Georgia Tech Step-Up 2012 at
The Advanced Semiconductor Technology Facility

GROWTH METHODS AND
CHARACTERIZATION OF LAYERED
LITHIATED TRANSITION METAL OXIDES

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Prior Work and Background

- Materials
- Applications
- Methods

Current efforts are highlighted, those within this program are underlined.
Prior Work and Background: Materials

History of Interest in Layered Transition Metal Chalcogenides, Intercalated with Alkali Ions:

- Host Metal – Ti, V, Cr, Mn, Fe, Co, Ni, Nb, Mo, W
- Host Chalcogens – Oxides, Sulfides, Selenides
- Guest Intercalates Ions – H, Li, Na, K

- General Formula A[A_{1-x}M_x]C_2
- Current interest, x=1: LiCoO_2, LiNbO_2
Prior Work and Background: Applications

- Applications
  - Rechargeable Ion Battery Cathode (usually Li⁺)
  - Electrochromic Materials
  - New electronic components
    - High Density Memory
    - Multi-level memory
  - Exploit magnetic ordering for novel RF applications
  - Exploit memristor behavior for neuromorphic computing
Prior Work and Background: Methods

- Methods
- Molecular Beam Epitaxy (MBE)
- Electrodeposition
- **Sputtering**
  - Step Up 2011 – Lesker PVD75 to Sputter LiCoO₂
  - Step Up 2012 – Denton Discovery 2 to Sputter LiNbO₂
STEP-UP 2012 Goal

- Develop Method of Depositing Lithium Niobite Crystals for Analog Memristor Devices to Support Neuromorphic Computing Applications
  - Bring up newly acquired Denton sputterer
  - Characterize deposition rate vs. sputtering parameters
  - Co-deposit Li$_2$O with Nb in oxygen atmosphere for alternative method of depositing Lithium Niobite (LiNbO$_2$) Thin Films
Prior Work and Background: PLAN

BRING UP TOOL

CHARACTERIZE TOOL

Deposition Rates vs.
- Power
- Pressure
- Flow Rate
- Temperature

Plasma vs.
- Pressure
- Flow Rate

ANALYZE CO-DEPOSITS

Li₂O / Nb
With O₂ / Ar

Set Stoichiometric Ratios
Estimate Co-deposition param.
Codeposit
- XRD
- Raman
- XPS
Prior Work and Background: Progress

BRING UP TOOL

CHARACTERIZE TOOL

ANALYZE CO-DEPOSITS
Li$_2$O / Nb
With O$_2$ / Ar
Plan – New Sputterer

- 2011 Lesker PVD75
- 2012 Denton Discovery
Plan – New Sputterer

**2011 Lesker PVD75**
- Operational
- Heated platen to 400C (requires post anneal)
- 2 RF Guns
- Passive platen (“rain”)

**2012 Denton Discovery**
- Repair/Characterization
- Direct heat die to 800C
- 3 Guns (2 RF+ 1 DC) (faster deposit of metals)
- Active (plasma) platen (“snow”)

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**Note:** The text in the diagram is not legible due to the image quality.
Theory – Anatomy of Sputtering
Water Cooling of Magnets and Target

Progress:
- Replace Broken Magnets
- New magnets interfere with cooling
Theory – Anatomy of Sputtering
Lesson Plan # 1
Importance of Magnet Temperature
Currie Engine
Theory – Anatomy of Sputtering
Ring / Center Magnet on Ni plate

Progress:
- Matching correct magnet polarity on all gun
- Guns are installed NN, NS, SN or SS?

Lesson Plan
How do you know North vs. South?
Theory - Anatomy of Sputtering

Lesson Plan # 2

Right Hand Rule
Theory – Anatomy of Sputtering
Target (material to be deposited)
Theory – Anatomy of Sputtering
DC or RF E field between “Snout” & Target
Theory – Anatomy of Sputtering

And if the field is large enough ...

Progress:
- Identified defective tuner
- Identified defective power supply
- 1 of 3 guns non-functional
Theory – Anatomy of Sputtering
But recall the magnets behind the target…
Theory – Anatomy of Sputtering
And moving electrons in a magnetic field...
Theory – Anatomy of Sputtering
When spiraling electrons strike a gas atom...
Theory – Anatomy of Sputtering

Finally, when Ar plasma bashes into target...
Despite a long list of additional tribulations ...

- Broken magnets
- Correcting magnet polarity
- Adapting cooling system to new magnets
- Failed RF Supply and Tuner
- Shutter proximity to target
- Shorted RF cable
- Sputtering on opposing gun
- DC gun “holds down” RF gun
- Vacuum sensor failed
- Failed turbo pump cable
- Delay in adapting working supply to oxide gun
- Degradation in oxide plasma power
- Undersized heater relay
- Warping platen at high temperature
- The zero power bug – SW interpretation
- And of course lab evacuations
Success – As of last week, we …

Struck plasma and deposited from the right gun …
Success – As of last week, we ...

Struck plasma and deposited from the center gun ...
Success – As of last week, we ...

Simultaneously had plasma on two guns and platen …
Success – As of last week, we ...

Co-deposited Li$_2$O and Nb!
Success – As of last week, we ...

Cracked our oxide target 😞
Nevertheless we made significant progress ...

- We went from zero plasma / no deposition, to two guns depositing onto active platen
Nevertheless we made significant progress

- Characterized tool for deposition rates of Nb alone and Li₂O alone to aid in estimating stoichiometry during co-deposition.

![Graph showing Niobium Deposition Rate versus DC Gun Power](image-url)
Nevertheless we made significant progress

- How do we know which oxide species?
Nevertheless we made significant progress

- Planning deposit recipes – deposition rate versus:
  - Target material / gun type
  - Chamber pressure
  - Flow rate

![Graph: Striking RF Power of Li$_2$O Target on Denton Discovery Center Gun versus Chamber Pressure](image)
Conclusion

- Ongoing effort to characterize sputter
  - Begin high temperature depositions (800°C)
  - Repeat and expand characterizations deposition rates at high temperature
    - Versus pressure
    - Versus RF power
    - Versus flow rate
    - Versus gas mix
- Design co-deposition, characterize
  - XRD
  - Raman
  - XPS
  - VSM
  - Ion Mobility
More magnetics - ordering - LiCoO$_2$
More magnetics - ordering - LiCoO$_2$
References


[2] K. Kang, G. Ceder, Factors that affect Li mobility in layered lithium transition metal oxides, PHYSICAL REVIEW B 74, 1-7


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