



# Senior Design Group

# May 14-03

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

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Client: ISU Nanolab, Dr. Chaudhary and John Carr

# Project Description

## MOTIVATION, GOALS, AND OVERVIEW

# Organic Photovoltaic Cells

3

A promising new energy technology...

...but only if **efficiency** can be improved!

**Electronic defects** are one major obstacle

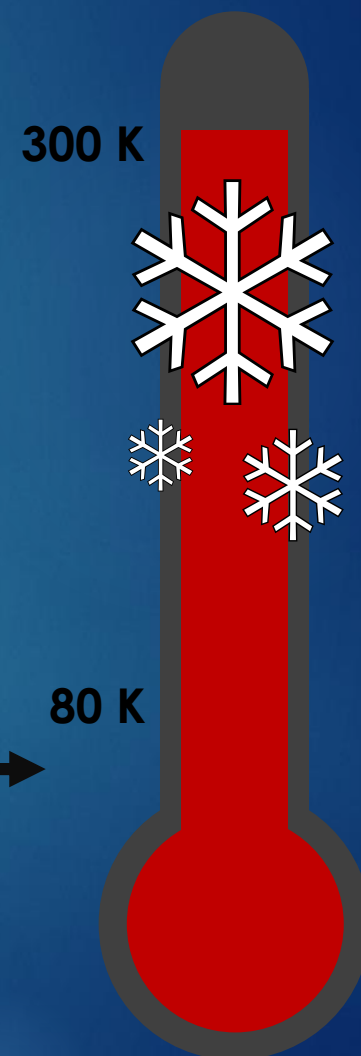
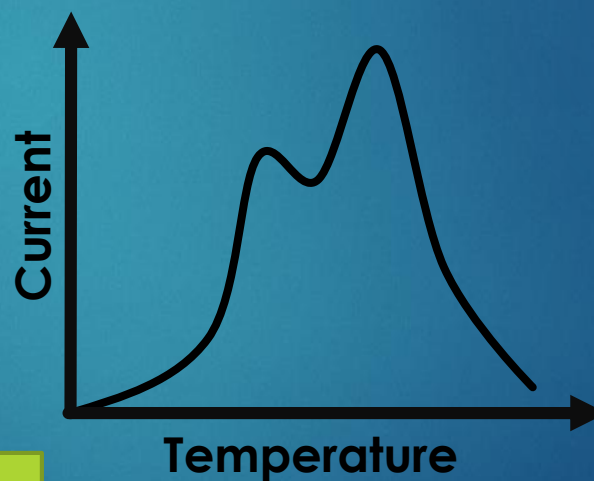
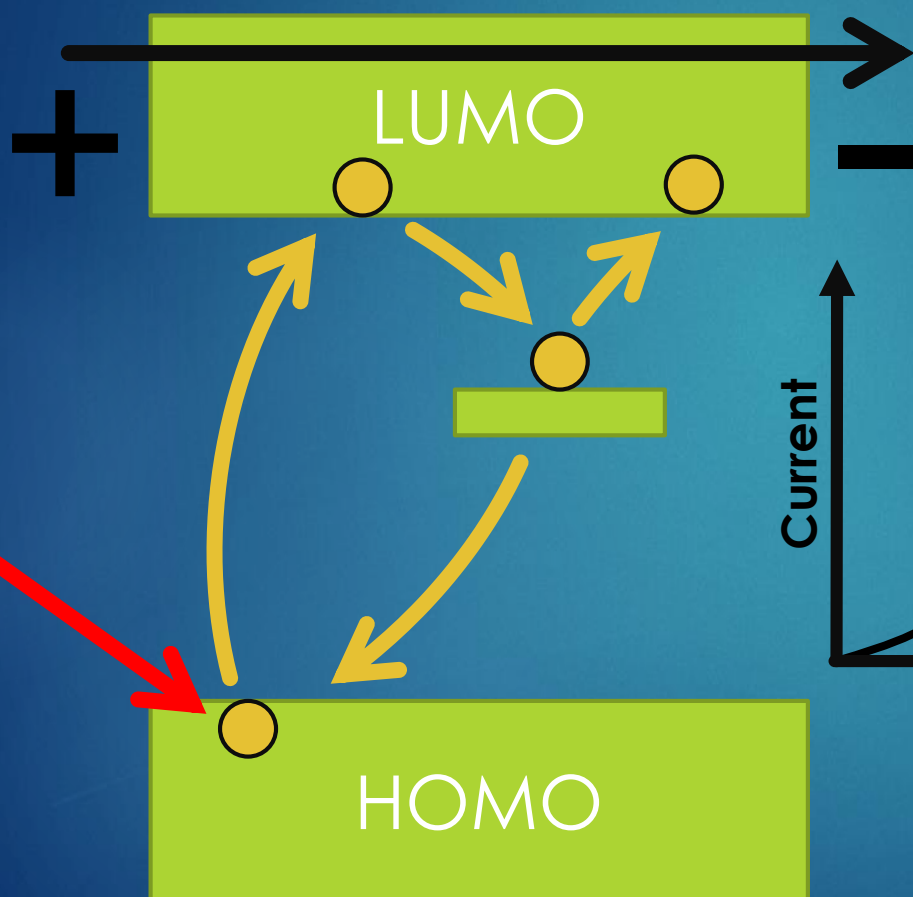
The method of **Thermally Stimulated Currents (TSC)** can help us examine those defect states...

...but setups are expensive and complicated!

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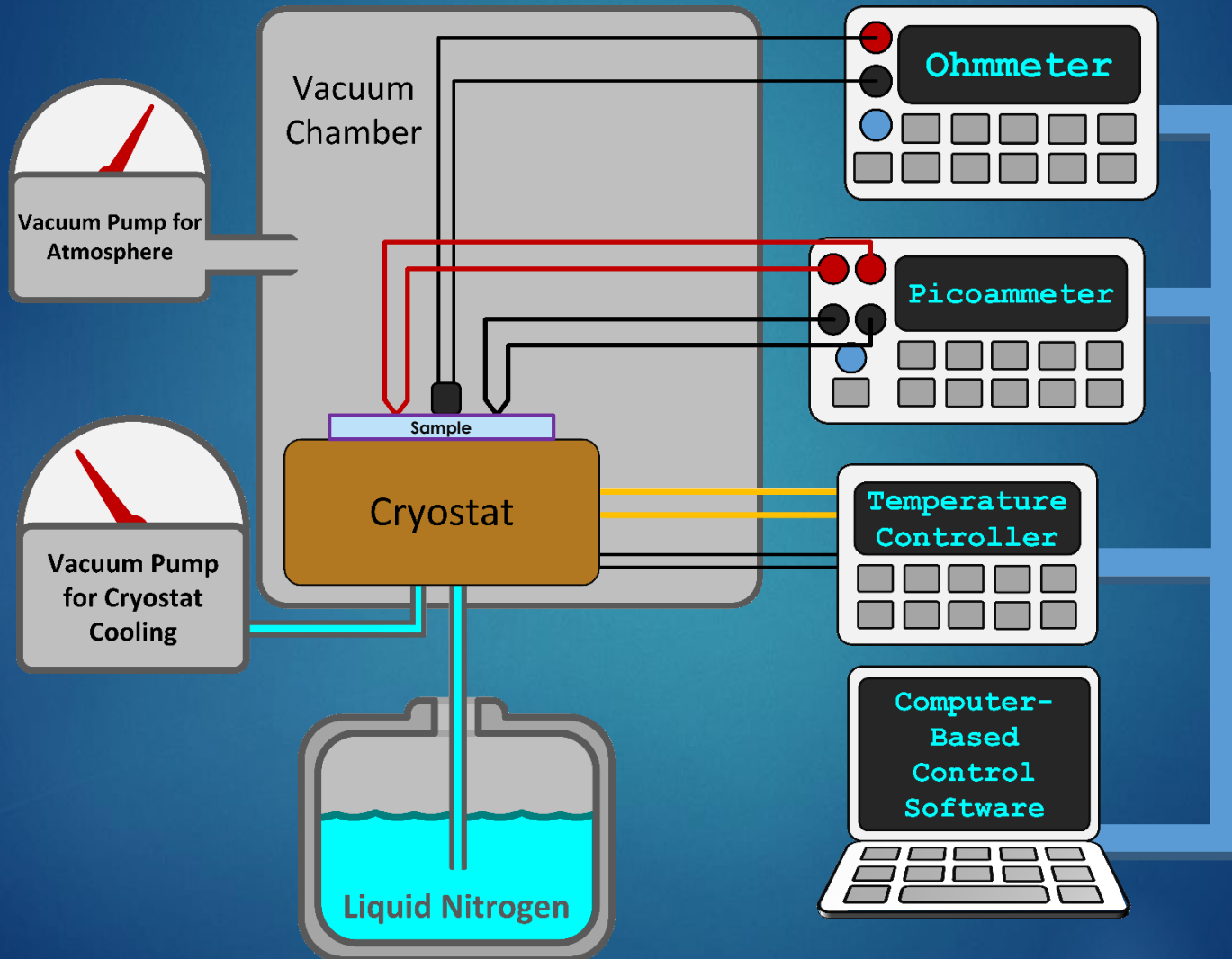
**Our project is to design and build a system that can simplify TSC measurements for our client.**

# Thermally Stimulated Current Measurement



# System Overview

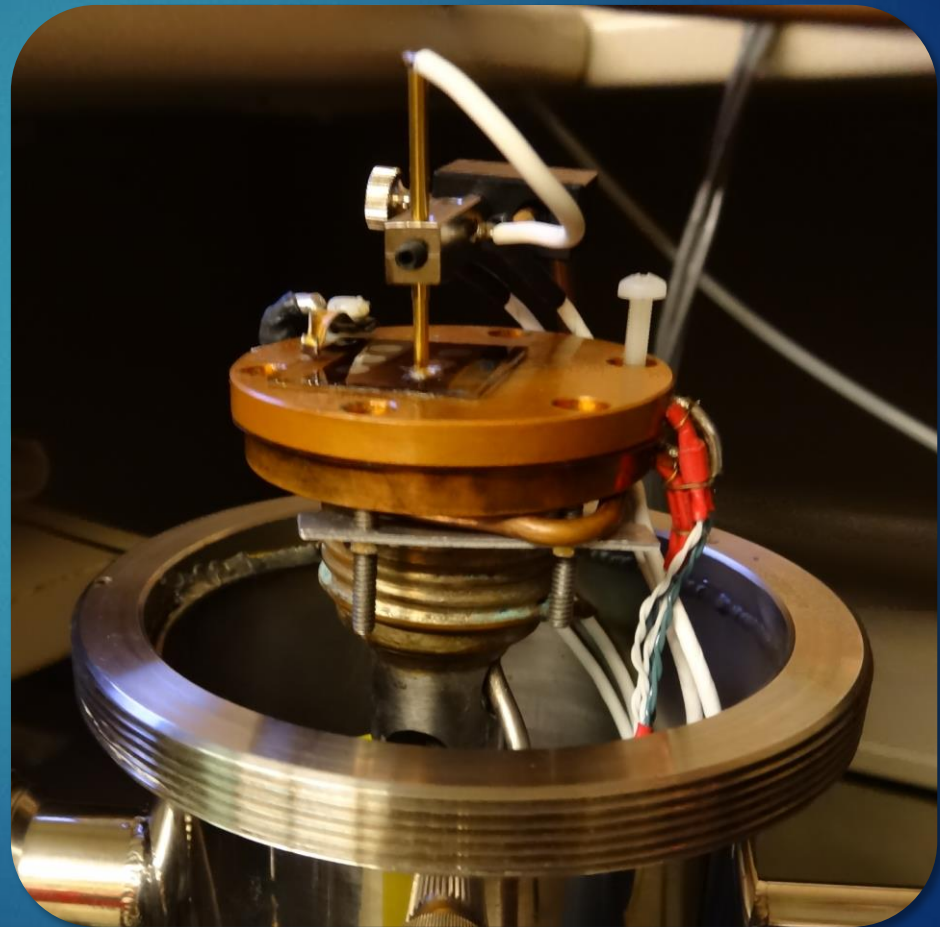
5





# System Overview

6



# Project Goals

7

## 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



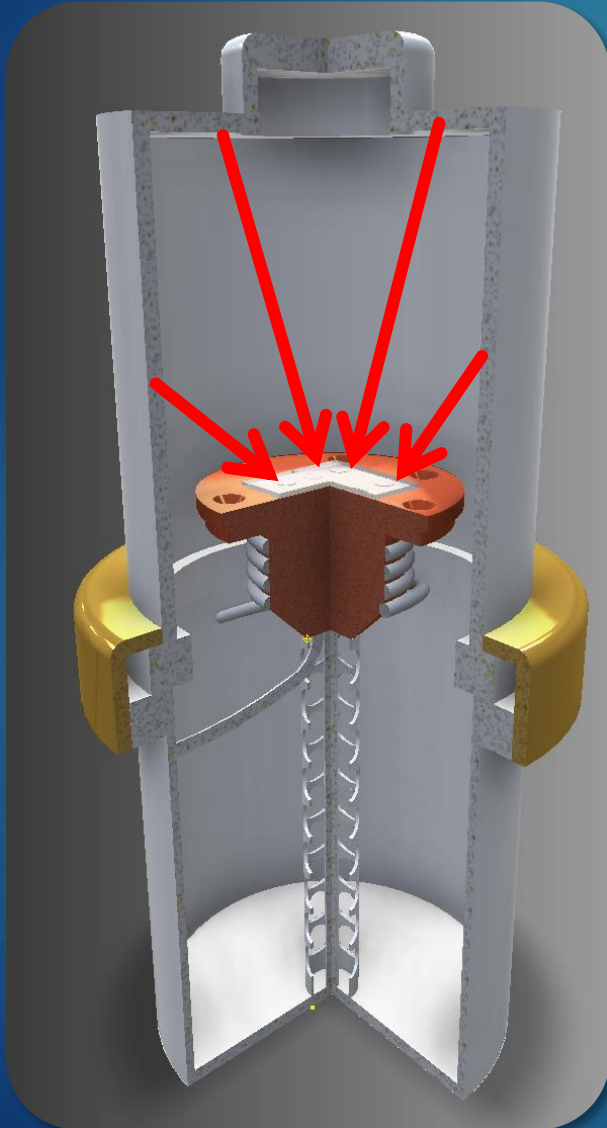
# Project Implementation

HOW WE HAVE SOLVED THE PROBLEM

# Project Schedule

ID	Task Name	2013				2014			
		Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr
1	Research	■							
2	Part Acquisition		■						
3	Testing new parts		■						
4	Preliminary Runs			■					
5	Software Design				■				
6	System Assembly					■			
7	Full system tests						■		
8	Troubleshooting							■	

# Cold Shroud

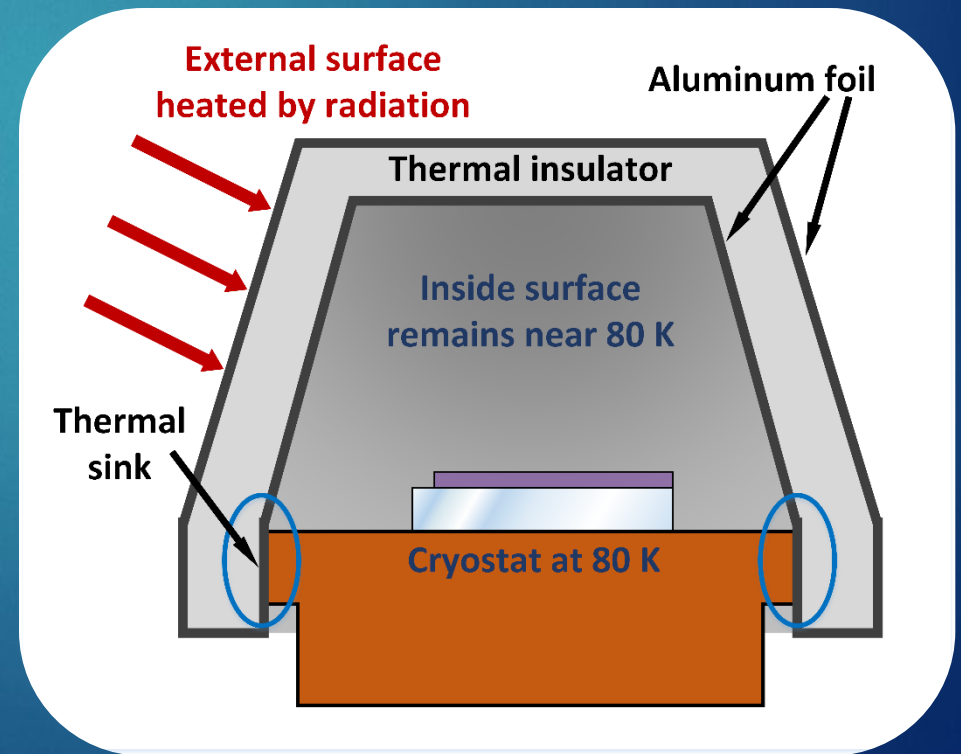
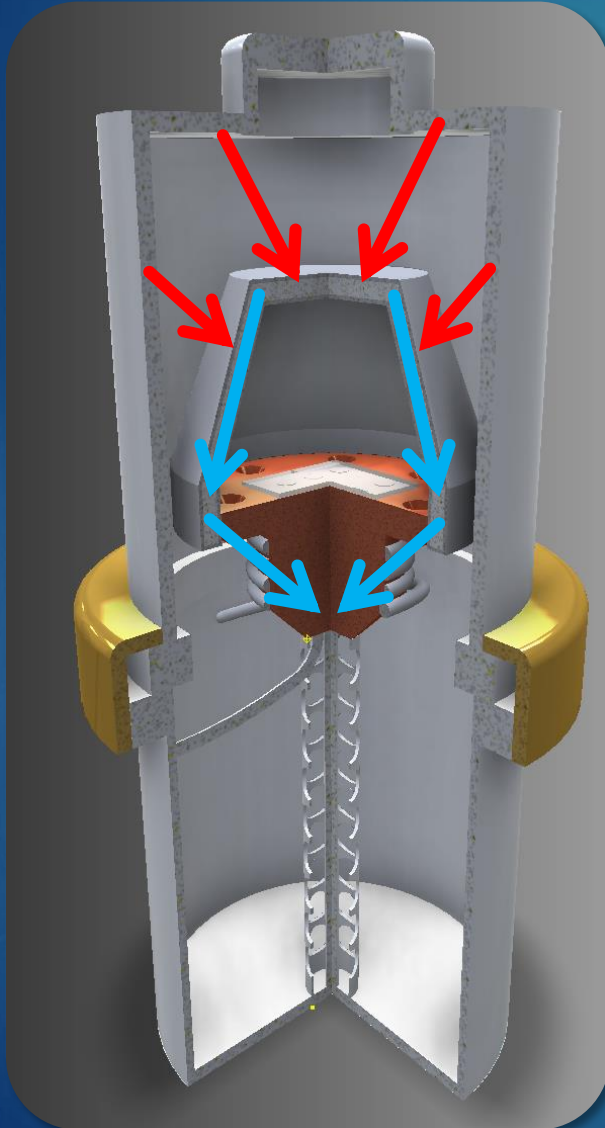


- ▶ Radiative heating is one of the largest problems
- ▶ Solution: mask with a reflective, cold surface

# Cold Shroud

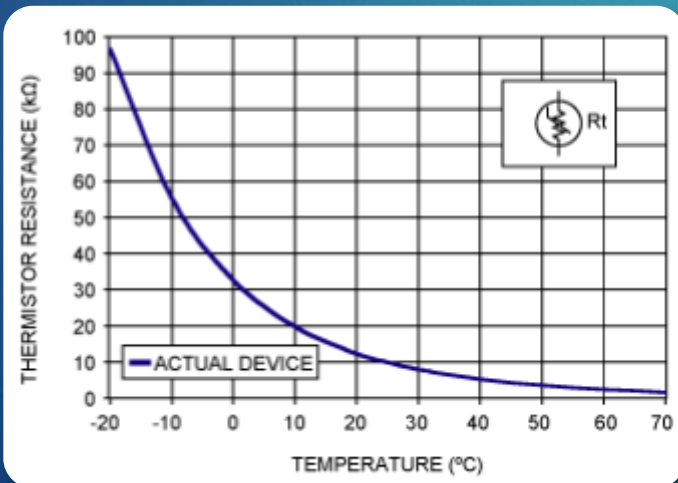
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- ▶ Radiative heating is one of the largest problems
- ▶ Solution: mask with a reflective, cold surface

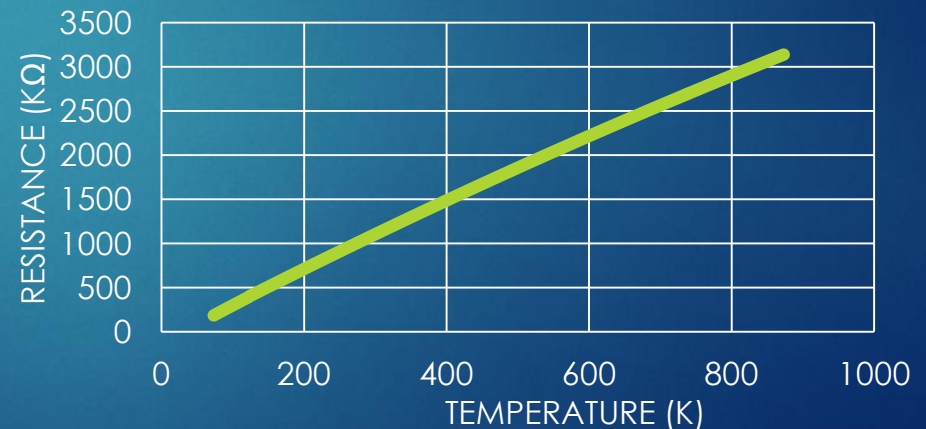


# Temperature Sensors

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



### Platinum RTD

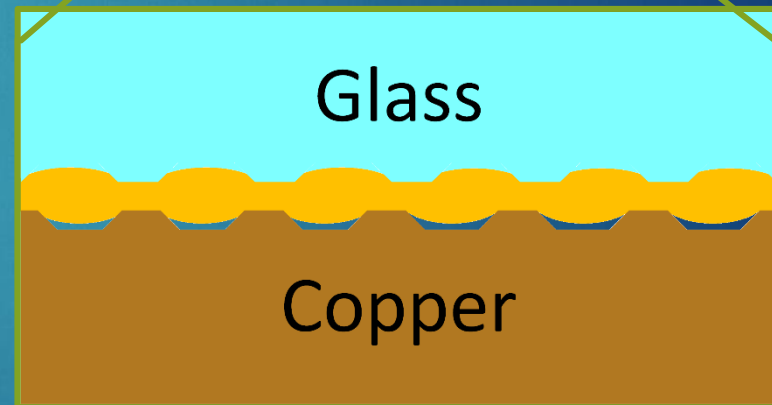
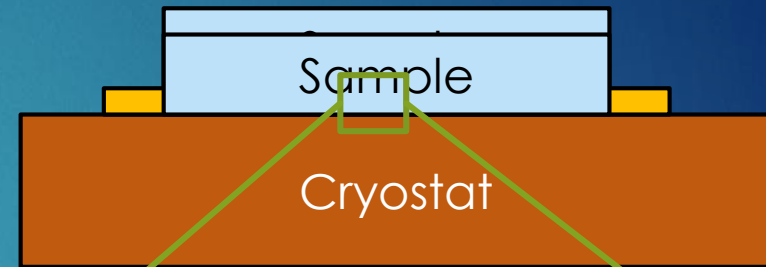




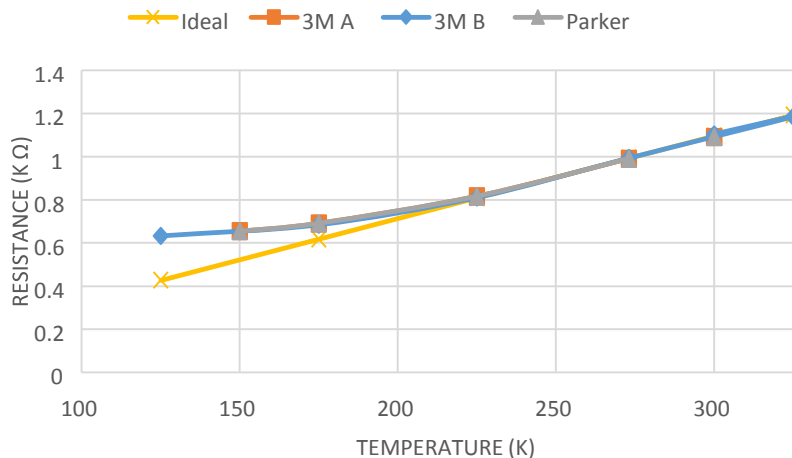
# Thermal Interface Materials

14

- ▶ Conformable Pads
  - ▶ Parker Chomerics
  - ▶ 3M



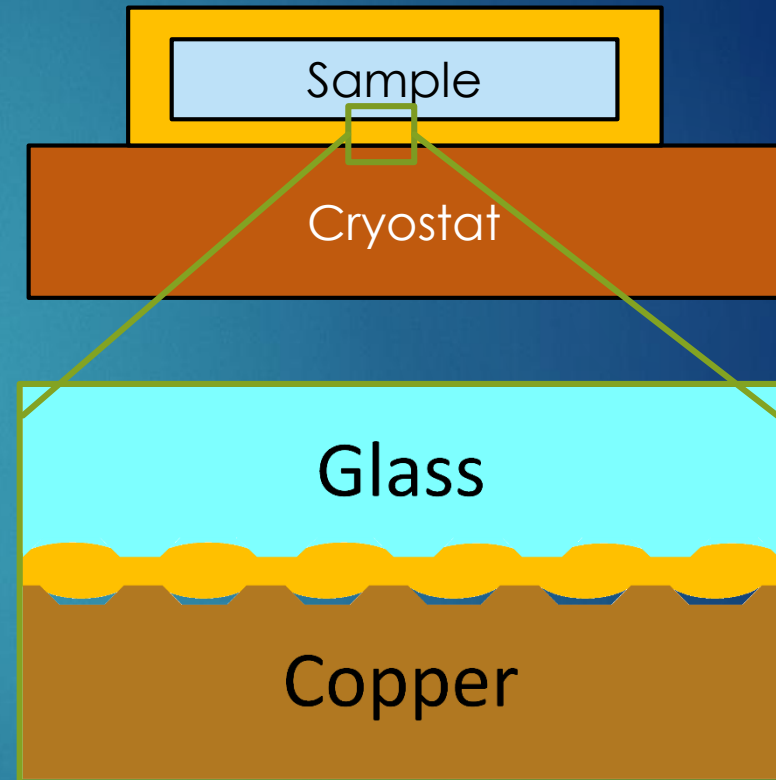
### THERMAL PAD FREEZEOUT



# Thermal Interface Materials

15

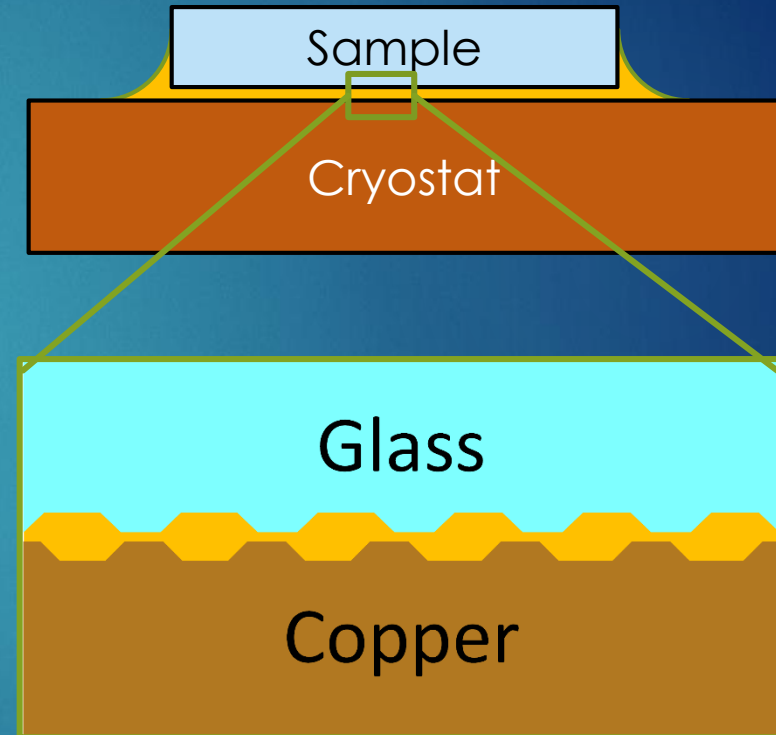
- ▶ Conformable Pads
  - ▶ Parker Chomerics
  - ▶ 3M
- ▶ Cryogenic Epoxy
  - ▶ Stycast 1266
  - ▶ Reacted with organic layer!



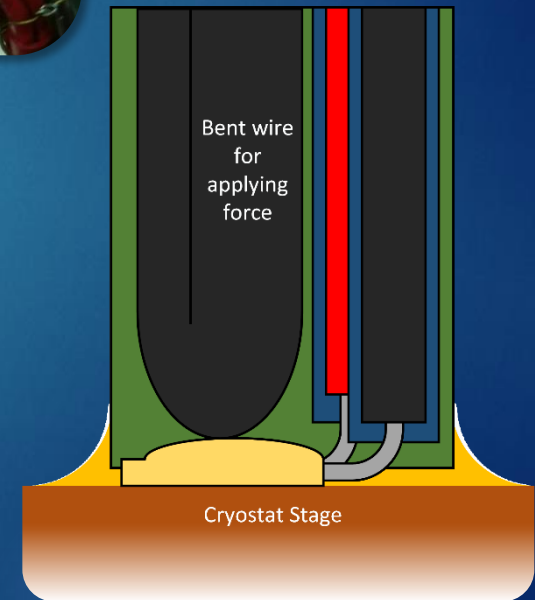
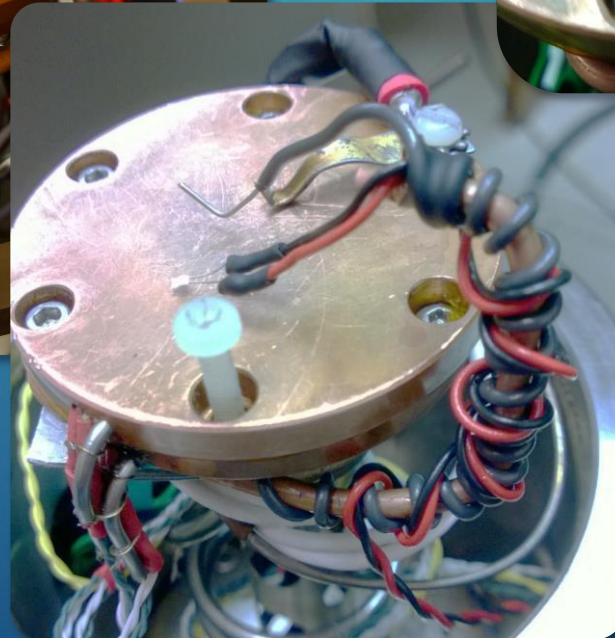
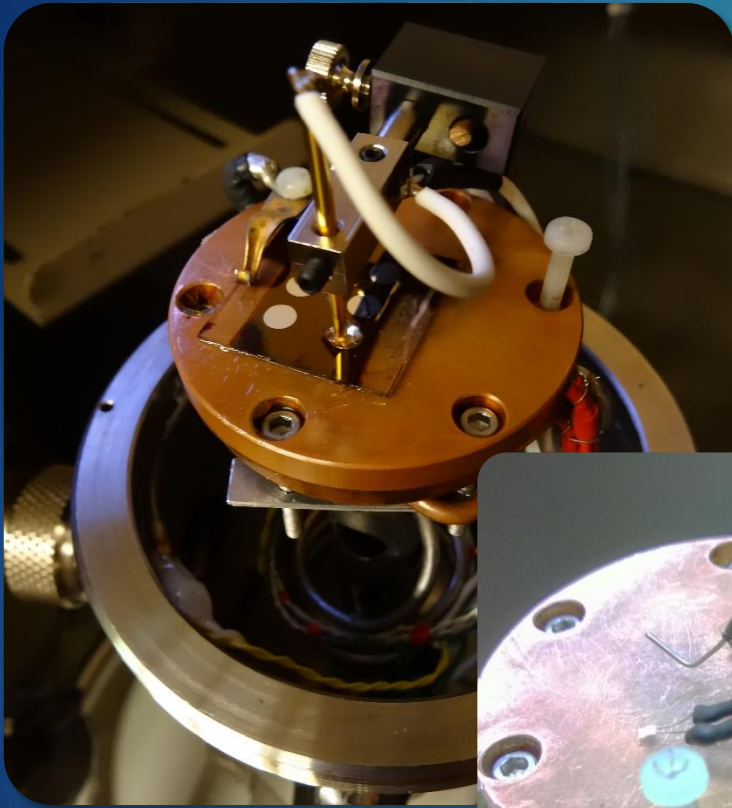
# Thermal Interface Materials

16

- ▶ Conformable Pads
  - ▶ Parker Chomerics
  - ▶ 3M
- ▶ Cryogenic Epoxy
  - ▶ Stycast 1266
- ▶ Cryogenic Vacuum Grease
  - ▶ Apiezon N Grease
- ▶ Cryogenic Varnish
  - ▶ Lakeshore VGE-7031



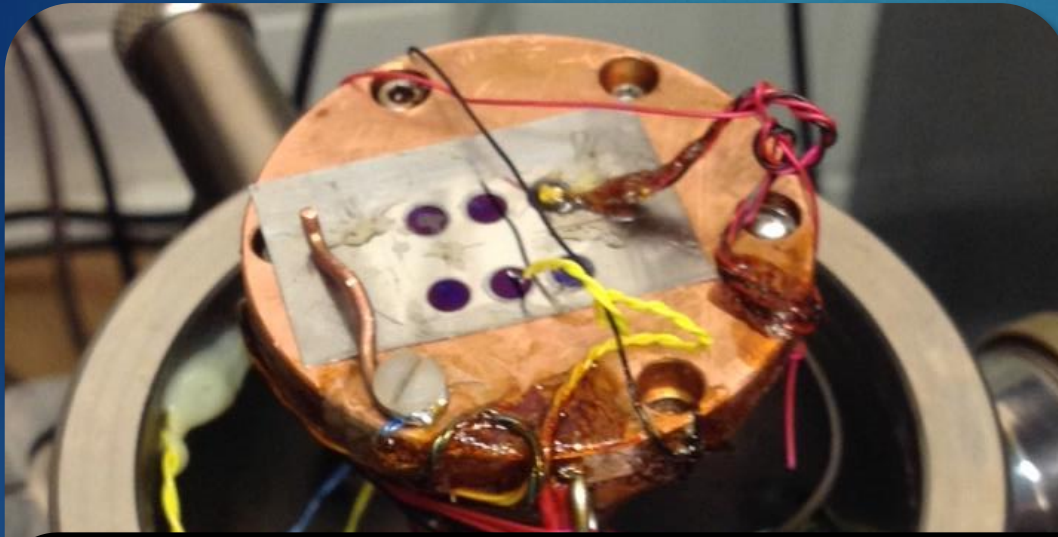
# Probe Effects



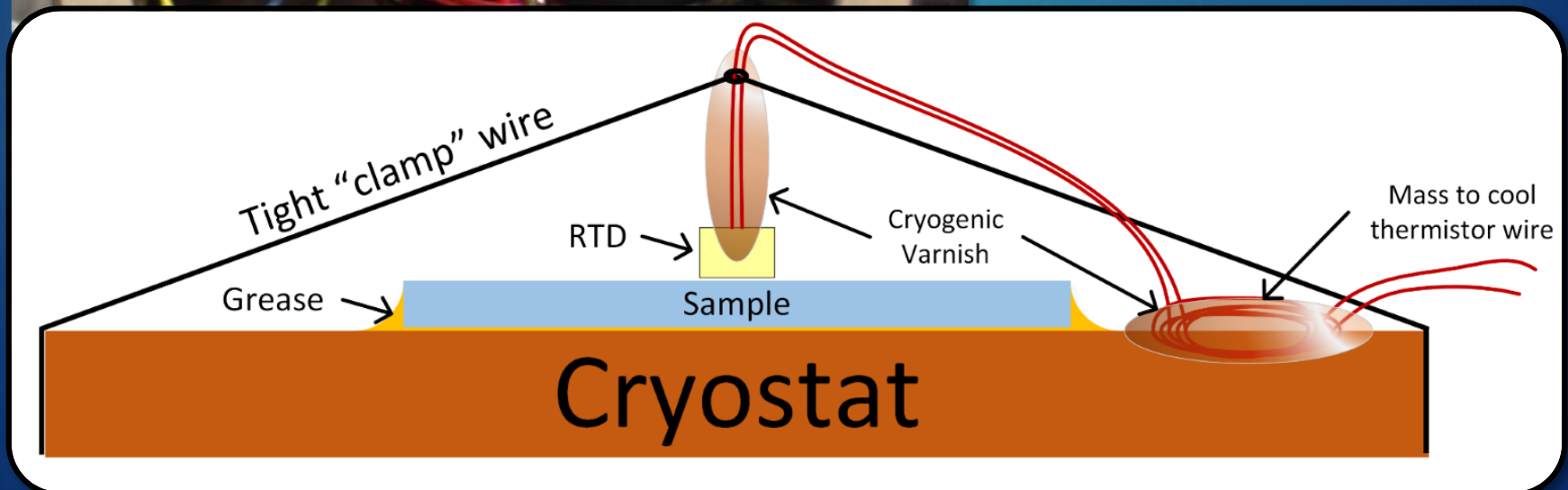


# Probe Effects

18



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





# Automation Software

19

## Flexible

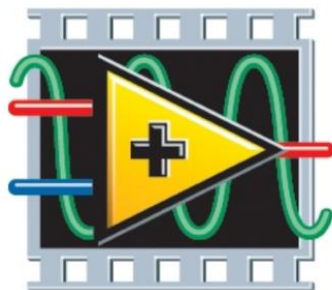
- Allow for experiment customization
- Support multiple modes

## Robust

- Elegantly handle problems
- Keep user safe

## Accessible

- Intuitive GUI
- Legible output




NATIONAL INSTRUMENTS

LabVIEW™

# Automation Software

20

## Thermally Stimulated Current Dashboard

Export Results to:   Lakeshore GPIB Keithley GPIB

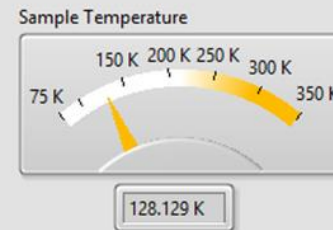
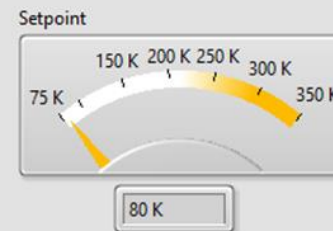
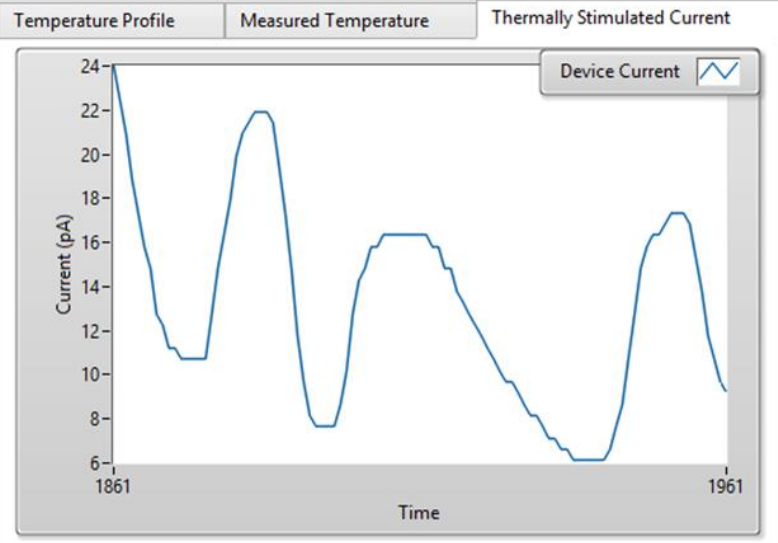
Choose Profile

Excitation Length

Excitation Amplitude

Ramp Rate

Target Temperature



 **START**

Experiment Started

7:32:52.430 PM  
12/9/2013

 **ABORT**

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

Thermally Stimulated Current



# Conclusion

21

- ▶ 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

22

- ▶ 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

25

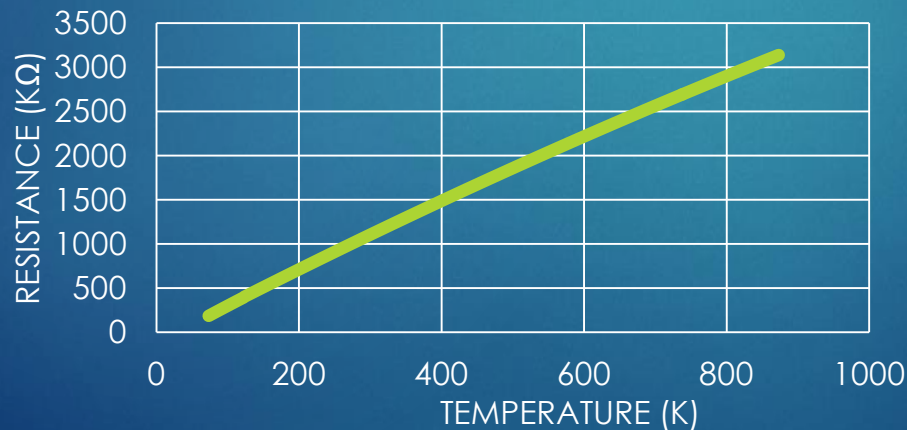
- ▶ T. Benanti & D. Venkataraman. "Organic solar cels: An overview focusing on active layer morphology". *Photosynthesis Research*, Vol. 87, 2006, pp. 73-81.
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# US Sensor PPG102A6

26

- ▶  $1000 \Omega \pm 0.06\%$
- ▶ Range:  $-200^{\circ}\text{C}$  to  $+600^{\circ}\text{C}$  (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

27

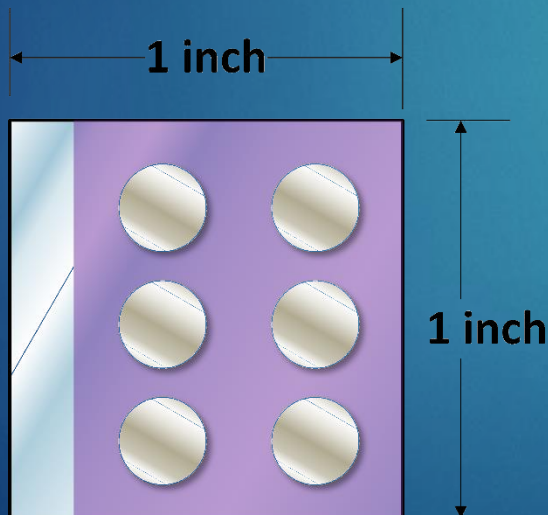
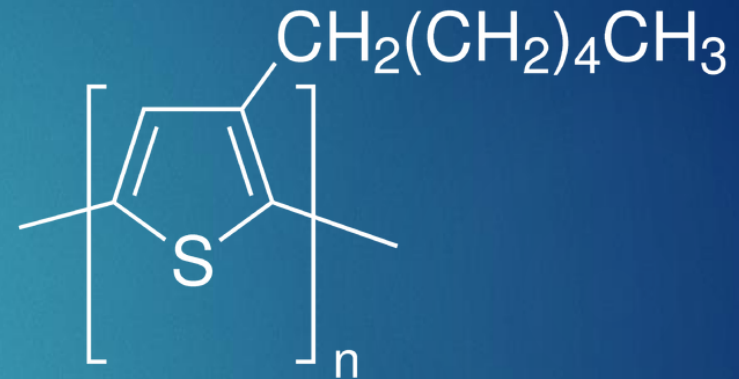
- ▶ Temperature Range: 0.15 K to 300 K
- ▶ Thermal Conductivity (80 K) =  $0.1 \text{ W m}^{-1} \text{ K}^{-1}$
- ▶ Vapor Pressure (273 K) =  $2.67 \times 10^{-7} \text{ torr}$
- ▶ Volume Resistivity  $2 \times 10^{16} \text{ } \Omega \text{ m}$



# Solar Cell Samples

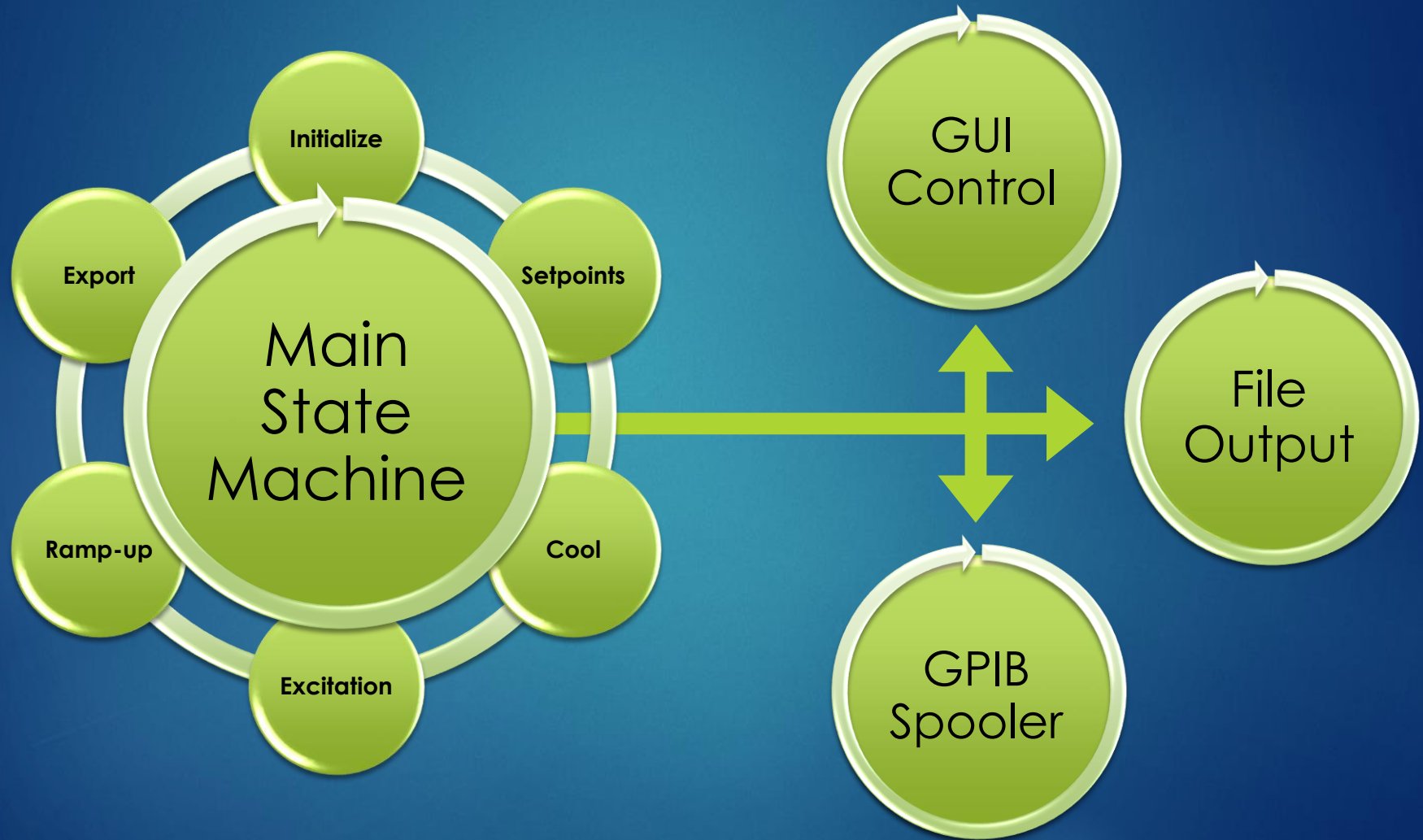
28

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





# Software Architecture



# 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

31

- ▶ 5½ digit display
- ▶ Resolution: 10 fA
- ▶ 1000 reads per second
- ▶ Accuracy:  $\pm 0.4\%$
- ▶ Coaxial hookups, triax adapters for low currents
- ▶ \$1,660 (with educational discount)



# VGE-7031 Cryogenic Varnish

32

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

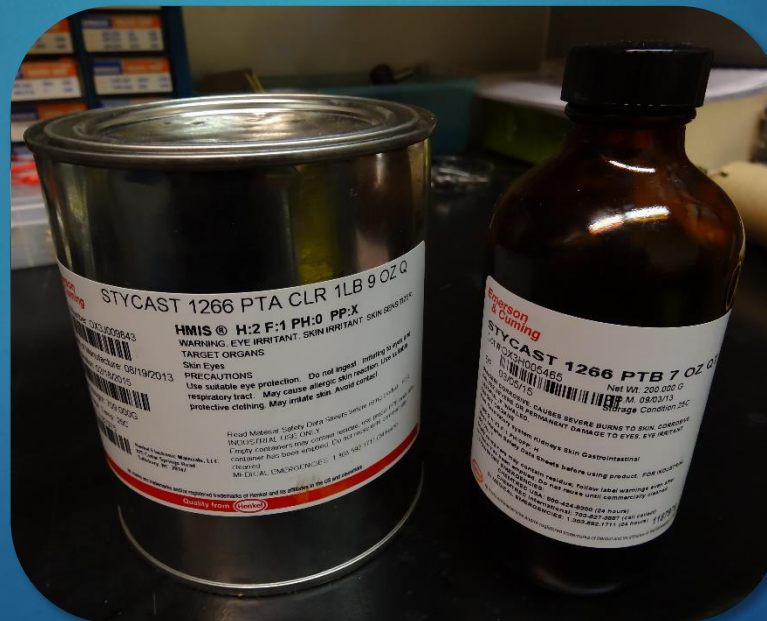




# Stycast 1266 Cryogenic Epoxy

33

- ▶ 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

34

- ▶ 3M:
  - ▶ 5519S, 5591S
- ▶ Parker Chomerics:
  - ▶ Therm-A-Gap G579
- ▶ All three highly conformable, slightly tacky, electrically insulating
- ▶ Thermal conductivity (300K)  
 $1-3 \text{ Wm}^{-1}\text{K}^{-1}$

