Silicon Electroplating for Solar Cells and Thin Film Transistors
(summer school tutorial)

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Outline

* Motivation
* Description of thin film devices: TFT, PV, NUC and PV-NUC hybrid
* Rationale for electrodeposition from ionic liquids
  - active metals and water
  - formation of active metals in liquid salts
    > high temperature molten salt aluminum
    > low temperature molten salt aluminum
    > low temperature silicon electrodeposition
* Preliminary electrodeposition of thin films of silicon for PVs
* Conclusions
Motivation for electroplating silicon

Electrodeposition of silicon offers a clean, effective and inexpensive alternative processing to conventional silicon processing for making a number of devices that employ thin layers of semiconducting silicon, including:

- Thin film transistors (TFTs),
- Photovoltaics (PVs)
- and hybrid photovoltaic and capacitor device, a “so-called” Renewable Hybrid Power Source (RHPS),
Description of Thin Film Transistors for electronics applications

A thin-film transistor (TFT) is a field-effect transistor. It’s made by depositing thin film of a semiconductor active layer, a dielectric layer and metallic contacts over a supporting substrate. 

http://en.wikipedia.org/wiki/Thin-film_transistor
Description of 3D Thin-Film Photovoltaic Device for electrical power generation

Photovoltaic component of a Renewable Hybrid Solar Power Source (RHPS)
Description of 3-D Thin Film Nanowire Ultra Capacitor (NUC) for electrical storage and power delivery

Electrical energy storage component of a Renewable Hybrid Solar Power Source (RHPS)
Description of Renewable Hybrid (PV – NUC )Power Source Device

Hybrid Device

NUC - PV

Storage

Nanowire Ultra Capacitor

Solar Cell

Photo Voltaic Schottky Diode

Hybrid Device

Storage

Solar Cell

Nanowire Ultra Capacitor

Photo Voltaic Schottky Diode
Schematic diagrams of a simple hybrid device in a monolithic substrate divided into two sectors:

i. storage sector (NUC)
ii. solar cell sector (Schottky diode)
Description of Renewable Hybrid Solar Power (RHSP) Source (continued)

Why use a PV-NUC hybrid design?

Problems– line loss and intermittency
• PV current-voltage characteristic is non-linear exhibiting a “knee” feature with diode-like behavior and best conversion efficiency is when the operating point is in the vicinity of the “knee”.
• PV power generation is inherently intermittent due to variation of ambient conditions

Solution- hybrid design
• distributed storage (NUC) minimizes resistance to PV and allows maximal opportunity to hold PV at $V_{\text{max}}$ so power generation is at the knee region
• allows steady power by discharge of storage device during any intermittency in the PV power generation
• NUCs can handle many thousands of charge / discharge cycles and are more reliable and durable than batteries for storing solar energy.

Hybrid design maximizes solar power generation and avoids intermittency during solar power generation that could cause instability and other problems.
Electro-Plating Processing for Renewable Hybrid Power Source (RHPS)

RHPS = Silicon Photovoltaic Cell (PV) + Nanowired Ultra Capacitor (NUC)

A Silicon Photovoltaic Cell (PV) is a conventional Si photovoltaic cell made using the low-cost and clean silicon electrodeposition from an electrolyte of ionic liquid (e.g. n-butylpyridinium chloride with a suitable silicon species SiCl₄ or Si₂Cl₆) under an inert (N₂) atmosphere at 120°C to avoid water and oxidation of the growing Si films.

A nanowire ultra capacitor (NUC) is a new kind of high energy density dielectric capacitor invented at University of Arizona that consists of an array of metal nano wires formed by electroplating in a nano porous membrane from a bottom metal plate and with an unconnected top metal plate.

- Plating consumes much less energy than conventional PV processing.
- Plating is conformal permitting construction of NUC and textured PV device geometries in the same monolith which leads to high conversion efficiency.
- This plating processing is environmentally friendly, no volatile organic compounds (VOCs) or other noxious vapors. NUC disposal sends no toxic materials to landfills.
Real Structure of NUC and Nanoporous Membrane

Structures in a nanowire ultra capacitor (NUC):

a) schematic diagram of overall NUC structure (yellow color indicates metal white is the dielectric material, grey is the air gap),
b) SEM image of the top view of a dielectric membrane fabricated in our laboratory with the inserted zoomed-in image of a top fragment,
c) SEM image of membrane cross-section (side view) showing nanopores containing electroplated Cu nano-wires.
## Comparing NUC and ordinary dielectric capacitor

<table>
<thead>
<tr>
<th>Capacitor</th>
<th>Capacitance</th>
<th>Capacitance Gain</th>
<th>Total Mass Capacitance</th>
<th>Capacitor Density</th>
<th>Capacitor Density Gain</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(µF)</td>
<td>(Relative to M16)</td>
<td>(gram)</td>
<td>(µF/gram)</td>
<td>(Relative to M16)</td>
</tr>
<tr>
<td>Blank (M16) 2 electrodes on Anodisc</td>
<td>0.0060</td>
<td>1</td>
<td>0.047</td>
<td>0.13</td>
<td>1</td>
</tr>
<tr>
<td>Nanowires In Anodisc (M24) Air filled</td>
<td>16.8</td>
<td>2,800</td>
<td>0.050</td>
<td>338</td>
<td>2,600</td>
</tr>
<tr>
<td>Nanowires+IL1 (T4) IL filled</td>
<td>1170</td>
<td>195,000</td>
<td>0.055</td>
<td>21,400</td>
<td>164,700</td>
</tr>
<tr>
<td>Nanowires+IL2 (M29) IL filled</td>
<td>1000</td>
<td>166,000</td>
<td>0.060</td>
<td>16,580</td>
<td>127,500</td>
</tr>
</tbody>
</table>

**Notes:**
1: IL1 is 2-fluoropyridinium trifluoromethyl sulfonylimide; IL2 is 1 butyl 2,3 dimethylimidazolium tetrafluoroborate
2: The blank (M16) is an Anodisc with 200 nm pores and both the top and a bottom electrodes are planar.
3: M24 is the same as M16 but with nanowires electroplated on the bottom electrode and a top planar electrode
4: M16, M24, T4 and M29 have identical pore diameters, area and thickness
5: Gain is the increase relative to the blank, M16, a capacitor with no nanowires in the Anodisc.
Energy density for one NUC measured at 1V extrapolated to higher voltages

<table>
<thead>
<tr>
<th>Operating Voltage (V)</th>
<th>Spec. Capacitance of NUC + pIL (µF/g)</th>
<th>Energy Density (Wh/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>650000</td>
<td>0.09</td>
</tr>
<tr>
<td>100</td>
<td>650000</td>
<td>900</td>
</tr>
<tr>
<td>150</td>
<td>650000</td>
<td>2000</td>
</tr>
</tbody>
</table>

* Extrapolated numbers in red

If possible …
high voltage operation leads to very high capacitor energy densities!
3 Reasons for electroplating Si photovoltaics

1) Allows the forming of complex Si structures

- e.g., vertically oriented nano-Schottky diodes structure (below)
- ...which is virtually impossible to make using conventional Si processing

2) Clean (no VOCs, noxious waste or fumes)

3) Inexpensive low-energy processing and good efficiency
Rationale for electrodeposition from ionic liquids

Electrodeposition of semiconducting silicon from salt electrolytes to make for the RHPS
Rationale for electrodeposition from ionic liquids

Why use non-aqueous salt electrolytes for Si electroplating?

It is really the only option because ...

Metals spontaneously react with molecular water in aqueous electrolytes.

The driving force to form a metal-oxide from a metal in the presence of water is the heat of formation ($\Delta H^\text{f}$) of the metal oxide:

<table>
<thead>
<tr>
<th>Metal Oxide</th>
<th>$\Delta H^\text{formation}$ (calories/gram)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>reactive metals</strong></td>
<td></td>
</tr>
<tr>
<td>aluminum oxide (Al$_2$O$_3$)</td>
<td>- 4000 (= -1676 kJ/mole)</td>
</tr>
<tr>
<td>silicon dioxide (SiO$_2$)</td>
<td>- 3418</td>
</tr>
<tr>
<td><strong>unreactive metals</strong></td>
<td></td>
</tr>
<tr>
<td>copper oxide (Cu$_2$O)</td>
<td>- 278</td>
</tr>
<tr>
<td>platinum oxide (PtO$_2$)</td>
<td>- 84 (= -80 kJ/mole)</td>
</tr>
<tr>
<td>gold oxide (Au$_2$O)</td>
<td>&gt; 0 (Au-oxide unstable)</td>
</tr>
</tbody>
</table>

In aqueous electrolytes, metal forms metal-oxide by the “Local Cell” process, which

- prohibits the smelting of a “reactive” metal, e.g., aluminum, in air
- even interferes with catalysis by an “unreactive” noble metal, platinum
The “Local Cell” Process

Shows how water is corrosive

- Water (H₂O) reacts with a reactive metal (M), like aluminum or silicon, forming undesirable metal oxide (MO) as:

\[ M + H_2O \rightarrow MO + 2\ e^- + 2\ H^+ \]  \hspace{1cm} (RXN 1)

- The liberated electrons are consumed by proton to form hydrogen (reaction 2a). e⁻ can be consumed by oxygen to form water (reaction 2b).

\[ 2\ e^- + 2\ H^+ \rightarrow H_2 \]  \hspace{1cm} (RXN 2a)

\[ 2\ e^- + \frac{1}{2} O_2 + H_2O \rightarrow 2\ OH^- \]  \hspace{1cm} (RXN 2b)

- Adding reaction 1 with reaction 2a (or reaction 2b) gives the net undesirable metal oxide formation reaction, reaction 3a (or reaction 3b).

\[ M + H_2O \rightarrow MO + H_2 + \text{heat} \]  \hspace{1cm} (RXN 3a)

\[ M + \frac{1}{2} O_2 \rightarrow MO + \text{heat} \]  \hspace{1cm} (RXN 3b)

**Metal-Oxide (MO) FORMATION (RXN 3a) occurs in the presence of WATER even in anaerobic environments because the electron (e⁻) is more stable on H⁺ than on the reactive metal (M).**

Similarly, in the presence of oxygen from air, RXN 3b occurs because e⁻ is more stable on O₂ than M.
Historical Perspective: Hall Process

Charles Martin Hall
(1863-1914)
Founded Aluminum Company of America (ALCOA) in 1907.
Brought aluminum to America in 1886 using High-Temperature Molten-Salt Electrochemistry

Hall’s motivation:
Aluminum is one of the most abundant element on the planet, yet …
in 1886, aluminum cost as much as precious metal silver, $8 per pound.
By 1914, the cost of aluminum was down to 18 cents due to Hall.

Still today, Al costs <$1 per pound and Ag cost >$200 per pound
Hall-Héroult Process: first electroplating of a reactive metal

Unlike Fe, Al can not be smelted, because it spontaneously reacts with water

Al is electroplated from Al-oxide (Al$_2$O$_3$) in a water-free molten salt electrolyte

- The molten salt, called “Cryolite”, contains: Na$_3$AlF$_6$, AlF$_3$, Al$_2$O$_3$ melted together at 900°C
- Large voltage window is achievable in cryolite salt electrolyte (V$_{\text{cryolite}}$ >> 1.23 V, the voltage limit in water).
- Al electroplates at a cathode at a potential of -1.662 volt vs. S.H.E.

Graphite Anode reaction

\[
3 \text{Al}_2\text{O}_3\text{F}_4^{2-} + 3\text{C} + 24 \text{F}^- \rightarrow 3 \text{CO}_2 + 6 \text{AlF}_6^{3-} + 12 \text{e}^- 
\]

Aluminum Cathode reaction

\[
12 \text{Na}^+ + 4 \text{AlF}_6^{3-} + 12 \text{e}^- \rightarrow 12 \text{Na}^+\text{F}^- + 4 \text{Al} + 12 \text{F}^-
\]

Net reaction

\[
2 \text{Al}_2\text{O}_3 + \text{C} \rightarrow 4 \text{Al} + \text{CO}_2
\]

Charles M. Hall, US Patent 400,766; filed July 9, 1886; patented April 2, 1889; Paul T. Héroult, of Paris, FR 175,711; April 23, 1886.
What did Charles Hall teach us?

- **Salt electrolytes allows water-free processing on a cathode**
- **Avoids reactions 1-3, the corrosion of metal by water**
  
  Practically allowed manufacture of the reactive metal, aluminum.

- Water (H₂O) reacts with a reactive metal (M), like aluminum or silicon, forming undesirable metal oxide (MO), shown below.
  
  \[
  M + H_2O \rightarrow MO + 2 \text{e}^- + 2 H^+ \quad \text{(Reaction 1)}
  \]

- The liberated electrons are consumed by proton to form hydrogen (reaction 2a) or by oxygen reduction to form water (reaction 2b).
  
  \[
  2 \text{e}^- + 2 H_2O \rightarrow 2 \text{OH}^- + H_2 \quad \text{(Reaction 2a)}
  \]
  \[
  2 \text{e}^- + \frac{1}{2} O_2 + H_2O \rightarrow 2 \text{OH}^- \quad \text{(Reaction 2b)}
  \]

- Adding reaction 1 with reaction 2a or reaction 2b gives the net undesirable metal oxide formation reaction (reaction 3a or reaction 3b).
  
  \[
  \begin{align*}
  M + H_2O & \rightarrow MO + H_2 \quad \text{(Reaction 3a)} \\
  M + \frac{1}{2} O_2 & \rightarrow MO \quad \text{(Reaction 3b)}
  \end{align*}
  \]

Undesirable reactions 3a and 3b are avoided in the absence of water.
Reflections on Metal Making

- **Coinage and noble (Cu, Ag, Au) metal making**

  *Low or no negative heat of formation*
  - Au oxide (>0 cal/g)  
  - Ag oxide (-32 cal/g)  
  - Cu oxide (-278 cal/g)  
  
  Can plate and smelt in presence of water, air

- **Base (Fe, Steel) metal making**

  *Moderate heat of formation*
  - Fe oxide (-1230 cal/g)  
  
  Can smelt & plate in water, air

- **Active (Al, Si) metal making**

  *High negative heat of formation*
  - Al oxide (-4000 cal/g)  
  - Si oxide (-3418 cal/g)  
  
  Can’t plate Al salts in water or readily smelt

Hall achieved a great success making ALUMINUM in a molten salt at 900°C.

Yet for many years further use of neat salt electrolytes was ignored!
A new tack: Low Temperature Aluminum Electroplating


- Conditions
  For AlCl₃ in n-butylypyridinium chloride salt electrolyte, the melting temperature is 30°C and the plating potential is -2.1V vs. Ag/AgCl
  (A higher temperature alternative is AlCl₃ in NaCl salt electrolyte, melting temperature is 175°C and the plating potential is -1.9V vs. Ag/AgCl)

- Electrolyte forming reactions:
  At low Al concentrations, mononuclear Al species form in the melt.
  \[ 2R^+Cl^- + Al_2Cl_6 \xrightleftharpoons{k_1} 2R^+AlCl_4^- \]
  At high Al concentrations, dinuclear Al species form in the melt.
  \[ 2R^+AlCl_4^- + Al_2Cl_6 \xrightleftharpoons{k_2} 2R^+Al_2Cl_7^- \]

- Plating cell and reactions for Al electrowinning:
  Cathode reaction:
  \[ 2R^+AlCl_4^- + 6 e^- \rightarrow 2Al + 2R^+Cl^- + 6Cl^- \]
  Al Anode reaction:
  \[ 2Al \rightarrow 2Al^{3+} + 6 e^- \]
  Net reaction:
  Transfer Al from anode to cathode
Low temperature electroplating of Al introduced ionic liquid electrochemistry

In mid 1950’s first shown by Hurley and all.

In the mid 1970’s, R. Osteryoung showed a mixture of 1-butylpyridinium chloride and 2 AlCl₃ melts at 35°C forming a low temperature molten salt, [1-butyl pyridinium][heptachloro aluminate].

The reduction the heptachloroaluminate anion [Al₂Cl₇]⁻ in this “ionic liquid” is the limiting cathode process, forming aluminum metal at the low temperature of 35°C.

In the 1990’s, there were only a handful of papers and patents on low-temperature liquid salts (ionic liquids)

They are now being studied at an ever-quickening pace with thousands of papers per year.

What are these ionic liquids?
Properties of Ionic Liquids

- Ionic Liquids (ILs) are molten salts with melting points below 100°C.
- Room temperature ionic liquids (RTILS) melt below 25°C.
- Composed of over 99.99% ions
- Low vapor pressures
- Highly conductive
- Excellent solvating properties
- Thermally stable, for both high and low temperature uses.
- Large voltage window for electrochemical reactions ($V_{\text{cell}}$ up to 5 volt)

- Low water activity
  - allows plating of “active metals” (Al, Si Zr, etc.)
  - important to activity of Pt-catalyzed cathode
  - may permit use of non-Pt catalysts

http://www.nature.com/nature/journal/v439/n7078/images/nature04451-f2.2.jpg
Typical components for room temperature ionic liquids (RTILs)

- Room temperature ionic liquids (RTILs) consist of:
  - Bulky and asymmetric organic positive ions (cations), such as:
    - 1-alkyl-3-methylimidazolium,
    - 1-alkylpyridinium,
    - N-methyl-N-alkylpyrrolidinium
    - ammonium ions.
  - Negative ions (anions) include:
    - halides (Cl⁻, F⁻, Br⁻, I⁻) which generally give high melting salts,
    - Inorganic anions such
    - tetrafluoroborate
    - hexafluorophosphate
    - large organic anions
    - bistriflimide
    - triflate
    - tosylate
    - formate
    - alkylsulfate
    - alkylphosphate
    - glycolate

  Tailorable
  Properties, such as:
    - melting point,
    - viscosity,
    - solubility
    - stability
    - are determined by the identity and geometry of substituents on the cation and anion.
Simple preparation of ILs

Amine + Acid → Stirring in acetone/dry-ice bath → Solution → Heated at 120°C in Silicon bath → H2O Removal → Dried in Vacuum Oven at 90°C

Example: ammonium hydrogen sulfate salts

Mass analysis, Karl-Fischer titration
Samples: H2O = 100 : <1 (mol%)
Conductivity of Ionic Liquids

[\text{[NH}_4^+][\text{HF}_2^-] \quad \text{7.7M aqueous LiCl solution}]

1.1M aqueous LiCl

4:6 DMAN-MAN

EAN

IL electrolytes have ionic conductivities rivaling aqueous solutions

Requirements of Electrolytes For Plating Active Metals

CONDUCTIVITY

• Conductor for ions, Ionic Conductivity $> 10^{-2}$ Siemen/cm
• Insulator for electrons, Electron Conductivity $< 10^{-9}$ Siemen/cm

COMPATIBILITY WITH ELECTROPLATING OF ACTIVE METALS

• No water, no passive layer (metal oxide, “tar”) formation

STABILITY

• Stable to electric field; no reaction with products

All electrolyte properties accessible by *tailoring* the ionic liquid

Preliminary electrodeposition of thin films of silicon for PVs
Silicon Plating from Ionic Liquid Electrolyte

- Ionic liquid electrolyte: RCl (n-butylpyridinium chloride) and silicon (SiCl₄, NaSiF₆, SiHCl₃, or Si₂Cl₆).

- Reactions
  - IL Rxn with Si: \(2 R^+ Cl^- + Si_2Cl_6 \rightarrow 2 R^+ SiCl_4^-\)
  - Cathode Rxn: \(R^+ SiCl_4^- + 3 e^- \rightarrow Si + R^+ Cl^- + 3 Cl^-\)
  - Anode Rxn: \(Si + R^+ Cl^- + 3 Cl^- \rightarrow R^+ SiCl_4^- + 3 e^-\)
  - Net Rxn: *Transfer of Si from anode to cathode*

- 3 electrode cell
  - Cathode, reference (Ag/AgCl), anode
  - RT to 120°C operating temp (avoid water)
  - Inert gas purge

N. Borisenko et al., "In Situ STM Investigation of Gold Reconstruction and of Silicon Electrodeposition on Au(111) in the Room Temperature Ionic Liquid 1-Butyl-1-methylpyrrolidinium Bis(trifluoromethylsulfonyl)imide", J. Phys. Chem. B 2006, 110, 6250-6256

Si Electroplating vs. conventional Si processing

Motivation for electroplating Silicon

*it’s a cleaner, lower energy and more versatile alternative to conventional Si processing*

- **Electroplating Si from Ionic Liquid**
  - Simple process to make complex structures
  - Can plate doped Si
  - Relatively low T process (< 120°C)
  - Environmentally friendly (no VOCs)
  - Relatively low cost

- **Silicon production from Si-wafer processing**
  - High temperature, Energy Intensive process
    - React sand w/ Zn metal (950°C) OR
    - CVD on pure Si rod (1150°C)
    - Purified material melted down (1414°C), Czochralski process
  - Complex processes to make structures in wafer
    - Si wafer prep.: Wafer sliced, lapped, polished, masked, etched, etc.
    - High temp process, Doping, annealing, oxide growth, etc.
  - Hazardous waste and process fluids (HF, SiH₄)
  - High cost
Silicon plating on Metal

Electrodeposition of silicon on titanium in a room temperature ionic liquid: butyl, 3-methylimidazolium chloride, [Bmim] BF$_4$

Cyclic voltammogram of titanium in [Bmim]BF$_4$ with ferrocencene but no SiCl$_4$ under N$_2$ atmosphere. Scan rate: 10mV/s. Temperature: 25°C.

Cyclic voltammogram of titanium in [Bmim]BF$_4$ saturated with SiCl$_4$ under N$_2$ atmosphere. Scan rate: 10mV/s. Temperature: 25°C.
Electroplating of Si on titanium metal at constant potential

Chronoamperometry of titanium metal in [Bmim]BF₄ electrolyte saturated with SiCl₄ at constant potential of -2.0 V vs. Fc/Fc⁺ under a N₂ atmosphere at a temperature of 25°C.
Scanning Electron Microscopy (SEM)

SEM image of silicon on titanium and bare titanium substrate
Energy Dispersive Absorption of X-rays (EDAX)
Analysis of Silicon on Titanium

EDX for the Ti sample before Si electrodeposition

EDX for the Ti sample with Si deposited at -2.0V for 10 minutes
Cost versus Efficiency Tradeoff

Efficiency per length $\propto \tau^{1/2}$
(where $\tau$ is hole-electron pair recombination time)

- **Thick Film**
  - Large Grain
  - Single Crystal Si
  - CVD Si

- **Thin Film**
  - Small Grain
  - Polycrystalline Solids
  - Electroplated Si

Long $d$
Requires High $\tau$
High Cost

Short $d$
Allows Lower $\tau$
Low Cost

*Thin electrodeposited Si films allow high efficiency with low $\tau$ lead to low cost!*

“RADIAL PN JUNCTION, WIRE ARRAY SOLAR CELLS”, B. M. Kayes, M. A. Filler, M. D. Henry, J. R. Maiolo III, M. D. Kelzenberg, M. C. Putnam, J. M. Spurgeon, K. E. Plass, A. Scherer, N. S. Lewis, H. A. Atwater California Institute of Technology, Pasadena, CA 91125
Versatility of electroplating Si: *Photovoltaics*

ELECTROPLATING allows forming *complex Si structures* at low temperatures. This is almost *IMPOSSIBLE* to make using *CONVENTIONAL Si PROCESSING*.

E.g., vertically oriented nano-structured Schottky diodes

Brews and Palusinski
Conclusions

- Electrochemical conversion in salt electrolytes avoids water and gives a low cost and clean manufacturing process for making thin layers of semiconducting silicon
  - photovoltaics on a small (<1 Watt) to large (1 Mega W) scale
  - TFTs for displays, power chips etc.

- Electroplating in nanoporous substrates allows mass manufacture of a low-grade but practical and inexpensive semiconducting silicon devices
  - TFTs
  - Ultracapacitors (nano copper wire array)
  - Photovoltaics (nano Schottky diode array)

- The hybrid photovoltaic-capacitor device electrodeposited within the same monolithic structure that is stable to chemical, thermal and radiation exposures
  - overcomes stability problems in grid due to intermittencies (sub-second to minutes) intrinsic with solar derived power
  - stores energy to give power when solar generator is not on for long times (hours…when sun is not out)
  - provides a renewable power-source
  - can provide grid power or autonomous power in remote locations.

Questions?