Senior Design Group

May 14-03

Design and implementation of a cryogenic electrical characterization system for organic photovoltaic cells

Martin Andersen, Mouhamadou Diallo, Nicholas Rodriguez, and Joshua Straquadine

Client: ISU Nanolab, Dr. Chaudhary and John Carr
Project Description

MOTIVATION, GOALS, AND OVERVIEW
Organic Photovoltaic Cells

A promising new energy technology... 
...but only if **efficiency** can be improved!

Electronic defects are one major obstacle

The method of **Thermally Stimulated Currents (TSC)** can help us examine those defect states... 
...but setups are expensive and complicated!

Our project is to design and build a system that can simplify TSC measurements for our client.
Thermally Stimulated Current Measurement

LUMO

HOMO

Current

Temperature

300 K

80 K
System Overview
## Project Goals

### Where we started
- Liquid nitrogen cryostat and vacuum chamber
- No temperature verification
- Current meter with 10 pA resolution
- Manually controlled instrumentation setup

### What was needed
- Must be able to bring sample to 80 K
- Need a method of real-time measurement
- Need 1000x better current resolution
- Experiment operation and data collection must be automated
Market and Literature Survey

- No dedicated commercial systems for TSC exist!
- High quality cryostats cost $30,000 or more
- Most research papers publish very few details
- Spoke with researchers in physics department
  - Significant experience with cryogenic systems
- Researched one individual component at a time
  - Temperature Sensors
  - Thermal interface materials
  - Insulation
  - Low current measurements
Project Implementation

HOW WE HAVE SOLVED THE PROBLEM
## Project Schedule

<table>
<thead>
<tr>
<th>ID</th>
<th>Task Name</th>
<th>2013</th>
<th>2014</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Sep</td>
<td>Oct</td>
</tr>
<tr>
<td>1</td>
<td>Research</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Part Acquisition</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>Testing new parts</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>Preliminary Runs</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Software Design</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>System Assembly</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>Full system tests</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>Troubleshooting</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Cold Shroud

- Radiative heating is one of the largest problems
- Solution: mask with a reflective, cold surface
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Temperature Sensors

- Thermocouples
- Silicon Diodes
- Thermistors
  - NTC Semiconductor
  - PTC Metal

Platinum RTD

![Graph showing the relationship between temperature and resistance for Platinum RTD.](image-url)
Thermal Interface Materials

- Conformable Pads
  - Parker Chomerics
  - 3M

![THERMAL PAD FREEZEOUT](chart.png)

- Cryostat
- Sample
- Glass
- Copper
Thermal Interface Materials

- Conformable Pads
  - Parker Chomerics
  - 3M

- Cryogenic Epoxy
  - Stycast 1266
  - Reacted with organic layer!
Thermal Interface Materials

- Conformable Pads
  - Parker Chomerics
  - 3M

- Cryogenic Epoxy
  - Stycast 1266

- Cryogenic Vacuum Grease
  - Apiezon N Grease

- Cryogenic Varnish
  - Lakeshore VGE-7031
Probe Effects

Bent wire for applying force

Cryostat Stage
Probe Effects

- Thinnest wires possible, cooled on cryostat
- Wire tension used to hold tip down
  - Separated from sample surface
Automation Software

Flexible
- Allow for experiment customization
- Support multiple modes

Robust
- Elegantly handle problems
- Keep user safe

Accessible
- Intuitive GUI
- Legible output
Automation Software

Thermally Stimulated Current Dashboard

Export Results to: C:\Users\Joshua\Dropbox\Senior Design Group May1403\Automation

Choose Profile: Fractional

Excitation Length: 120 s

Excitation Amplitude: 5 V

Ramp Rate: 5 K/min

Target Temperature: 80 K, 150 K, 200 K

Setpoint

75 K, 150 K, 200 K, 250 K, 300 K, 350 K

Sample Temperature

75 K, 150 K, 200 K, 250 K, 300 K, 350 K

128.129 K

Thermally Stimulated Current

Program opened at 7:32:31 PM on 12/9/2013

Initializing...
Keithley SMU GPIB initialization passed
Lakeshore GPIB initialization passed
Gathering experiment setpoints...

Setpoints submitted by user:
Excitation Length = 120 ms
Excitation Amplitude = 5 V
Ramp Rate = 5 V/min
Temperature Setpoints = 80 K, 150 K, 200 K
Conclusion

- We have designed a setup that cools the sample thoroughly close to 80 K
- We are able to reliably measure the temperature in real time
- We have a working probe setup
- All individual components of the system are up and running
Next Semester

- Full system assembly
  - GPIB control and data collection
  - Institution of new picoammeter

- Continual improvements in insulation

- Optimization of experimental procedures
  - Cooling rates, excitation profiles, soak times
Questions?

THANK YOU FOR YOUR TIME!
Appendices
References

US Sensor PPG102A6

- 1000 Ω ± 0.06%
- Range: -200°C to +600°C (73.15 K to 873.15 K)
- Platinum-Nickel Leads
- Linear TCR: 3,850 ppm/K
- $22.00 each

R-T Diagram
Apiezon N Grease

- Temperature Range: 0.15 K to 300 K
- Thermal Conductivity (80 K) = 0.1 W m\(^{-1}\) K\(^{-1}\)
- Vapor Pressure (273 K) = 2.67 x 10\(^{-7}\) torr
- Volume Resistivity 2 x 10\(^{16}\) Ω m
Solar Cell Samples

- Poly-3-hexylthiophene
- Bulk heterojunction
- Manufactured onsite using glass slides pre-deposited with ITO

![Chemical Structure: CH₂(CH₂)₄CH₃]

![Diagram of solar cell layers: Glass (0.7 mm), Indium Tin Oxide (ITO) (140 nm), Organic Active Layer (100-300 nm), Aluminum (150 nm)]
Software Architecture

Main State Machine

- Initialize
- Setpoints
- Export
- Ramp-up
- Cool
- Excitation

GUI Control

File Output

GPIB Spooler
Lakeshore Temperature Controller

- Two cryogenic temp. sensors embedded in cryostat
- 3-mode variable power heater, 50 W
- Accurate down to 1.2 K
- Thermal EMF compensation for resistive sensors
Keithley 6485 Picoammeter

- 5½ digit display
- Resolution: 10 fA
- 1000 reads per second
- Accuracy: ±0.4%
- Coaxial hookups, triax adapters for low currents
- $1,660 (with educational discount)
VGE-7031 Cryogenic Varnish

- Clear modified phenolic
- Easy to apply and remove
- Rigid when dry, dissolves in alcohol
- Vacuum compatible to $10^{-9}$ Torr
Stycast 1266 Cryogenic Epoxy

- 2-part formula, 100 : 28 mix ratio by weight
- Low viscosity, lowered still by applied heat
- 30 minute working life, 8-16 hr. cure at 300 K
- Optically clear, electrically insulating
Conformal Thermal Pads

- **3M:**
  - 5519S, 5591S

- **Parker Chomerics:**
  - Therm-A-Gap G579

- All three highly conformable, slightly tacky, electrically insulating

- Thermal conductivity (300K) 1-3 Wm\(^{-1}\)K\(^{-1}\)