Low energy, light ion sputtering experiments of liquid Sn using IIAX

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Outline

• Overview of IIAX system
  – Ion beam
  – Target assembly
  – Sputter measurements
• Data
  – Analysis
  – Example data
• Status and future work plan
Motivation:
Concept of a flowing liquid Sn divertor surface

• ALPS program investigating flowing liquid metals for advanced divertor surfaces [1]
  – Power density capabilities
  – Component lifetime
    (no net erosion = no replacement needed)
  – Energy conversion efficiency

• While there has been a recent return of primary focus to solid metal PFC studies (W and Be in particular) in support of ITER, having enough data to develop a reliable model is achievable in the short term to maximize benefits from previous research

Advantage of using liquid Sn: Vapor pressure!

- Sn has an evaporative flux many *orders of magnitude* lower than Li
- Friendly & abundant (cheap!)
- Evaporation curves based on theory by [1] and fits from [2] and [3].

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Liquid Sn divertor study…


• Limitations on use of Sn based on this study:
  • Self-sputtering process; too much self-sputtering leads to runaway sputtering and disruption
  • Uncertainty due to lack of temperature-dependent sputtering yields and model

• Sn self sputtering analysis
  • Mean Sn ion energy at 273 eV (High recycling regime & mean charge state of 2)
  • Mean impact angle of Sn+ at 22° (from normal) due to high Z
  • 42% of the total Sn sputtering is due to self-sputtering
  • 99.91% of Sn is redeposited (only 0.09% leaves near-surface area)


Ion-surface InterAction eXperiment (IIAX)
Colutron Ion Source

- Typically, a DC gas discharge supplies the ions
- Isolated source volume allows differential pumping (~10⁻¹ Torr in source ↔ 10⁻⁵ Torr in ion gun chamber)
- Produces a wide variety of beam species
  - Gaseous source
  - Solid source (Li⁺, Sn⁺, etc)
  - Molecular ions
  - Multiple charge states possible
- Ions extracted from region in front of ½ mm pinhole in anode (left-most end in this photo)
Sn ion source

- Same as gaseous ion source except a solid charge holder is inserted within the windings of the coil to vaporize material
- An Ar discharge assists in producing Sn ions for extraction
- A Wien filter is used down the beam line to filter out Ar ion components of the beam

Ar gas discharge between filament (cathode) and anode provides electrons to impact ionize Sn atoms

Extraction / acceleration region followed by ion filters & optics

Construction based on conversations with Dr. Lars Wählin from Colutron (Ion gun mfr.)

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Following bringing ions up to energy and the primary Einsel lens are vertical deflection plates and a velocity filter.

Wien filter:

- Velocity spectrum of 1000 eV ion beam extracted from an Ar-discharge supported Sn source.
- If 60.5V corresponds to 40amu (Ar\(^+\)), then 35.1V corresponds to 118.7amu (Sn\(^+\)).

Mathematical expression:

\[ \frac{E - field}{B - field} = v = \sqrt{\frac{2E_{ion}}{m_{ion}}} \]
Ion beam system: Neutral filter

- After velocity filter, beam exits ion gun chamber and enters the main (sample) chamber
- Horizontal deflection plates make a $3^\circ$ bend in beam to filter neutrals
- Neutral filtering is performed after entering the main chamber to minimize neutral component without extending beam path length with the addition of another chamber (sacrificing potential for additional differential pumping)
Sample chamber

- Decelerator also acts as a second Einsel lens
- Cylindrical chamber: 24” (~0.6 m) ID
- Bend exaggerated for clarity
- Not to scale
Prior target temperature was limited

• Two factors…
  – Poor thermal considerations in target/heater holder design limited target to ~550°C
  – Above ~420°C, the QCM units would fail due to being close to the hot target without active cooling

• Recent hardware upgrades to allow high temperature measurement
  – Repaired QCM head for electrically-isolated water cooling
  – Installation of new target holder
  – Goal: Samples at 1000°C (Heater rated for 1200°C)
Modification to QCM head: Electrically-isolated water cooling

• Benefits:
  – Greatly improved crystal stability (better signal to noise ratio) at all temperatures
  – Able to exceed 870°C without crystal failure with no apparent limit as of yet (heater power limit should be ~1100°C)
  – Maintaining the same crystal temperature for all target temperatures
  – Use of a ceramic break and deionized water maintains electrical isolation

• Drawbacks:
  – Greatly reduced mobility of QCM head due to stiff “flexible” water lines
  – Marginally degraded base pressure due to use of Swagelok fittings (low 8’s versus mid 9’s on a good day)
Heater & liquid sample holder redesign

- Thermal considerations
  - Minimized thermal contact between heater/target components and mounting hardware
  - Radiation shield around circumference (SS) and behind (Mo) heater to minimize radiative losses

![Diagram of heater and sample holder with labels for each component: Mounting assembly & circumferential radiation shield, Mo radiation shield, Macor (or BN) isolator, Mo retention shield, Mo retention ring, Sample, Standard Heatwave UHV Heater. Note: Mo/Re sample clips not shown.]
New sample holder construction

• Currently, only one assembly ‘hard’ mounted
• Goal: Several interchangeable sample assemblies
• Quick assembly replacement (through 6” CF port)
• Two samples mounted with others ready to minimize down-time
• Need:
  – Design & construction time
  – Feedthrough
  – UHV-grade plugs
New sample holder in place

- K-type thermocouple
- Aperture to (bent) Faraday cup for beam diagnosis
- Mo/Re sample clips hold sample assembly together
• Presently, we’re limited by the heater power circuit to ~870°C but reaching 1100°C is achievable assuming $T^4$ scaling (has shown to be pessimistic so far so ~1200°C may be achievable)

• Some of this sample spilled out, but was otherwise well-behaved and showed a beautifully-reflective surface

• We may be looking at Sn contaminated with Mo due to alloy formation: Studies underway may lead to replacement of Mo parts with Ta if necessary
Sample area geometry:
Sample & primary QCM

- Ceramic components
- Heater stem for support
- Sample Heater
- Mo components
- Sample
- Quartz Crystal
- QCM Head
- Ion beam at 45° incidence
Determination of deposited mass via QCO frequency and beam current vs. time plot

- Before liquid target is irradiated with ion beam, a background flux measurement is completed
- QCM frequency difference slope increases when beam hits target measuring the sputtering flux
- When beam is off, some oxidation follows until original background flux is obtained

\[-\delta f_{\text{sp}} = \left| \delta f_{\text{sp+backg}} - \delta f_{\text{backg}} \right|\]

\[\Delta m \propto \frac{\Delta f_{\text{QCO}}}{D} = \frac{\langle \dot{m} \rangle}{\langle I \rangle} \frac{\Delta t}{\Delta t} = \frac{1}{\langle I \rangle} \langle \dot{m} \rangle \]

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Analysis of raw sputtering data

\[ Y \propto \frac{\Delta m}{D} = \langle \frac{\partial m}{\partial t} \rangle \Delta t = \frac{1}{\langle I \rangle} \langle \frac{\partial m}{\partial t} \rangle \]

\[ \frac{\partial m}{\partial t} = -A_{\text{crystal}} N_{\text{at}} \rho_{\text{quartz}} \frac{\partial f_{\text{net}}}{\partial t} \]

\[ Y_{\text{SPUT}} = \frac{\langle \frac{\partial m}{\partial t} \rangle \cdot N_A}{\langle I_{\text{beam}} \rangle \cdot f_i \cdot f_{\text{geo}} \cdot SC \cdot M} + f_{\text{geo}} R_{\text{inc}} Y_{\text{ref}} \]

Physically, \( f_{\text{geo}} \) is the amount of ejected material that strikes the crystal based on system geometry.
Polar distribution of sputtered particles 1: Sn\(^+\) self-sputtering

In general…
- This “geometric factor” is just an integral over the QCO crystal surface that estimates what fraction of the sputtered material strikes (but not necessarily sticks to) the crystal
- VFTRIM simulations are now performed for each ion-target combination to generate sputtered particle distribution “data” to input into the computation of this geometric factor

(Polar angle)
- \(A \cdot \cos^n\theta\) fits the VFTRIM polar “data”
- Previously assumed \(\cos^1\theta\) polar distribution – This correction of \(n\) made little difference in the final result

\[1000 \text{ eV Sn}^+ \rightarrow \text{Sn} \text{ at } 45^\circ \text{ incidence}\]
Polar distribution of sputtered particles 2: He\(^+\) ion bombardment

Polar distribution of sputtered Sn particles by 700eV He ion bombardment at 45° incidence (\(\sin\theta\) term accounted for)

- VFTRIM Results
- Fit curve

Expected cosine distribution from an isotropic collision cascade compared to “under cosine” of the heavy ion bombardment.
Azimuthal distribution of sputtered particles 1: Sn⁺ ion bombardment

- Previously assumed azimuthal isotropy for simplicity
- Significant anisotropy due to oblique incidence
- Parameters $A$ and $B$ are varied using $A + B \cdot \cos(\varphi - \pi)$ to fit VFTRIM azimuthal distribution “data”

(NOTE: This function is just a guess that fits most data sets well and so doesn’t necessarily have a physical interpretation)

1000 eV Sn⁺ → Sn at 45° incidence
Azimuthal distribution of sputtered particles 2: He\(^+\) ion bombardment

- Light ion bombardment (also at 45\(^\circ\)) shows a decreased level of anisotropy in comparison to Sn self-sputtering
- Expected from momentum considerations

\[ 0.77 + 0.23 \times \cos(\pi - \phi) \]
Bottom line on sputtered particle angular distributions?

- Switching from an azimuthally-symmetric cosine (polar) distribution changed $f_{geo}$ from 0.12 to 0.18 for the typical IIAX geometry for most extreme case (Sn self-sputtering)
- Validity of using VFTRIM based on athermal particle ejection?
  - If surface temperature-enhanced sputtering is a thermal process, we would expect a more isotropic distribution
  - However, if the enhancement is kinetic-based, perhaps using the distributions from VFTRIM remain valid
- Regardless, for a given system, a location should be able to be found where the two $f_{geo}$ for symmetric and asymmetric cases are more comparable
Light ion sputtering of solid and liquid Sn

D\(^+\) sputtering of Sn at 45° incidence

He\(^+\) sputtering of Sn at 45° incidence
Sn sputtering results from 4 species

Ion sputtering of solid and liquid Sn at 45° incidence

Dashed lines indicate VFTRIM results

$T_{\text{melt}} = 232^\circ\text{C}$
Early data indicate that Sn self-sputtering is also not significantly enhanced by temperature at least up to 400°C.

These results are similar to those for both Ne⁺ and Ar⁺ sputtering of Sn (from a temperature enhancement perspective).

Important to note that higher temperatures may still yet show temperature-enhanced properties.
VFTRIM Simulations of Sn self-sputtering

- Sn ions are predicted to have a mean incident angle of 22° and an average energy of 270 eV \cite{1} for an ARIES-AT configuration with a liquid Sn divertor.

- Thus, equally important is the reduction from decreasing the angle of incidence.

- Normal-incidence runs may be performed in the future to complement the oblique work done here.

- D\(^+\) sputtering of liquid lithium was shown to have a drastic (10 to 1000 fold) increase as a result of increasing the temperature.


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Future Work

• Near-term:
  – Check for alloying between Sn and Mo via XPS/AES
  – Focus on light ion (He\(^+\) & D\(^+\)) sputtering of liquid Sn at higher temperatures – up to 1000\(^\circ\)C
  – Return to heavy ion sputtering (Ne\(^+\), Ar\(^+\), and/or Sn\(^+\))
  – Reduce ion energies used (ideally to 200 eV with use of decelerator)

• Longer term:
  – Temperature dependent sputtering measurements and modeling of liquid Sn under light and heavy ion irradiation
  – Mixed solid material sputtering relevant to ITER (W, Be, C, etc.) at high temperatures
Summary

• Need high temperature D⁺ and Sn⁺ (or approximate heavy ion) sputtering yield data from liquid tin to further evaluate Sn as a divertor surface material

• Remaining planned experimental data on Sn
  – Temperatures: 20 to 1000°C
  – Energies: 200 - 1000 eV
  – Ion species: D⁺, He⁺, Ne⁺, Ar⁺ (Sn⁺ ?)

• Model resulting data for simulation use and improved physical understanding

• Switch to hot solid surfaces, namely W & Be (?)
  (need to find niche here)

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VFTRIM Simulation Results for 45° incidence on solid Sn

Ion Energy (eV) vs. $Y_{sp}$ (atoms/ion)

- $D^+ \rightarrow Sn$
- $He^+ \rightarrow Sn$
- $Ne^+ \rightarrow Sn$
- $Ar^+ \rightarrow Sn$
- $Sn^+ \rightarrow Sn$ (Sputter)
- $Sn^+ \rightarrow Sn$ (Reflect)
- $Sn^+ \rightarrow Sn$ (Total)