

neutron

\( \gg 1 \text{ dpa} \)

- Radiation damage
- Solid transmutation (e.g. Re, Os in W)
- Gas production (\(^{1}H, ^{4}He\))
- Thermal conductivity degradation
- Embrittlement etc...

tritium

D/T

- Tritium behavior (permeation & retention)
- Tritium decay \(^{3}He\) effect
- Beta-induced effect etc...

Burning Plasma PMI

Ref: A. Hasegawa JNM 2016

Ref: M. Shimada TPE

### Irradiation temperature analysis

<table>
<thead>
<tr>
<th>Sub-capsule</th>
<th>$R_{\text{holder}}$ (mm)</th>
<th>Holder Material</th>
<th>Spacer Material</th>
<th>$T_{\text{avg}}$ (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>250 Top</td>
<td>16.17</td>
<td>Aluminum</td>
<td>Aluminum</td>
<td>290</td>
</tr>
<tr>
<td>250 Mid</td>
<td>16.17</td>
<td>Aluminum</td>
<td>Aluminum</td>
<td>238</td>
</tr>
<tr>
<td>250 Btm</td>
<td>16.17</td>
<td>Aluminum</td>
<td>Aluminum</td>
<td>220</td>
</tr>
<tr>
<td>500</td>
<td>16.15</td>
<td>Graphite</td>
<td>Graphite</td>
<td>482</td>
</tr>
<tr>
<td>800</td>
<td>16.15</td>
<td>Graphite</td>
<td>Graphite</td>
<td>723</td>
</tr>
<tr>
<td>1200</td>
<td>16.00</td>
<td>Graphite</td>
<td>Graphite</td>
<td>1041</td>
</tr>
</tbody>
</table>

*Note: ANSYS R16.2 results are shown in the diagram.*
Upgrade in plasma diagnostics

• Optical spectrometer was installed in FY16
  – Specification:
    • 0.75m focus length Czerny-Turner spectrograph (Andor Shamrock 750)
    • 1024x255 CCD camera (Andor iDus420BU)
  – Capability:
    • Provide plasma impurity measurement
    • He ion concentration measurement
    • Neutral gas temperature measurement

• First mixed-plasma (He + D) was achieved
  – In 30 year TPE history
  – He I emission lines was observed in newly setup optical spectrometer system
  – Subsequently desorbing helium atom was observed at elevated temperature during thermal desorption
  – Calibration of CCD camera is required for quantitative measurement of He ion conc.

• Next step is to produce tungsten fuzz.
New surface diagnostics at STAR

- **Glow discharge optical emission spectroscopy**
  - Quantitative elemental depth profile analysis
  - Nanometer resolution
  - Can analyze 10s or even 100s µm sample depth
  - *Installation completed in FY16*

- **X-ray photoelectron spectroscopy**
  - Surface sensitive chemical analysis
  - Excellent for quantification
  - Particularly useful for surface effects related to permeation
  - *Arriving FY17*

- **Scanning Auger electron spectroscopy**
  - AES provides elemental characterization
  - Scanning mode allows for microscopy
  - *Arriving FY17*

**NOTE:** all three diagnostics will be capable of handling low activation and tritium-exposed materials.
New additions to STAR

Coincidence positron annihilation spectroscopy (CDB-PAS)

Glow discharge optical emission spectrometry (GD-OES)

Scanning auger microprobe (SAM/SEM)

X-ray photoelectron spectroscopy (XPS)
Glow discharge optical emission spectroscopy
(installation completed in FY16)

**Technique**
- Elemental characterization with depth profiling.
- Analyzed 10s of elements simultaneously.
- Nano-meter resolution.
- Sputtering rates of nm/sec.

**Specifications**
- Capable of differentiating H and D.
- Realtime depth measurements.
- Monochromator can be used to look at unknown element.

**GD-OES** will measured the D depth profiling from bulk (>> 10µm) neutron-irradiated tungsten exposed to plasma

One of very few surface analysis techniques capable of measuring hydrogen.
Glow discharge optical emission spectroscopy

• The glow discharge burns a crater into a sample.
  – Crater shown is 4 mm diameter.
  – Crater size can vary from 1-10 mm with interchangeable anodes.

• Fast sputtering rate (over 2 µm/min).
  – Material dependent.
  – Analyze 100s µm in minutes.
X-ray photoelectron microscopy (XPS) and Scanning Auger electron spectroscopy/microscopy (SAM)

• XPS
  – Technique
    • Excellent chemical sensitivity.
    • Expansive libraries.
    • Capable of detecting elements, Li and larger.
  – Specifications
    • X-ray monochromator for high resolution XPS scans.
    • Multiple x-ray sources.
    • Sputtering ion gun for depth profiling.

• SAM
  – Technique
    • Excellent elemental sensitivity.
    • Limited quantification.
    • Rastering electron beam + Secondary electron detector = SEM
  – Specifications
    • LaB6 filament.
    • Sputtering ion gun for depth profiling.

• XPS and SAM will be installed in FY17