ISU ECpE Senior Design Group

MAY14-03

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

Design and implementation of a cryogenic electrical characterization system for organic electronic devices

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

Project Description

MOTIVATION, GOALS, AND OVERVIEW

ORGANIC ELECTRONIC DEVICES

Promising new technologies…

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

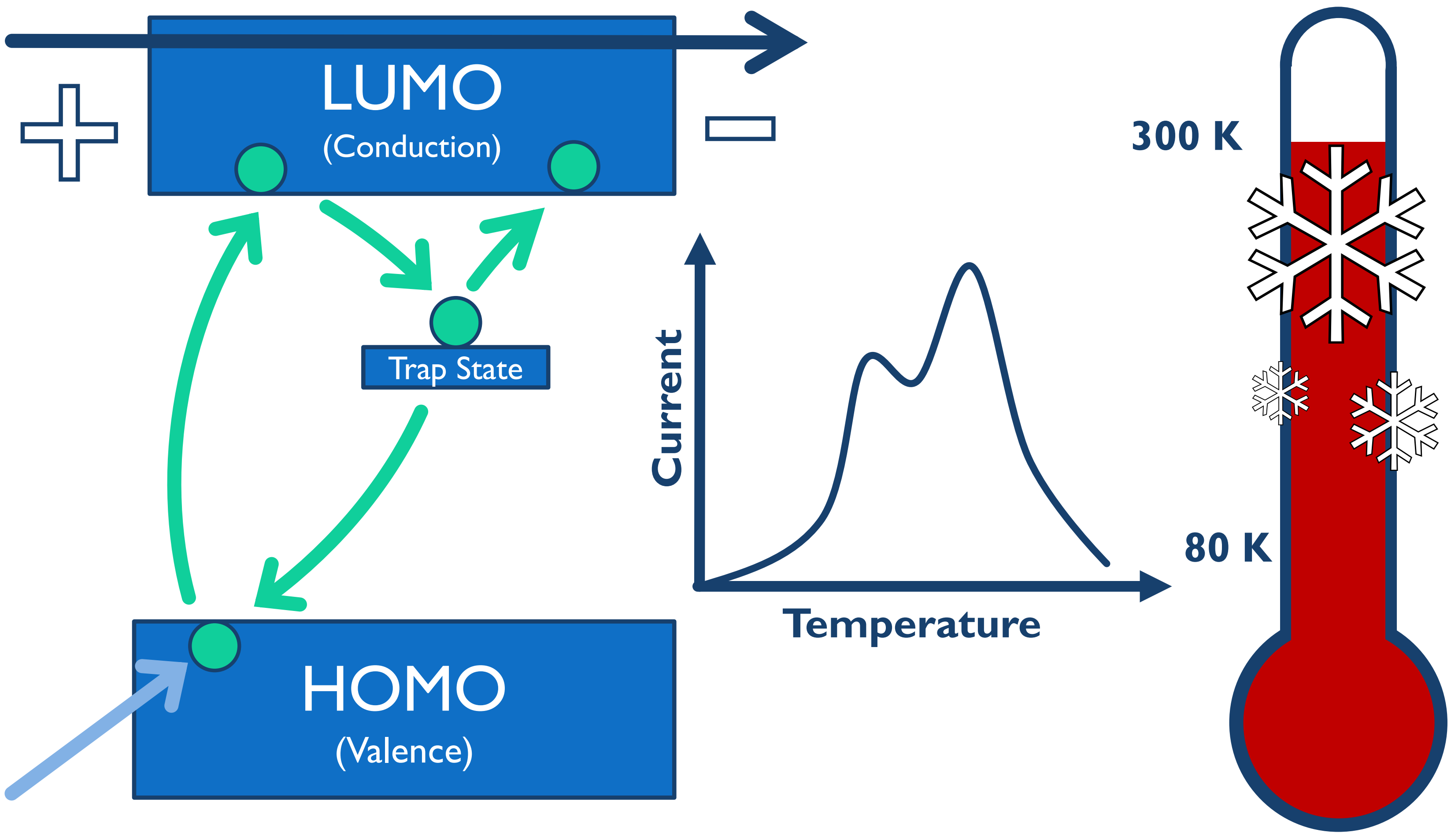
Electronic defects are one major obstacle.

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 simple TSC system for our client.

BRIEF OVERVIEW:
THERMALLY STIMULATED CURRENT MEASUREMENTS



PROJECT GOALS

Where we started

- Liquid nitrogen cryostat and vacuum chamber
- No verification of cell temperature
- 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 COMPARISON AND LITERATURE SURVEY

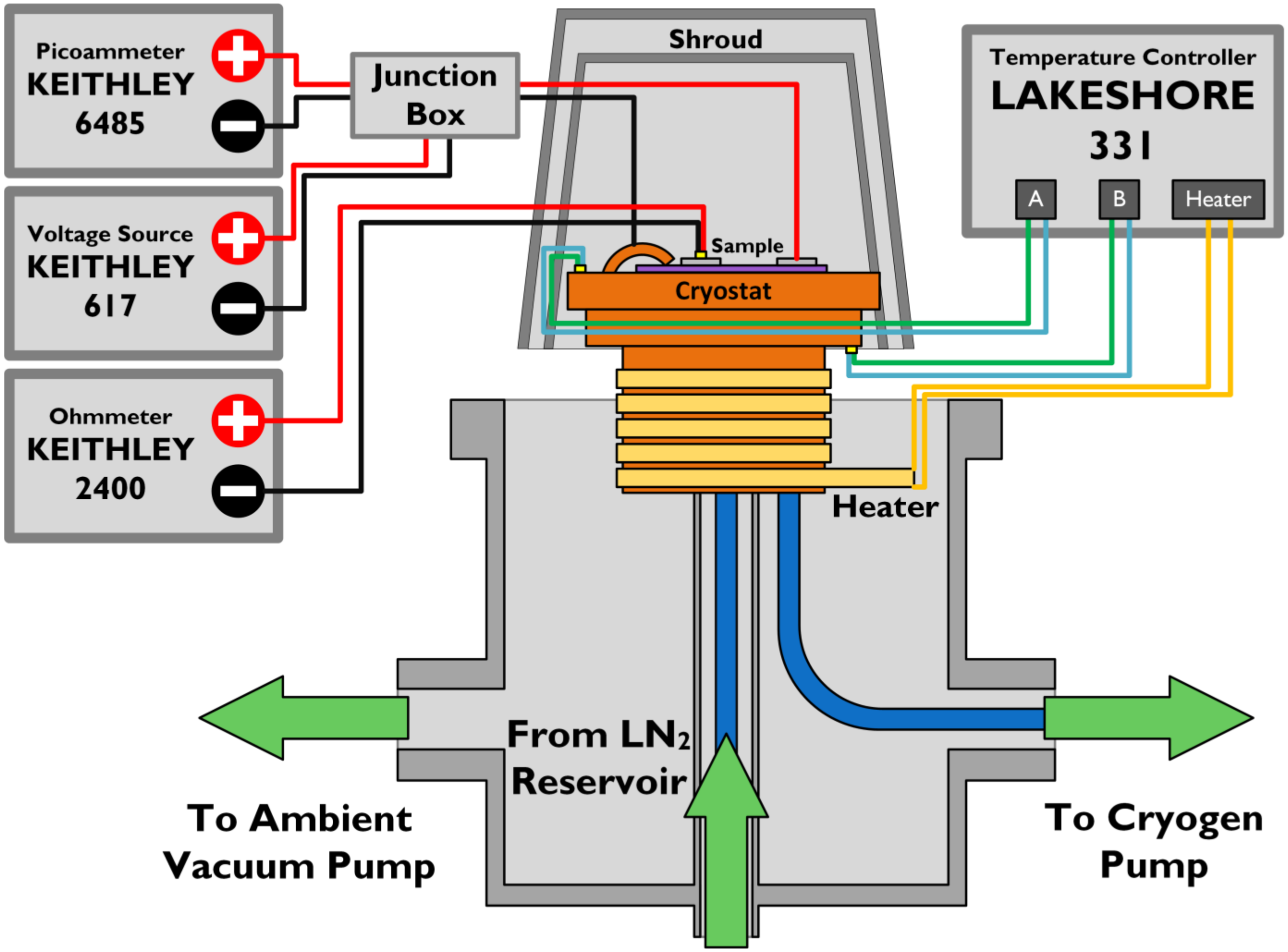
- No dedicated commercial systems for TSC exist!
- High quality cryostats cost $30,000 or more

- Most research papers publish very few implementation details

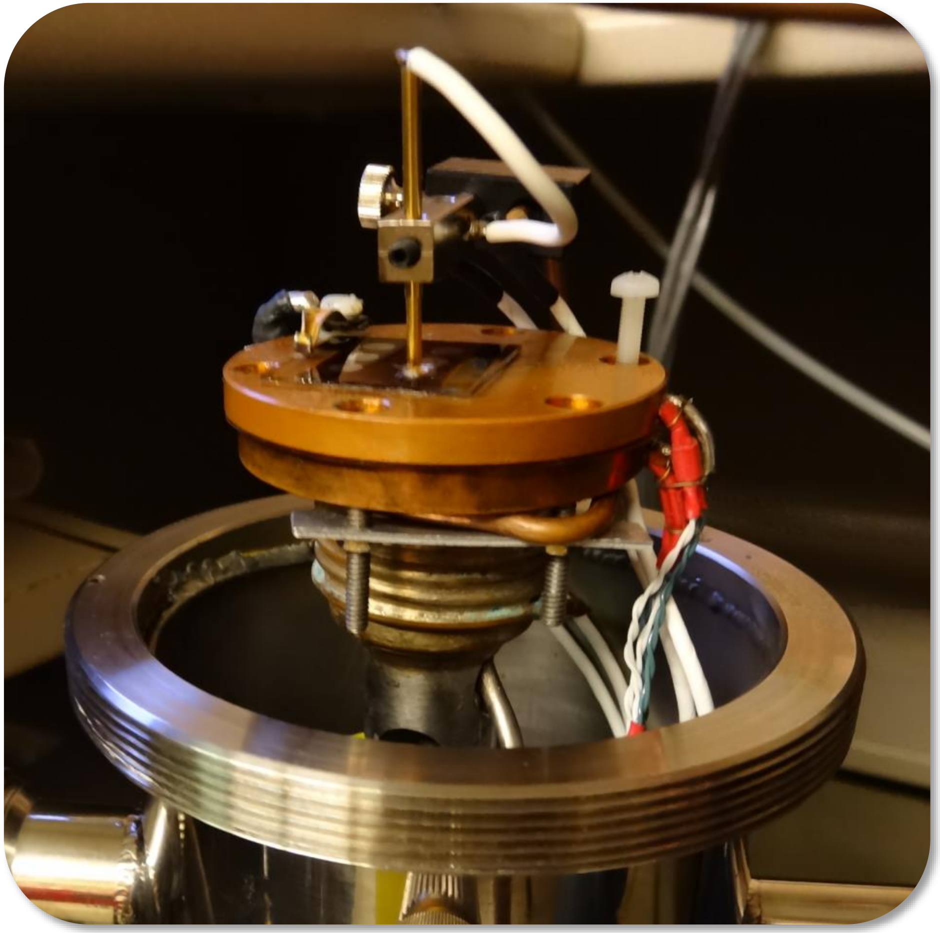
- Spoke with researchers from Ames Lab
  - Significant experience with cryogenic systems

- Compared literature for different components
  - Temperature Sensors
  - Thermal interface materials
  - Insulation
  - Low current measurements

SYSTEM OVERVIEW



SYSTEM OVERVIEW (BEGINNING)

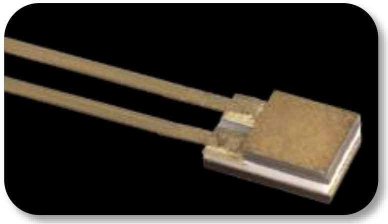


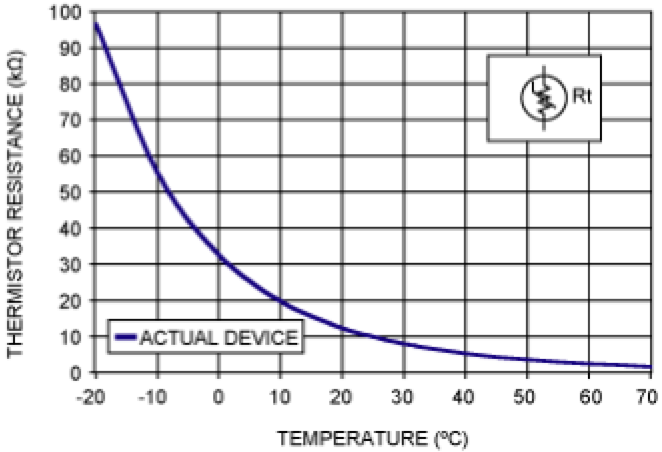
TECHNICAL CHALLENGES

AND THEIR SOLUTIONS

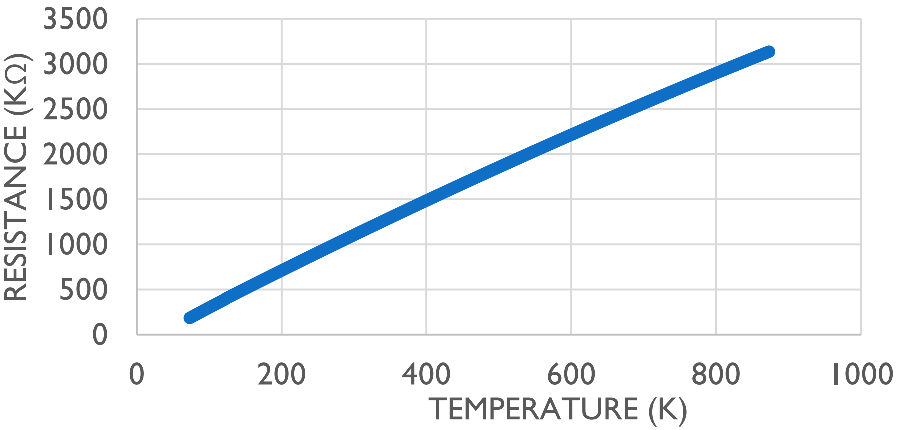
TEMPERATURE SENSORS

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

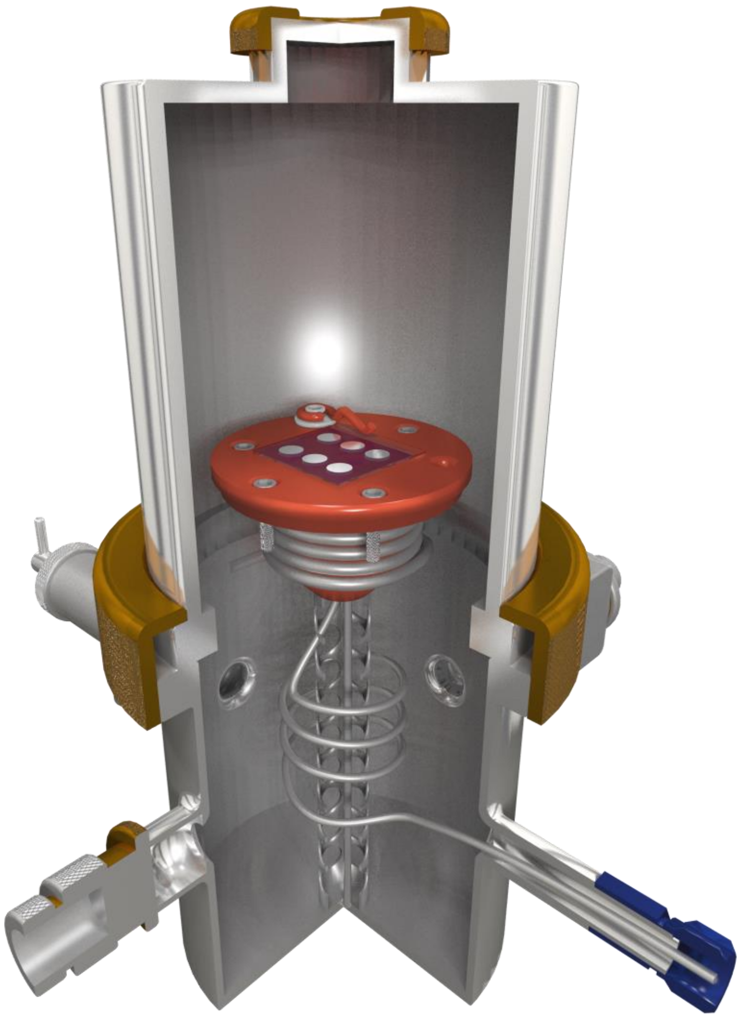




Platinum RTD

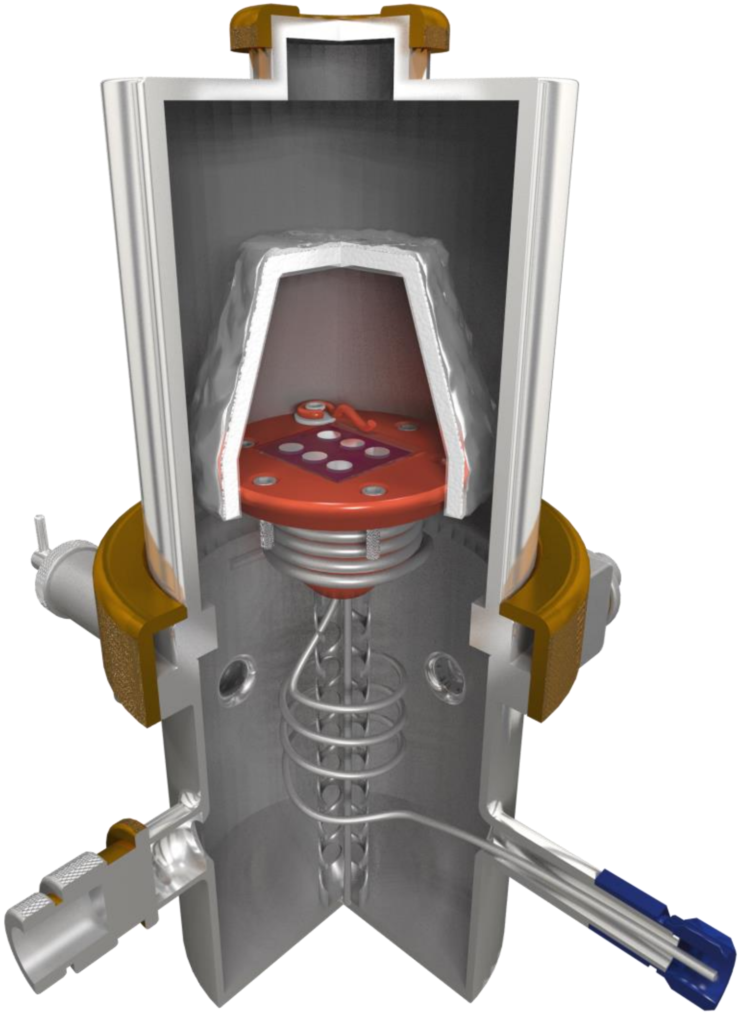


INSULATION



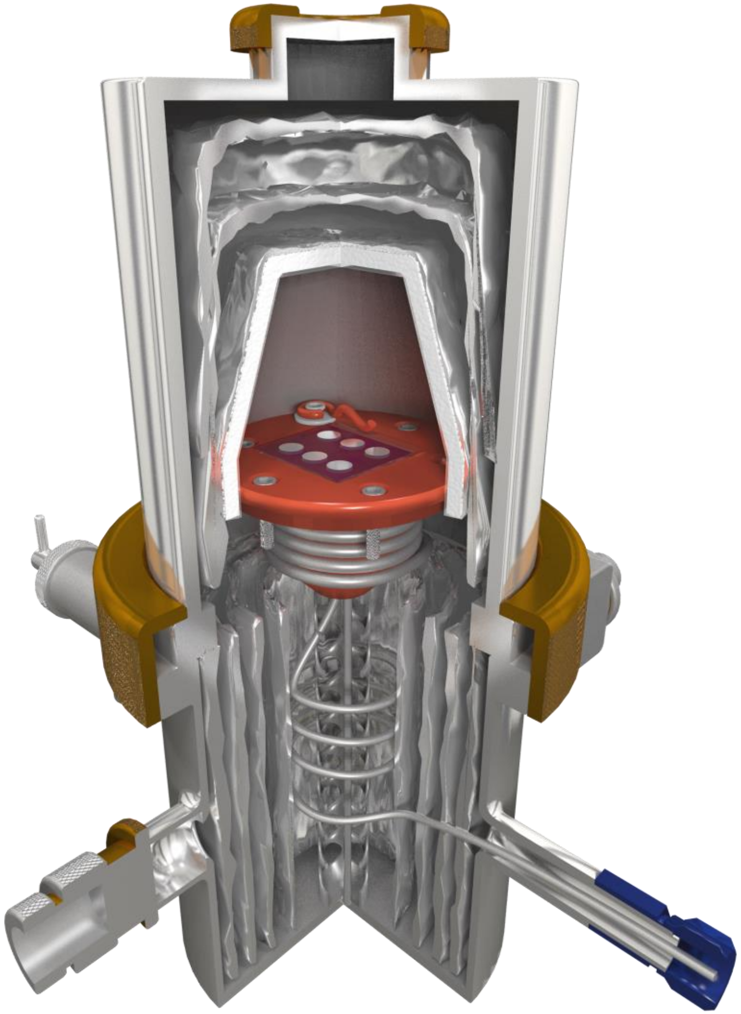
- Radiative heating is one of the largest problems
- Heater efficiency is low: poor temp. control
- Solution: mask with reflective layers

INSULATION



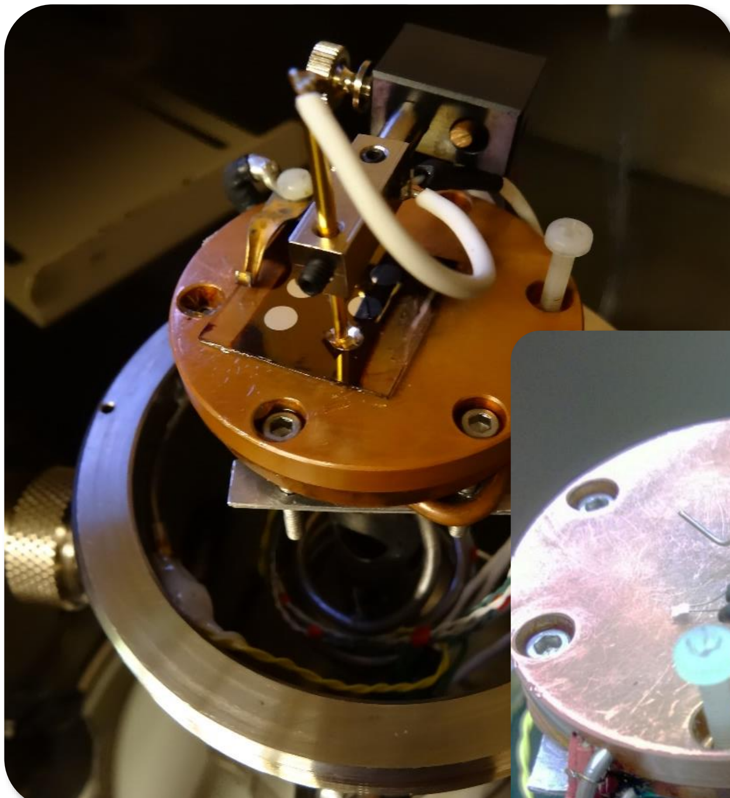
- "Cold shroud" reflects most heat
- Entire inner surface remains cold
- Prototype
  - Aluminum foil over Styrofoam, secured with zip-ties
- Final design
  - Layered aluminized Mylar sewn to Styrofoam with magnet wire

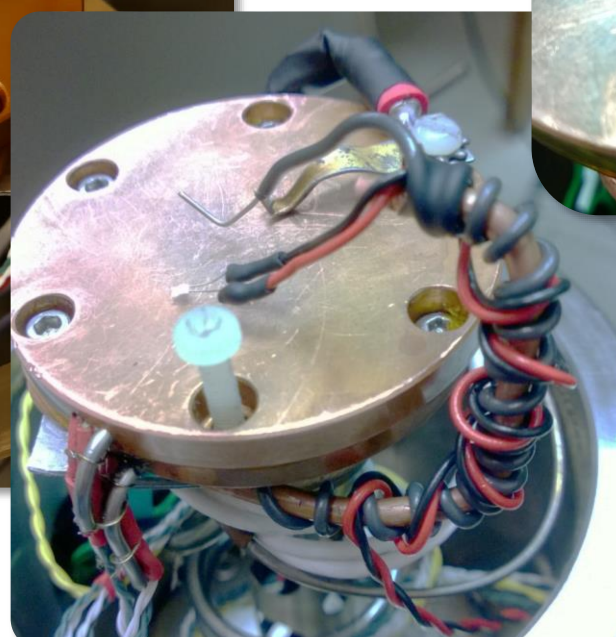
INSULATION

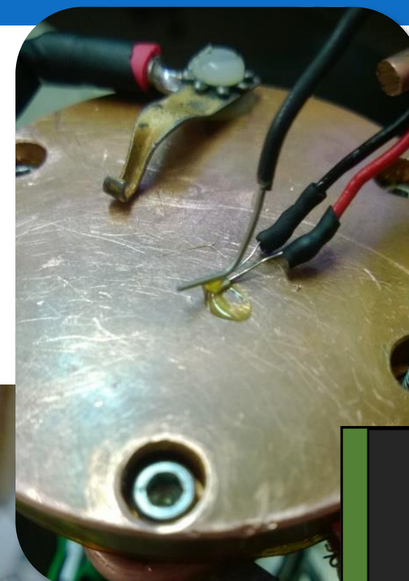


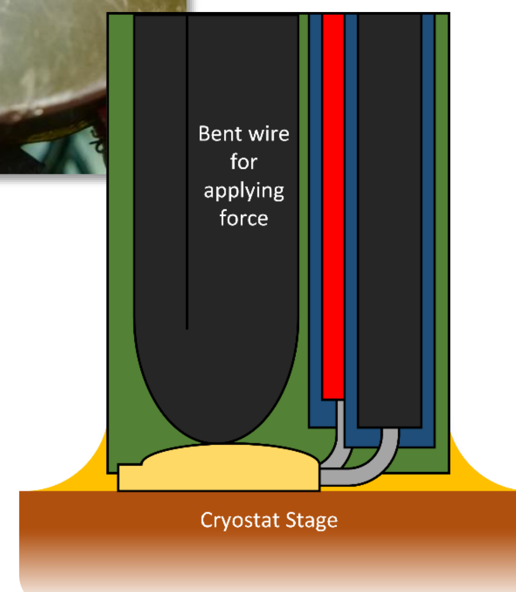
- Packing the remaining space with Mylar improves performance
  - Top Half: Lowers minimum temperature
  - Bottom Half: Increases heater efficiency, improving ramp rate control

PROBE EFFECTS



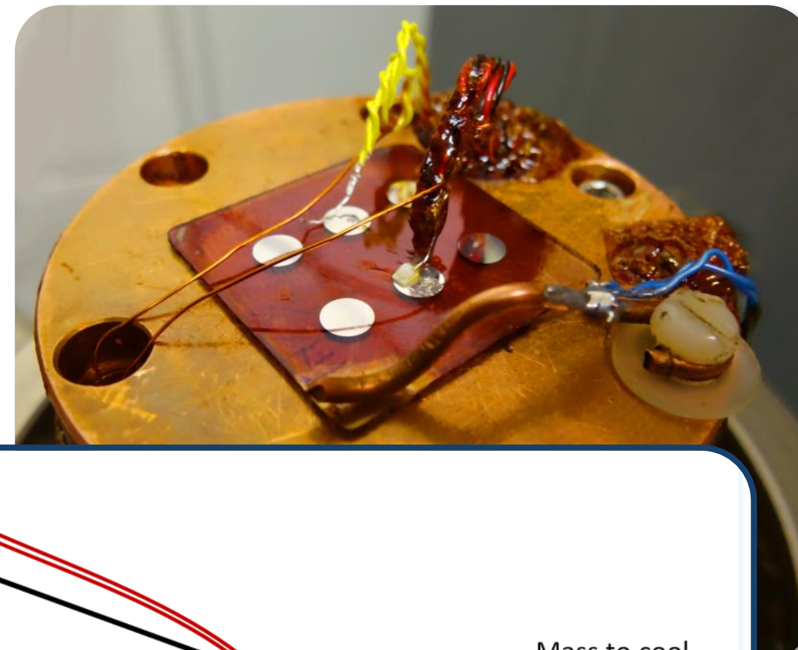


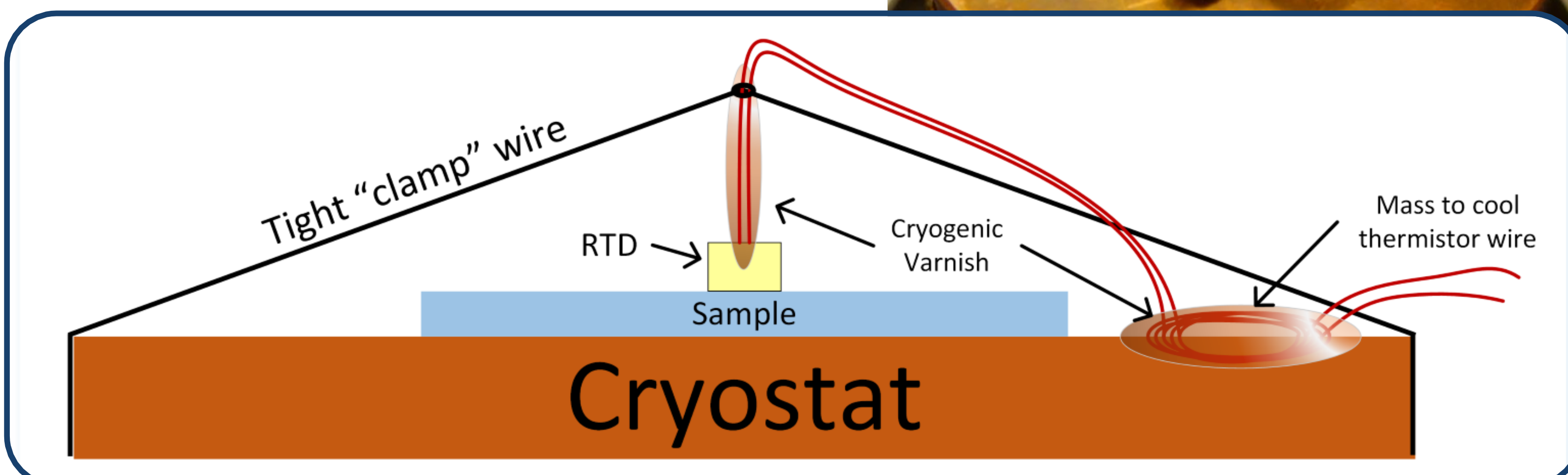




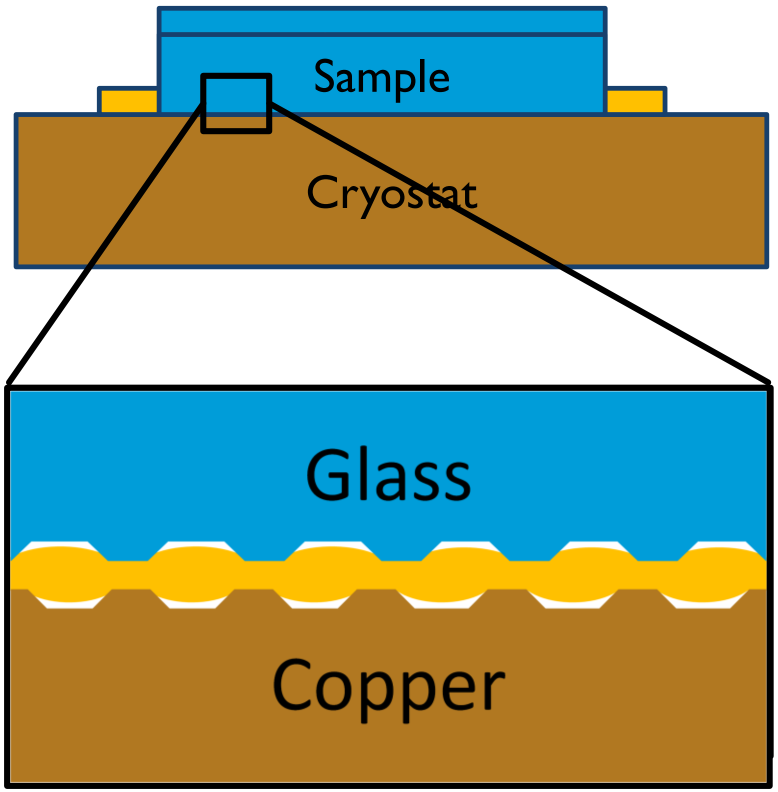
PROBE EFFECTS

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

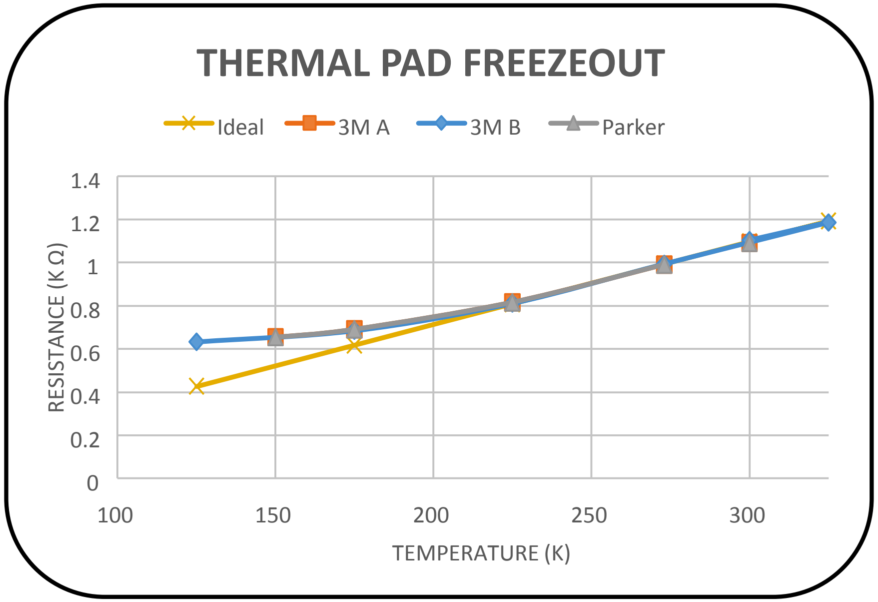




THERMAL INTERFACE MATERIALS



- Conformable Pads
  - Parker Chomerics
  - 3M



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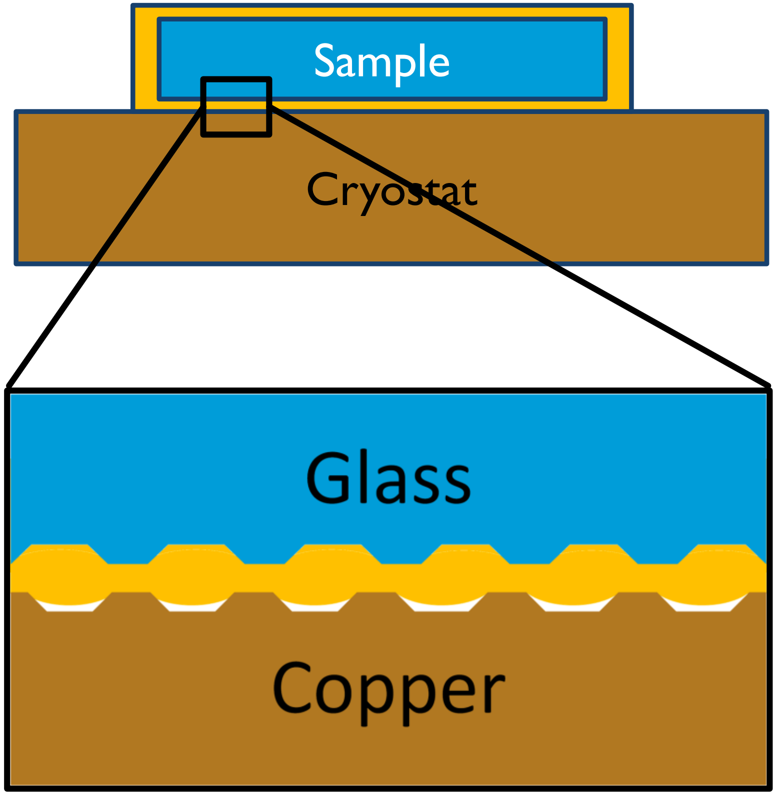
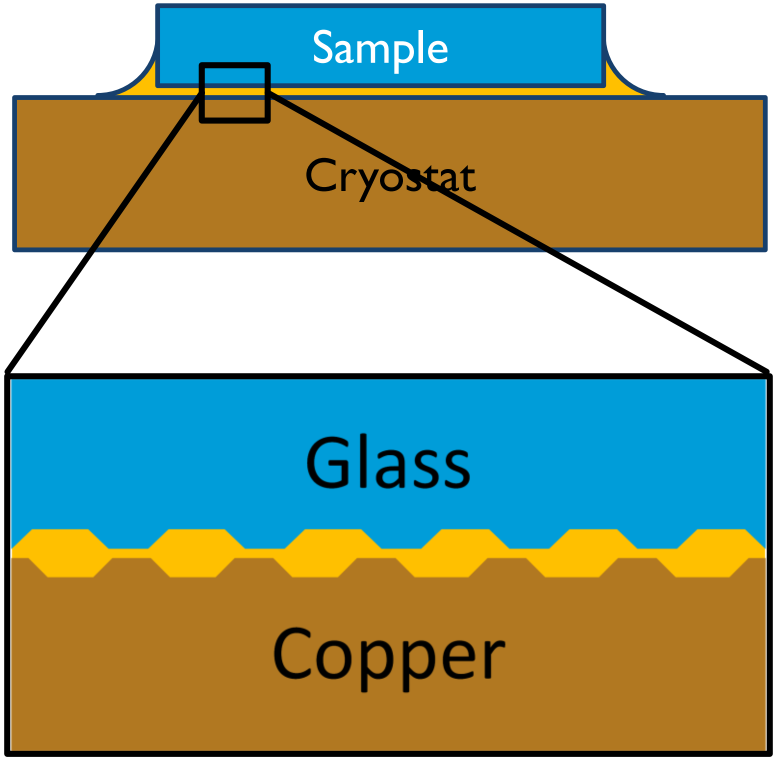


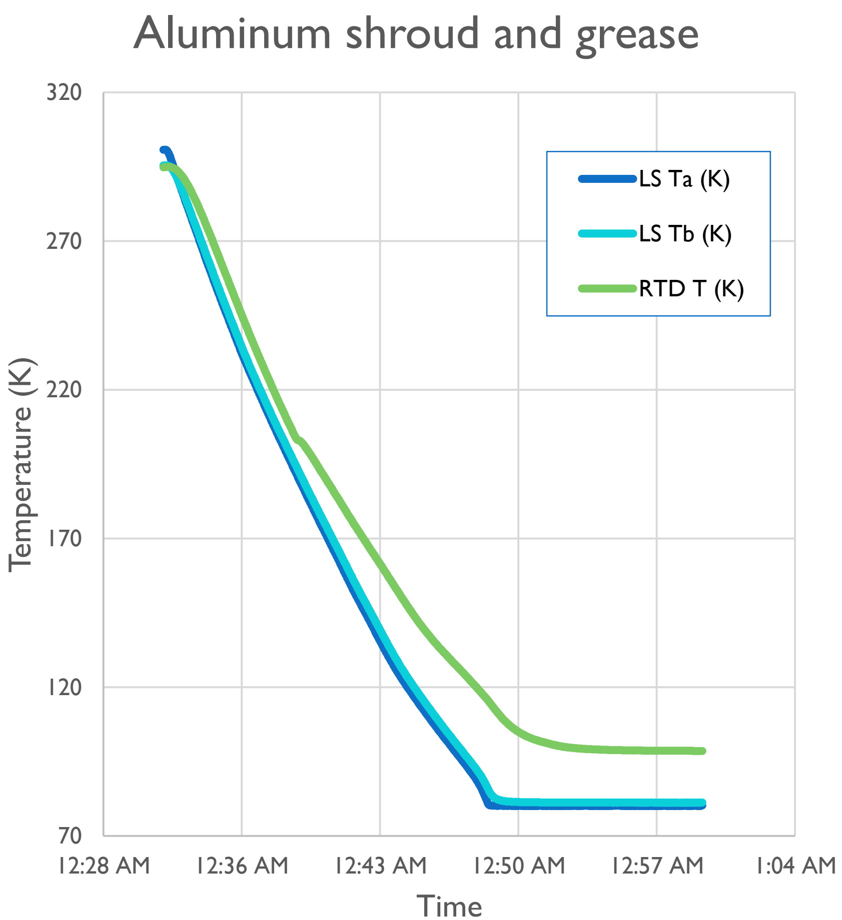
- Conformable Pads
  - Parker Chomerics
  - 3M
- Cryogenic Epoxy
  - Stycast 1266
  - Reacted with organic layer!

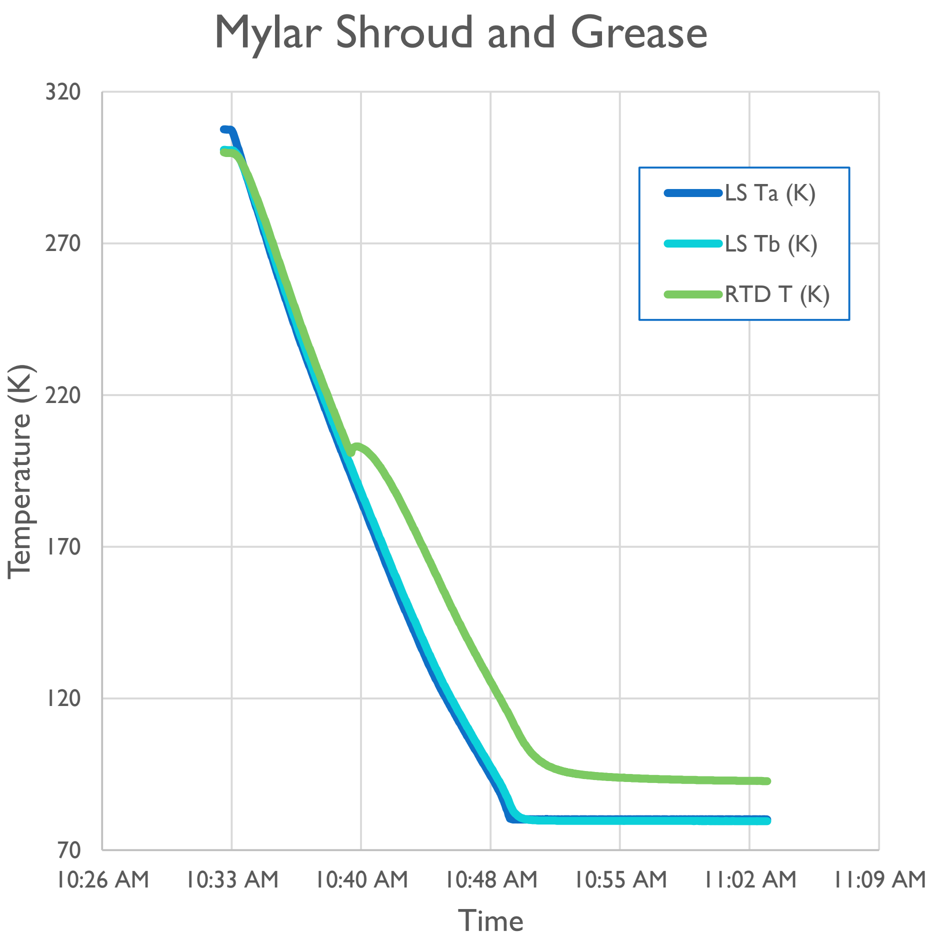
THERMAL INTERFACE MATERIALS



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

THERMAL INTERFACE MATERIALS





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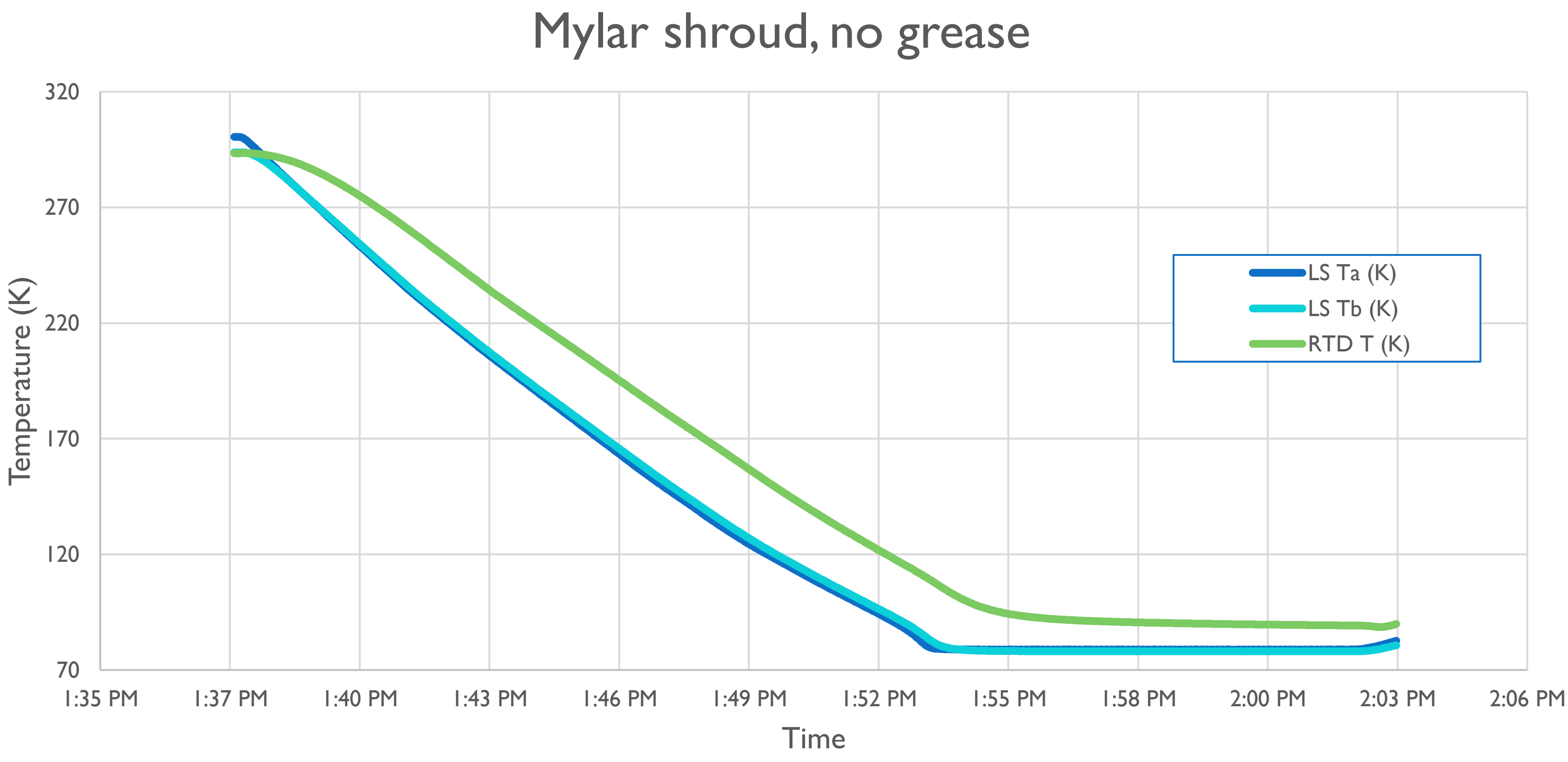
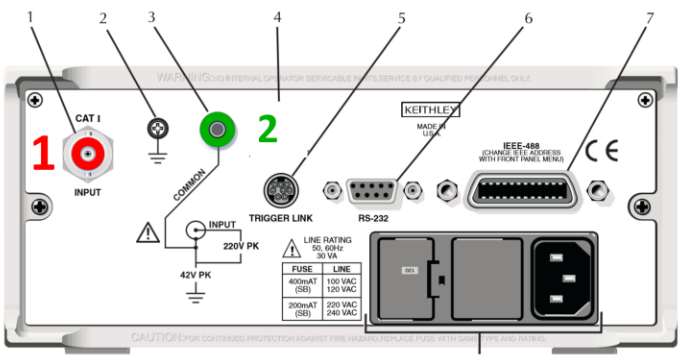


LOW CURRENT MEASUREMENTS

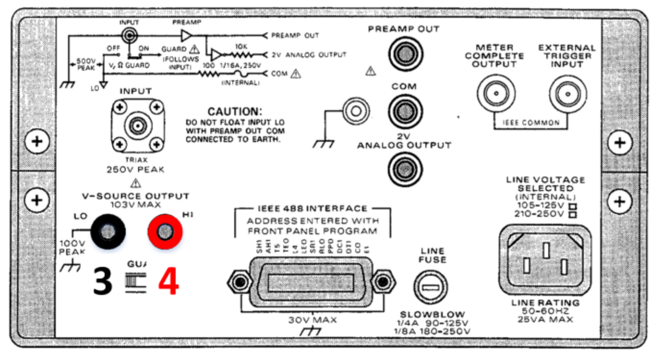
Keithley 6485 PicoAmmeter

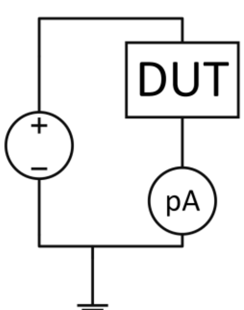
Rear Panel

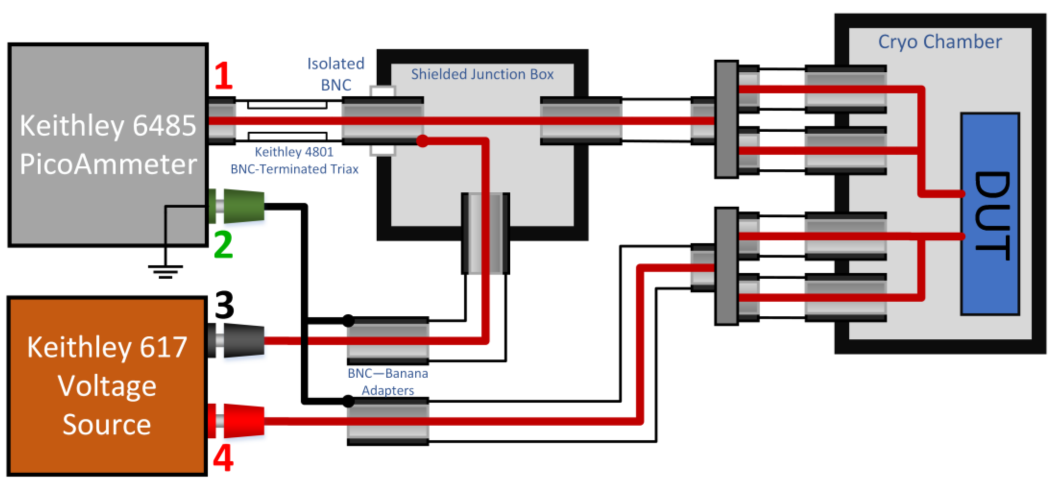


Keithley 617 Voltage Source

Rear Panel







AUTOMATION SOFTWARE

Flexible

- Allow for experiment customization
- Support multiple modes

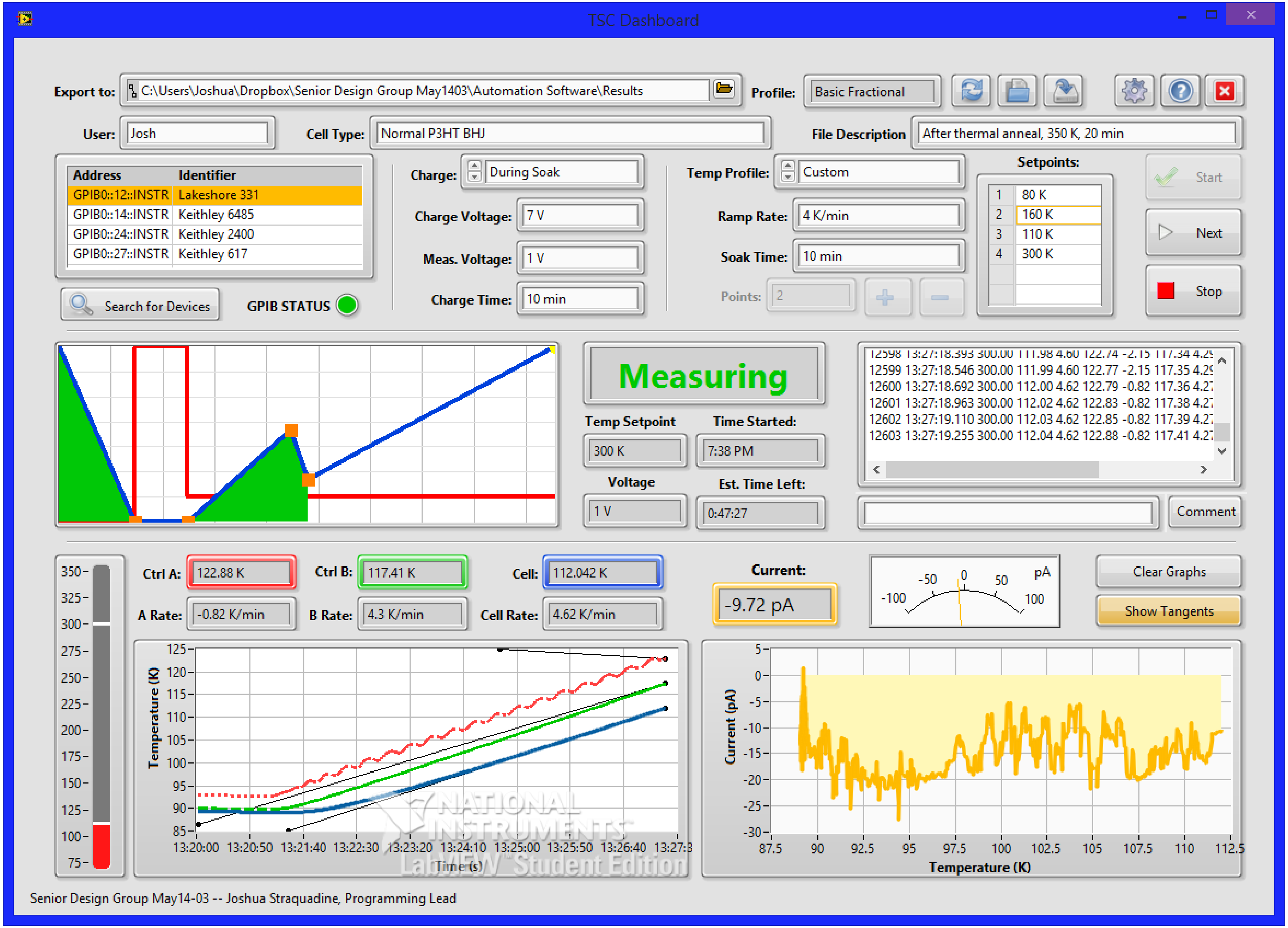
Robust

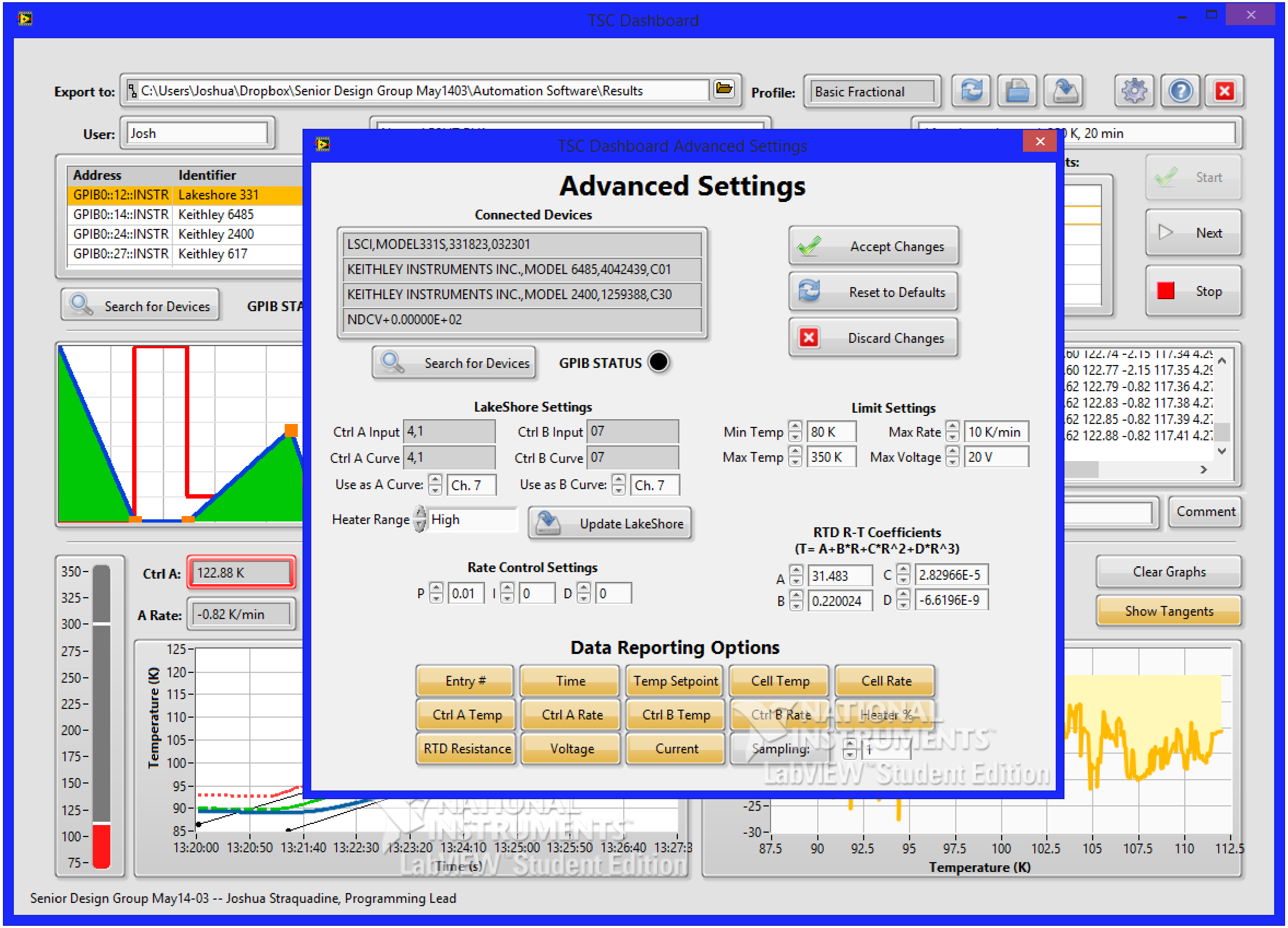
- Elegantly handle problems
- Keep user safe

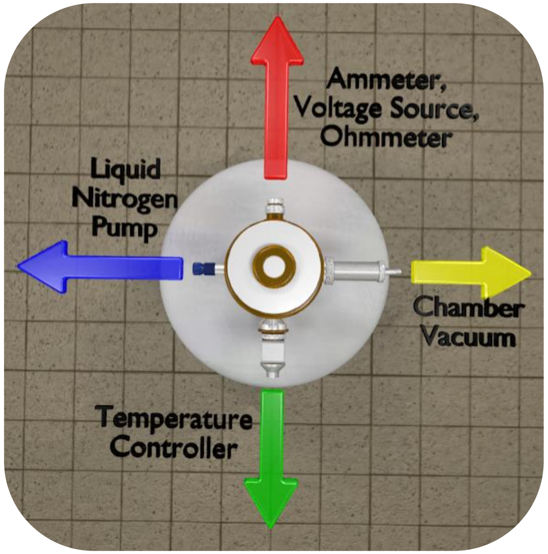
Accessible

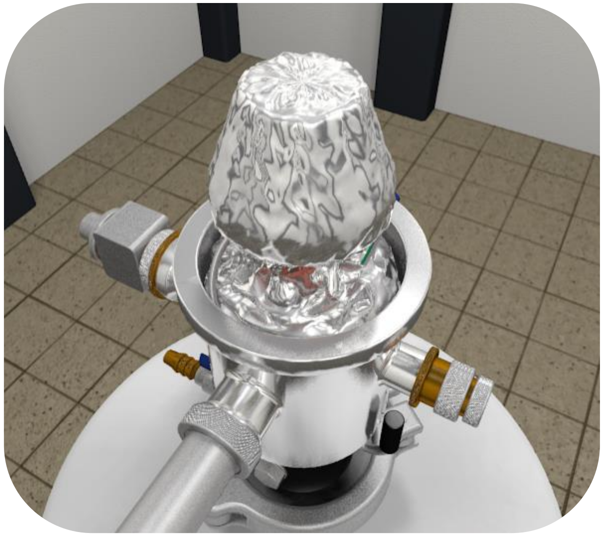
- Intuitive GUI
- Legible output

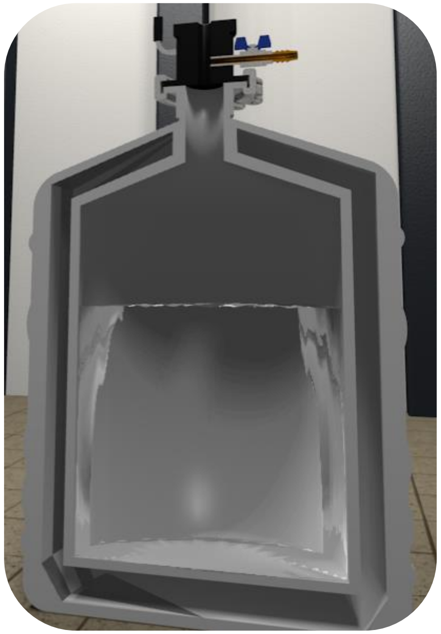
NATIONAL INSTRUMENTS LabVIEW™

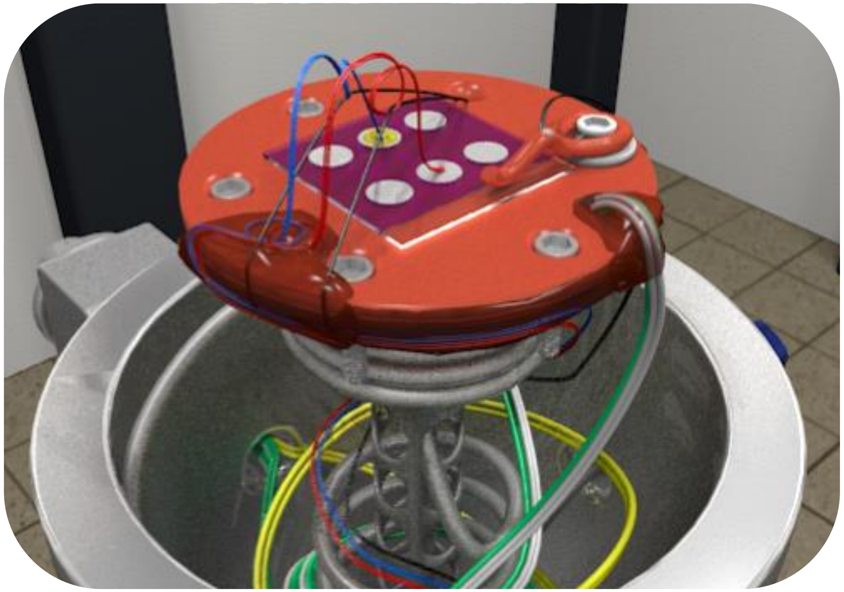

TSC Dashboard
Export to:
C:\Users\Joshua\Dropbox\Senior Design Group May1403\Automation Software\Results
Profile:
Basic Fractional
User:
Josh
Cell Type:
Normal P3HT BHJ
File Description
After thermal anneal, 350 K, 20 min
Address
Identifier
GPIB0::12::INSTR
Lakeshore 331
GPIB0::14::INSTR
Keithley 6485
GPIB0::24::INSTR
Keithley 2400
GPIB0::27::INSTR
Keithley 617
Search for Devices
GPIB STATUS
Charge:
During Soak
Charge Voltage:
7 V
Meas. Voltage:
1 V
Charge Time:
10 min
Temp Profile:
Custom
Ramp Rate:
4 K/min
Soak Time:
10 min
Points:
2
Setpoints:
1 80 K
2 160 K
3 110 K
4 300 K
Start
Next
Stop
Measuring
Temp Setpoint
300 K
Time Started:
7:38 PM
Voltage
1 V
Est. Time Left:
0:47:27
12599 13:27:18.546 300.00 111.99 4.60 122.77 -2.15 117.35
12600 13:27:18.692 300.00 112.00 4.62 122.79 -0.82 117.36
12601 13:27:18.963 300.00 112.02 4.62 122.83 -0.82 117.38
12602 13:27:19.110 300.00 112.03 4.62 122.85 -0.82 117.39
12603 13:27:19.255 300.00 112.04 4.62 122.88 -0.82 117.41
Comment
Ctrl A:
122.88 K
Ctrl B:
117.41 K
Cell:
112.042 K
A Rate:
-0.82 K/min
B Rate:
4.3 K/min
Cell Rate:
4.62 K/min
Current:
-9.72 pA
-100
-50
0
50
100
pA
Clear Graphs
Show Tangents
Temperature (K)
Time (s)
Current (pA)
Temperature (K)
Senior Design Group May14-03 -- Joshua Straquadine, Programming Lead


TSC Dashboard
Export to:
C:\Users\Joshua\Dropbox\Senior Design Group May1403\Automation Software\Results
Profile:
Basic Fractional
User:
Josh
Address
Identifier
GPIB0::12::INSTR
Lakeshore 331
GPIB0::14::INSTR
Keithley 6485
GPIB0::24::INSTR
Keithley 2400
GPIB0::27::INSTR
Keithley 617
Search for Devices
Start
Next
Stop
Comment
Clear Graphs
Show Tangents
Ctrl A:
122.88 K
A Rate:
-0.82 K/min
Temperature (K)
Time (s)
TSC Dashboard Advanced Settings
Advanced Settings
Connected Devices
LSCI,MODEL331S,331823,032301
KEITHLEY INSTRUMENTS INC.,MODEL 6485,4042439,C01
KEITHLEY INSTRUMENTS INC.,MODEL 2400,1259388,C30
NDCV+0.00000E+02
Accept Changes
Reset to Defaults
Discard Changes
Search for Devices
GPIB STATUS
LakeShore Settings
Ctrl A Input
4,1
Ctrl B Input
07
Ctrl A Curve
4,1
Ctrl B Curve
07
Use as A Curve:
Ch. 7
Use as B Curve:
Ch. 7
Heater Range
High
Update LakeShore
Limit Settings
Min Temp
80 K
Max Rate
10 K/min
Max Temp
350 K
Max Voltage
20 V
RTD R-T Coefficients
(T= A+B*R+C*R^2+D*R^3)
A
31.483
C
2.82966E-5
B
0.220024
D
-6.6196E-9
Rate Control Settings
P
0.01
I
0
D
0
Data Reporting Options
Entry #
Time
Temp Setpoint
Cell Temp
Cell Rate
Ctrl A Temp
Ctrl A Rate
Ctrl B Temp
Ctrl B Rate
Heater %
RTD Resistance
Voltage
Current
Sampling:
1
NATIONAL INSTRUMENTS
LabVIEW Student Edition
Senior Design Group May14-03 -- Joshua Straquadine, Programming Lead


Ammeter,
Voltage Source,
Ohmmeter
Liquid
Nitrogen
Pump
Chamber
Vacuum
Temperature
Controller







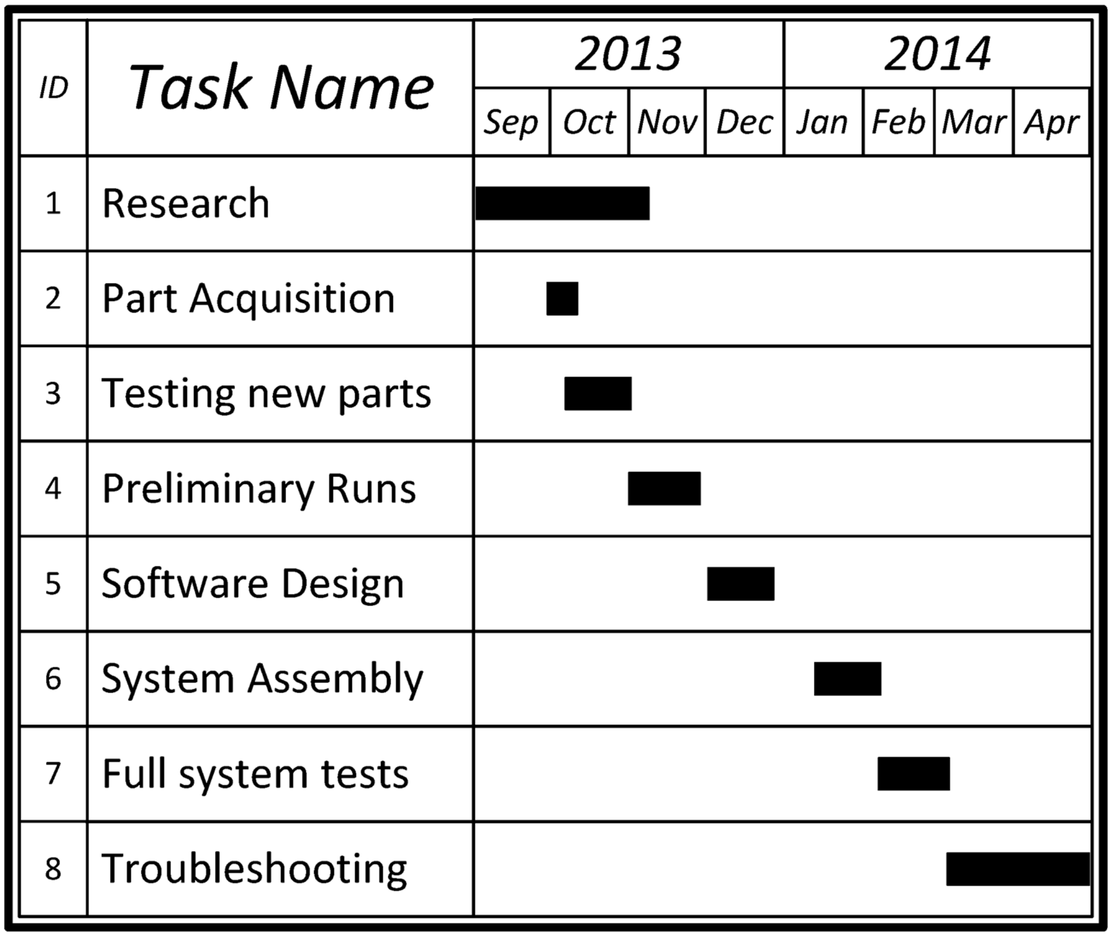
QUESTIONS?

APPENDICES

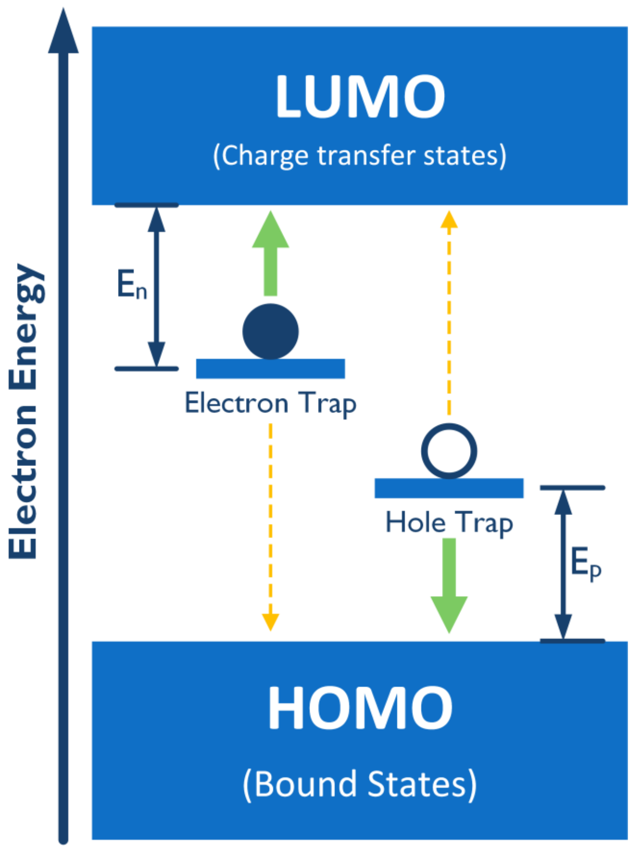
RESOURCE REQUIREMENTS

Resource	How will we get it?	Cost
Cryostat	Provided by client	---- N/A ----
Dewar flask	Provided by client	---- N/A ----
Liquid nitrogen	Provided by client	---- N/A ----
Aluminum Foil	Provided by client	---- N/A ----
Source measurement unit	Provided by client	---- N/A ----
Ohmmeter	Provided by client	---- N/A ----
Temperature controller	Provided by client	---- N/A ----
Computer with LabVIEW installed	Provided by client	---- N/A ----
GPIB interface cables	Provided by client	---- N/A ----
Thermally conductive grease	Provided by client	---- N/A ----
Wire shielding (conductive braid)	Provided by client	---- N/A ----
Cryogenic Varnish	Provided by client	---- N/A ----
New Platinum RTDs	PPG101A6, US Sensor (Digikey)	$ 58.44
Thermally conductive epoxy	Stycast 1266 Cryogenic Epoxy (CMR Direct)	$ 197.75
Thermal interface pad	5519 or 5591S Pads from 3M	$ 61.06
Connectors and wires	Digikey	$ 109.62
Junction Box Materials	Digikey	$ 50.48
Mylar Insulation	Edmund Scientifics	$ 31.90
Keithley 6485 Picoammeter	Keithley/Tektronix	$ 1,660.00
	TOTAL:	$ 2,169.25

PROJECT SCHEDULE



DE-TRAPPING KINETICS



De-trapping Rate:

$$R(T) = \nu_0 \cdot \exp\left(-\frac{\Delta E}{k_B T}\right)$$

ν_0 = attempt−to−escape frequency

Trap Characterization

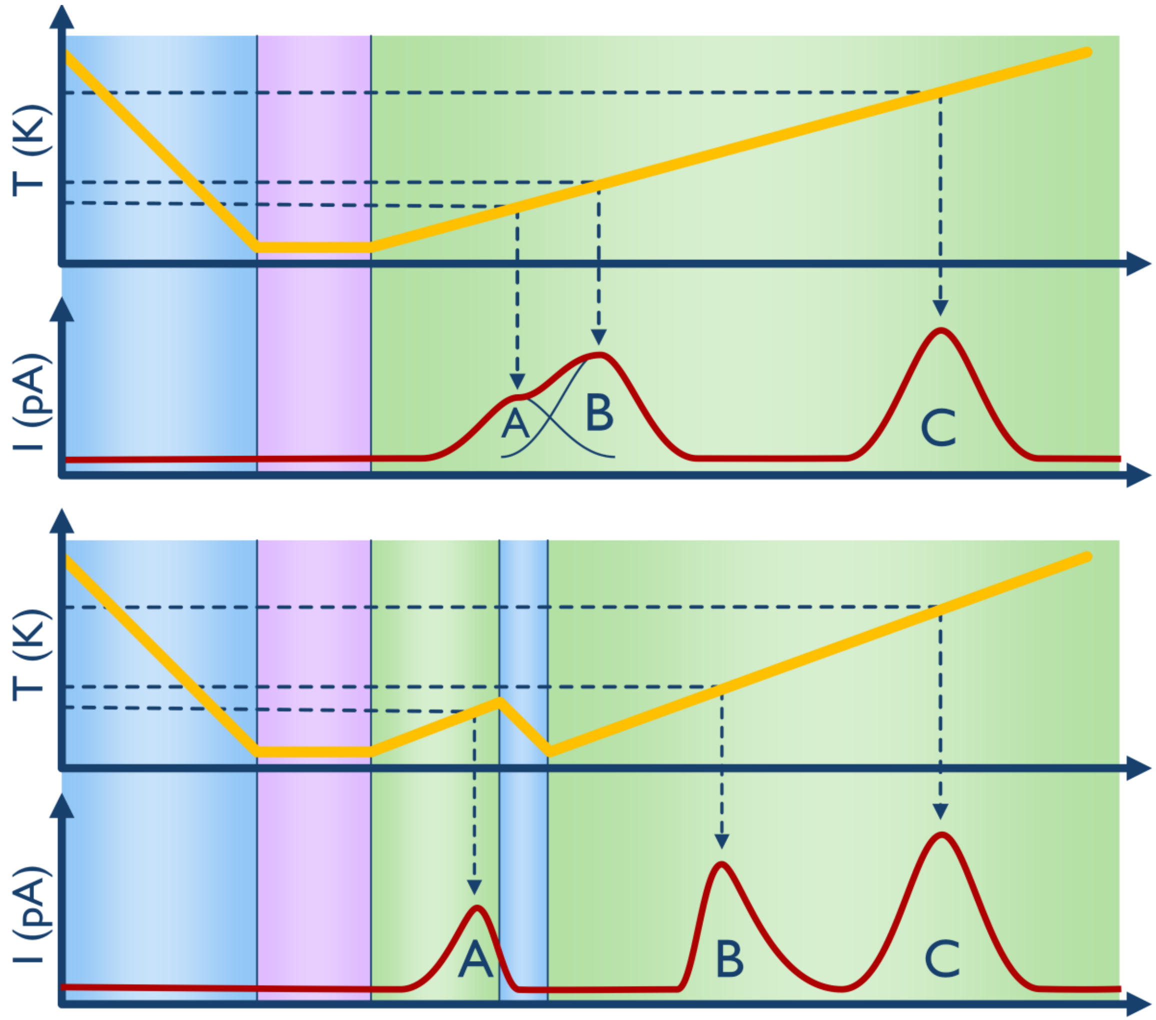
$$E_i = k_B T_m \frac{\ln\left(N\sigma v k_B T_m^2\right)}{\beta E_i}$$

N = effective density of states

v = carrier thermal velocity

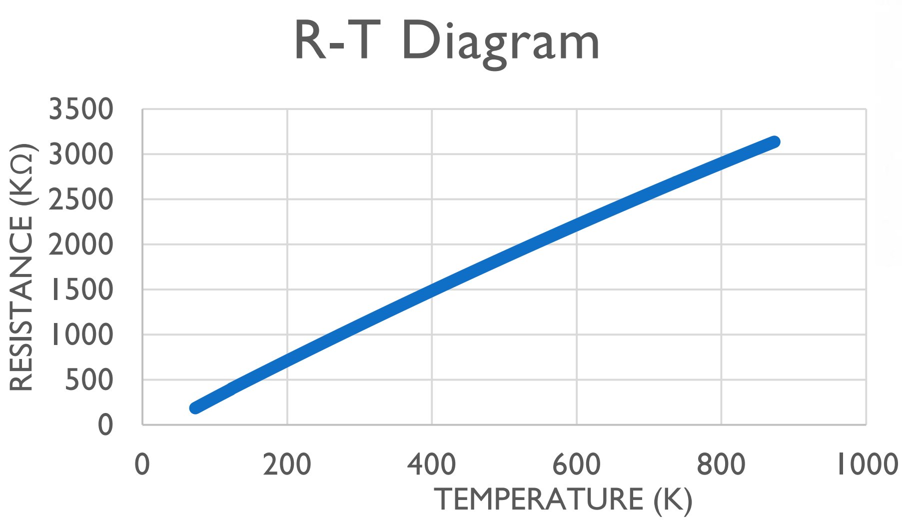
β = thermal scan heating rate

TSC AND FRACTIONAL TSC



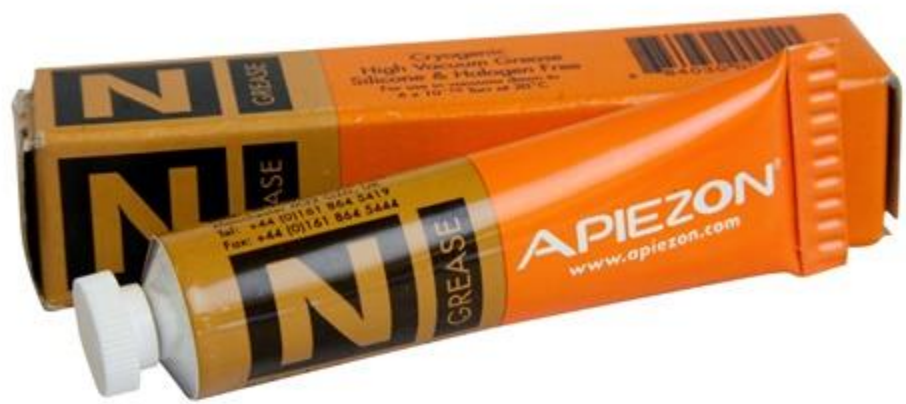
US SENSOR PPG102A6

- 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



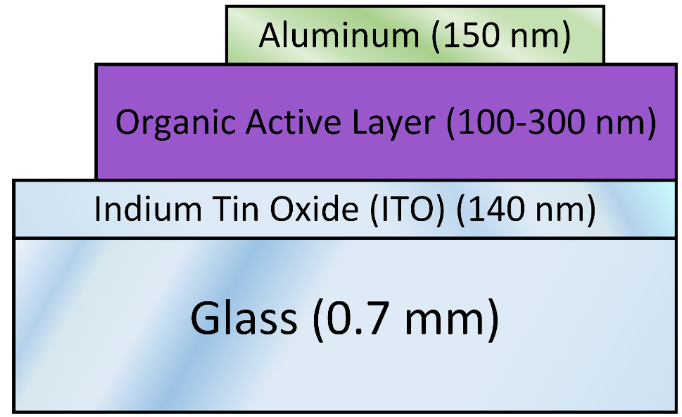
APIEZON N GREASE

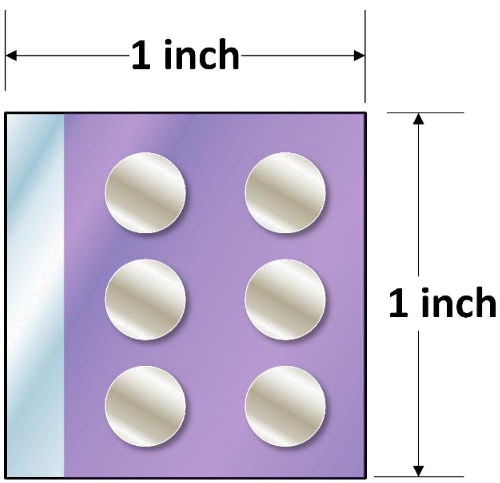
- Temperature Range: 0.15 K to 300 K
- Thermal Conductivity (80 K) = 0.1 W m^{-1} K^{-1}
- Vapor Pressure (273 K) = 2.67 x 10^{-7} torr
- Volume Resistivity 2 x 10^{16} Ω m

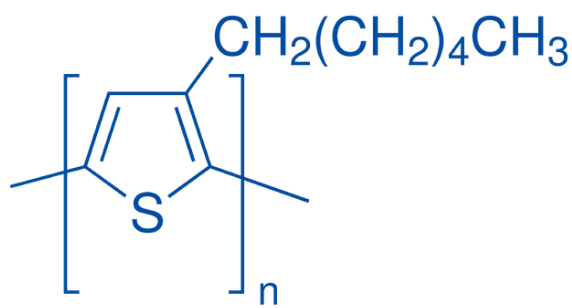


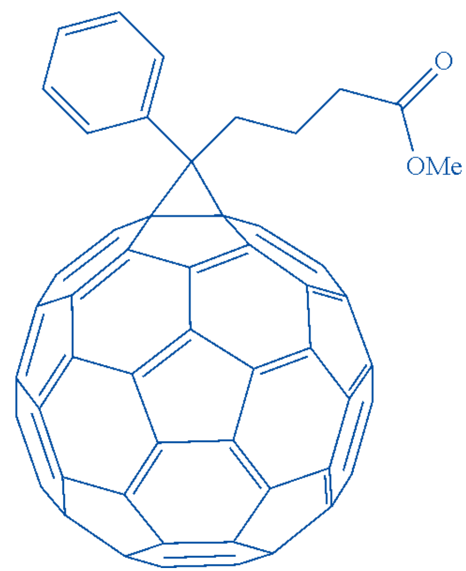
SOLAR CELL SAMPLES

- Bulk heterojunction
  - Poly-3-hexylthiopene
  - Phenyl-C61-butyric acid methyl ester
- Manufactured onsite using glass slides pre-deposited with ITO

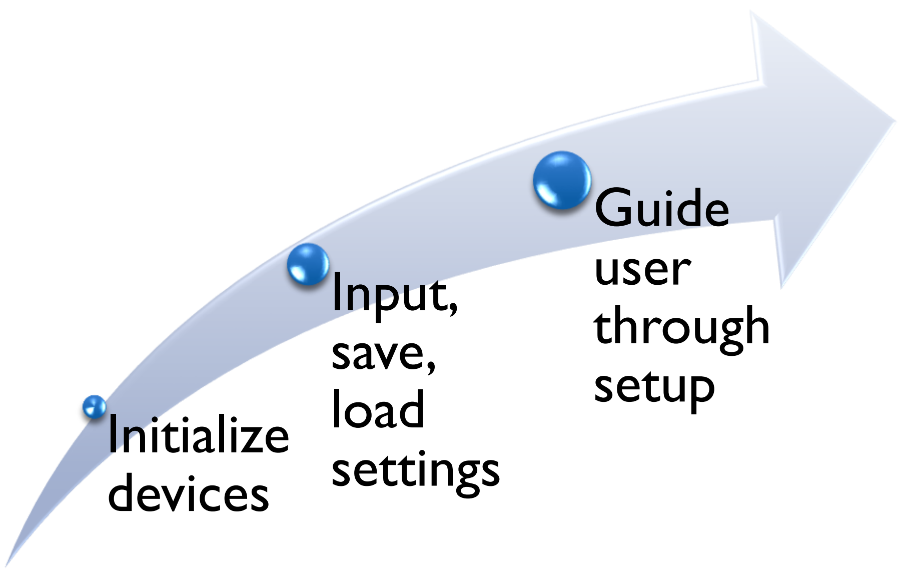


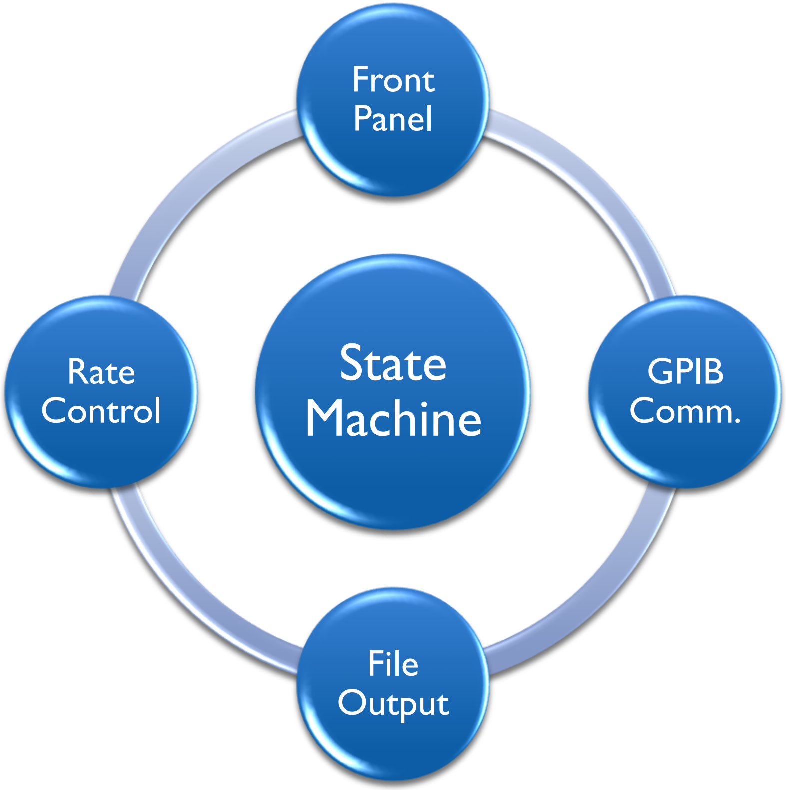






SOFTWARE ARCHITECTURE





LAKESHORE TEMPERATURE CONTROLLER

- 3-mode variable power heater, 50 W
- Accurate down to 1.2 K
- Thermal EMF compensation for resistive sensors

KEITHLEY 6485 PICOAMMETER

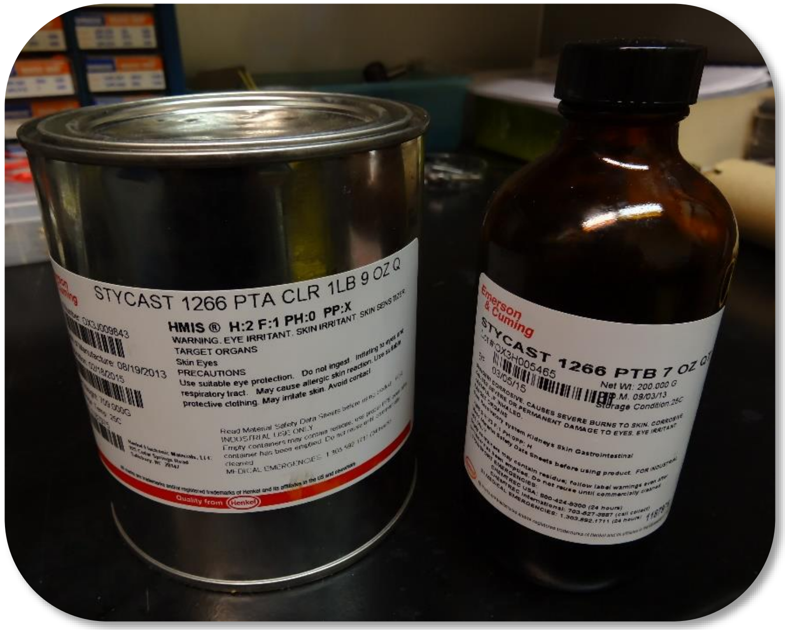
- 5½ digit display
- Resolution: 10 fA
- 1000 reads per second
- Accuracy: ±0.4%
- Coaxial hookups, triax adapters for low currents
- $1,660 (with educational discount)

VGE-7031 CRYOGENIC VARNISH

- Clear modified phenolic
- Easy to apply and remove
- Rigid when dry
- Dissolves in ethyl alcohol
- Vacuum compatible to 10^{-9} Torr

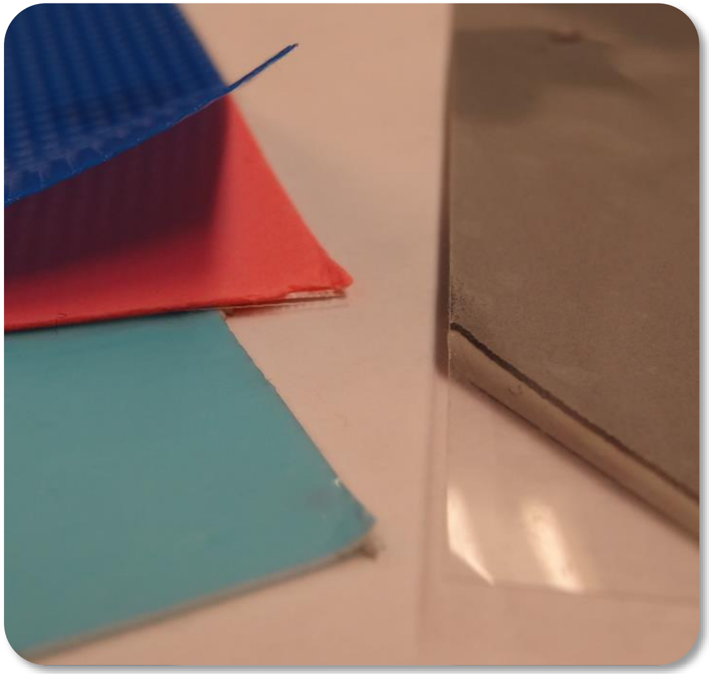
STYCAST 1266 CRYOGENIC EPOXY

- 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 $Wm^{-1}K^{-1}$



OUR WEBSITE

http://seniord.ece.iastate.edu/may1403/index.html