PROBLEM STATEMENT

In this project, the group will design and implement an experimental test setup to conduct trap-level spectroscopy measurements on organic photovoltaic (OPV) devices with the goal of characterizing the defect structure. Trap-level spectroscopy requires cryogenic temperatures, the lower the better. Our client is having difficulties getting the samples down to the target temperature of 80 K, so our task is to design improvements to the current system to make 80 K consistently attainable.

The defect characterization measurements will be made using a nonisothermal method of monitoring thermally-stimulated currents (TSC) created by charge carrier detrapping and thermal escape mechanisms as the sample is heated from cryogenic temperatures. The end goal of this project is an efficient and accurate method of studying electronic defects. In addition, the test setup will be fully automated and equipped with an easy-to-use computer interface programmed in LabVIEW.

DELIVERABLES

FIRST SEMESTER

- Design and fabricate improvements to a cryogenic vacuum chamber system to counteract poor thermal conductivity and radiative heating effects, with the goal of consistently cooling a sample to 80 K using liquid nitrogen. This deliverable can be subdivided into several smaller steps:
  - Evaluate the feasibility of, and potentially implement, a no-grease solution for low-thermal-impedance interface between the sample and cryostat
  - Design and build a layered enclosure within the vacuum chamber to minimize radiative coupling to the chamber walls
  - Determine the feasibility of encapsulating solar cells in a method which allows reliable and repeatable electrical and optical measurements of the cell, is optically clear, electrically insulating, and thermally conductive
- Determine a method of heating the sample at controllable of rates of 5-10 K per minute from 80 K to 300 K.
- Design a probe contact assembly for
- Determine a method of accurately measuring low currents (resolution in the fA range) with low noise and at least 1 sample per second
- Produce a well-labeled arrangement of cables and hookups which make the system easy to connect and disconnect as other experiments require
- Run at least one full test of the assembled system

SECOND SEMESTER

- Assess performance from first semester and make any required adjustments
- Develop control software in LabVIEW to automate the process of recording data
- Write a clear and cohesive system description and troubleshooting guide for future users of the system

SPECIFICATIONS

The new systems designed by this project as well as the improvements to the existing systems will all be expected to meet the following criteria.

VACUUM CHAMBER SYSTEM

- Chamber must be able to reduce the sample temperature to 80 K
- Must include a temperature sensor for real-time monitoring of the sample temperature
- Any improvements or additions to the efficiency of the system must fit inside the existing enclosure
- The cold trap enclosure must be easily and quickly removable by one person using no more than one tool
- A cold trap or other improvement must not interfere with electrical contact to the sample
- Requires a vacuum electrical feedthrough to enable the connection of enough wires to fulfill all measurement needs

CELL ENCAPSULATION/PREPARATION

ENCAPSULATION CHEMICALS

- Encapsulant must be optically clear with no visible clouding or discoloration
- Encapsulant must be thermally conductive, with conductivity of 1 $Wm^{-1}K^{-1}$ or higher
- Encapsulant must be electrically insulative with bulk resistivity in excess of 1 TΩ cm^{-1}
- Encapsulant must be chemically and mechanically stable down to at least 80 K

ENCAPSULATION METHOD

- Must significantly improve the repeatability of measurements and the endurance of cell contacts
- Must make contact to all six individual cells on each die
- Must be conducive to a contact failure rate of 33% or below (average of at least four working cells per die) after minimal training of the practitioner

THERMAL INTERFACE MATERIAL

- Must not leave a greasy residue which interferes with other types of downstream tests.
- Must have a thermal conductivity of 1 $Wm^{-1}K^{-1}$ or higher
- Must be conformal (Shore 00 Hardness of 70 or lower) to ensure good thermal contact

MEASUREMENT SETUP/CONTROL SOFTWARE

- Must enable all measurements to be made through GPIB interface
- Must encompass full automation of cooling, excitation, heating, and data collection
- Must be able to detect and suggest solutions to flaws in hardware setup or set-points
- Must elegantly and safely recover from software or hardware errors
- Measurement of thermally stimulated current must have resolution measured in fA
- Must record logs of all data in an organized and readable file format
- Log files must include date, time, setpoints used, information about the samples, and other important details deemed necessary by the client

EXPERIMENT DESIGN

- Hardware and software should support a customizable temperature profile for modified TSC methods such as thermal cleaning, fractional emptying, and reversibility cycles.
- Software must calculate an optimal power setting for the LN2 pump such that a single setting can be used for the duration of the test

CONCEPT SKETCH/MOCKUP

When the temperature is low enough, the sample will be excited. This can be done either by a voltage pulse, or a light pulse. We chose the voltage pulse because it makes for a simpler experimental setup and it will be easier for the user to customize the excitation.

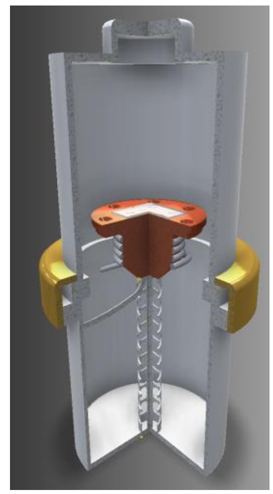
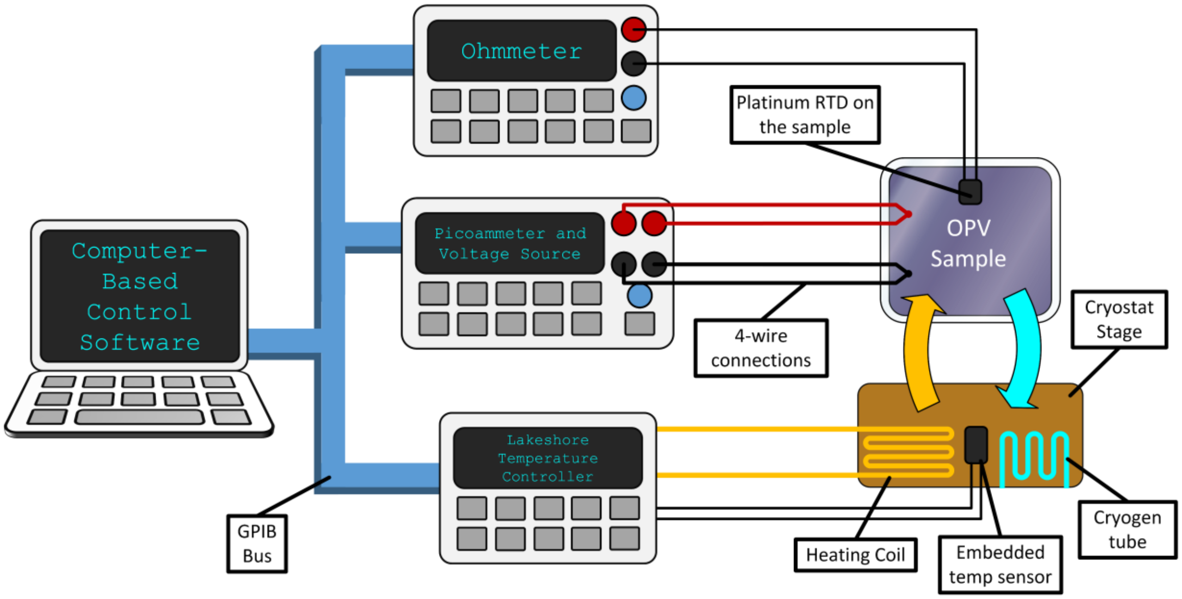


Figure 1: A rough sketch of the sample, placed on the copper cryostat head. The sample is a square slab of glass approximately 1 inch on a side. The glass is covered with a layer of indium tin oxide (ITO) which is a clear conductive material and forms one terminal of the solar cell. The ITO is left exposed along one edge (bottom right) where contact will be made. The active organic layer is deposited on top of that, and the six circles are aluminum pads, which form the other terminal of the solar cell. The metallic object in the center is a platinum resistive temperature detector (RTD), which measures the true temperature of the sample.

HARDWARE BLOCK DIAGRAM

The system as a whole will be centered on a cryostat head placed in a vacuum chamber. The solar cell sample will be placed on top of the cryostat surface with a thermal interface material (pad, grease, or some other thermally conductive filler) sandwiched between. The cryostat is cooled by a vacuum pump, which pulls liquid nitrogen from a Dewar flask through a tube in the cryostat. The temperature is monitored by a Lakeshore temperature controller, which also has a heating coil to stabilize the temperature at a predetermined set-point. Four-wire Kelvin point contacts will be made to the cell as shown in Figure 1, and a platinum RTD will be placed on the surface to measure the true temperature of the sample. Also shown here in dark grey is the cold trap which will encircle the sample and maintain thermal contact to the cryostat head. This layered structure of metal foil and thermally insulating material will be essential in bringing the sample down to 80 K. All of the measurement and control instruments, except for the vacuum pumps, will be controlled by computer software programmed in LabVIEW, and communicated via GPIB.



TEMPERATURE CONTROL UNIT

The cryostat consists of a container filled with liquid nitrogen connected to a Lakeshore Temperature Controller (model 331). An externally-powered vacuum pump pulls liquid nitrogen out of a Dewar flask through a copper head on which the cell sample is placed. The sample will be cooled by direct conduction of heat through the cryostat surface. The Lakeshore Temperature Controller monitors the temperature of the cryostat and controls a heating coil embedded in the copper assembly. The coil is used to maintain the temperature at a desired setpoint. The heating coil will also allow the user to raise the temperature of the sample at a desired rate. In order to reduce thermal loss through convection, the cryogenic chamber will be kept under vacuum of roughly 10^{-3} Torr.

MEASUREMENT EQUIPMENT

We need to keep track in our setup of the currents through the device and the temperature. Therefore our measurement equipment will consist of a source meter (Keithley 2400) to source and sense currents and an ohmmeter coupled with platinum RTD to serve as our temperature measurement equipment. We will be continuously assessing the measurement equipment, as the Keithley 2400 may not be able to measure with the required accuracy.

SOFTWARE & CONTROLS

The automation of the experimental procedure will be done through LABVIEW. We will interface our temperature controller device, the source meter and ohmmeter with the computer using GPIB communication. A flowchart of the program as planned is shown above.

Operating environment

Our system is designed to be operated ideally inside a laboratory room under standard ambient temperature and pressure conditions.

USER INTERFACE DESCRIPTION

HARDWARE

As far as hardware is concerned, the user will load the sample onto the cryostat by moving the contacts to make contact with the active aluminum area and to the ITO strip along one side. The user will then screw the chamber shut and make sure all the connections are properly made to the equipment. The software interface (described below) will prompt the user to turn on the vacuum pumps when the software is ready to collect data. Both are simple "On" switches. At the end of the process (prompted again by software) the user will screw open the vacuum chamber and remove the sample.

SOFTWARE

The software will have a graphical user interface which prompts for set points, shows progress information, and presents real-time temperature and current measurements. Upon starting the control program, it will automatically connect to the required devices and prompt the user to make any necessary connections or alterations to the hardware setup, as

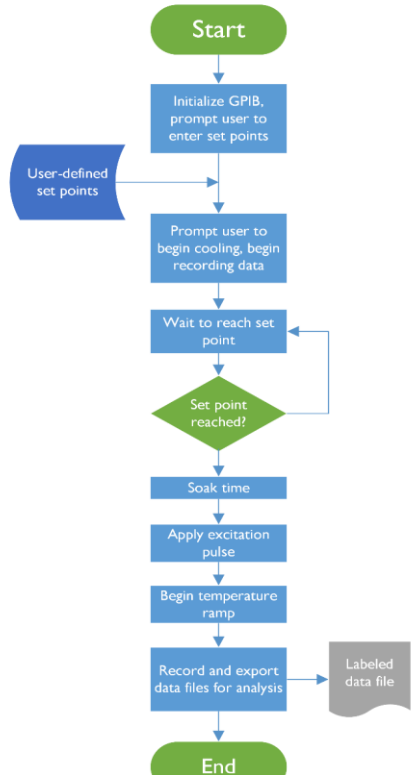


Figure 2: Software flowcharte

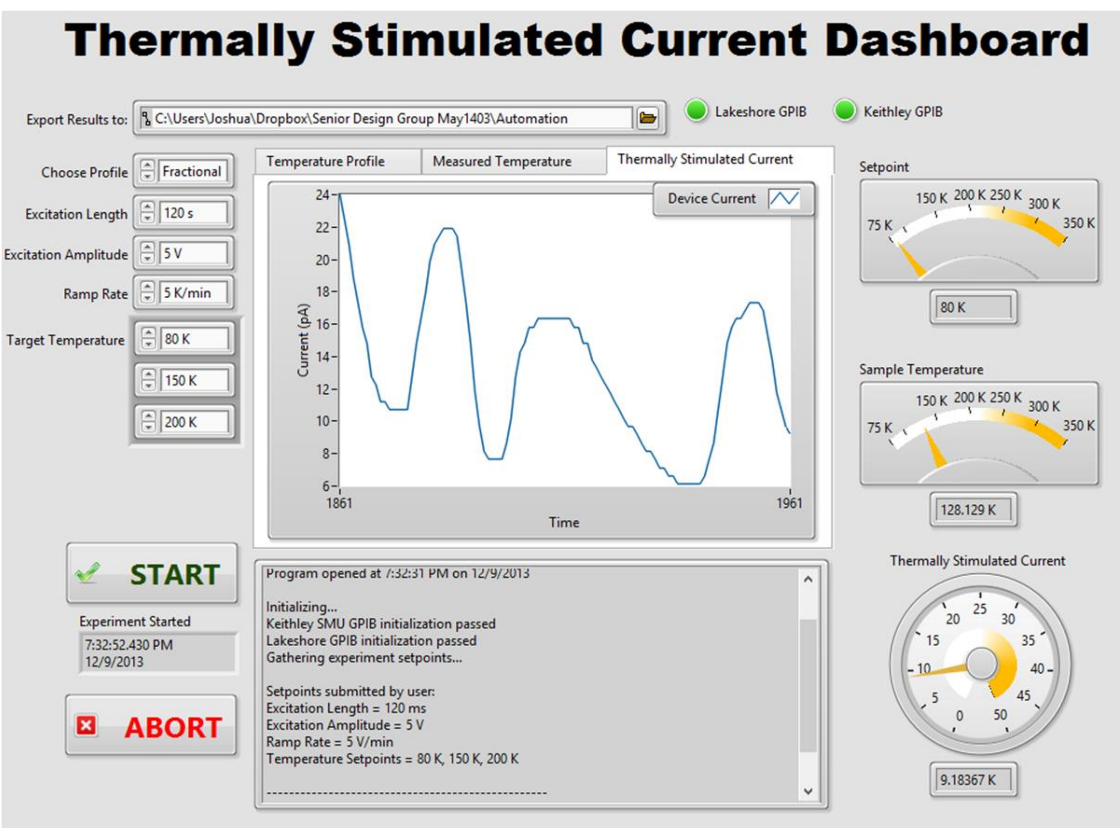


Figure 3: Screenshot of the Graphical User Interface

well as when to turn the cryogenic vacuum pump on. A mock-up of the intended design of the graphical user interface can be seen in the figure below.

FUNCTIONAL REQUIREMENTS

In order to be considered functional, the system must:

- Be able to reach the target temperature of 80 K
- Smoothly and accurately change the temperature at rates of 1 K per 5-10 minutes
- Make accurate current measurements with fA resolution at all temperatures
- Record all data in an organized and labeled, and accessible format
- Control the data acquisition process completely as defined by the user

HARDWARE

Easy assembly. It can't be completely pre-assembled because of the setup, but it should be made as easy as possible. A socket to ensure, the sample fits in the same place every time, easy to assemble inner- and outer faraday cages and custom cables that fit in a GPIB socket. This is as much to make testing easier, but more so to ensure consistent results. The test-cells should be thermally conductive but electrically isolating, so that we don't measure the capacitance between the aluminum circles and the thermally controlled copper pad. All metal parts (the cages, the cooling pad, the tank with nitrogen etc.) should be well connected to ground to ensure, that they don't pick up noise, which can ruin the experiment.

CONTROL & AUTOMATION

Temperature will be controlled by LabVIEW through the Lakeshore controller. All temperature measurements will be fed into LabVIEW somewhat continuously – sudden changes don't occur, so an update every second should suffice. The software will check if all is connected correctly, and if so, start the pulse when the temperature is satisfactory.

NON-FUNCTIONAL REQUIREMENTS

Our final product will not be going to market, so we have very few non-functional requirements. One important one, however, is that the interface should be easy to use and should look good for the researcher running the experiments. A simple schematic of the graphical user interface planned for interacting with the setup can be seen in Section 5.2.

WORK BREAKDOWN STRUCTURE

Every member of the group project is expected to remain up-to-date with all facets of the project. In order to ensure that the project plan is carried out successfully, each group member has been designated to a particular role as to preserve the group's stability and maximize work efficiency. Our group has decided on the following roles:

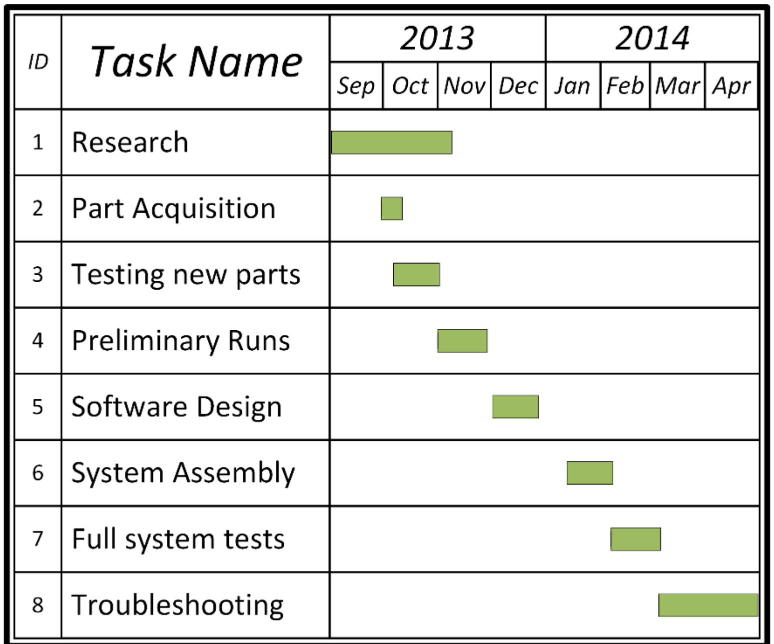
Josh Straquadine: Group leader
Nicholas Rodríguez: Editor, Secretary
Mouhamadou Diallo: Budget planner
Martin Andersen: Webmaster

Each member will specialize in parts of the project related to their academic background. Josh will lead with all of the semiconductor physics aspects, Martin will be in charge of signal integrity and noise management, Nick and Mouhamadou will be leads on the automation software. All members, however, will consult with each other about all major design decisions and specifications.

RESOURCE REQUIREMENTS

Resource	How will we get it?	Estimated 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
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
Vacuum feedthrough	Select a model from Kurt J. Lesker Vacuum Company	≈ $300.00
Connectors and wires	Speak with lab technicians at MRC	≈ $20.00

PROJECT SCHEDULE



RISKS

RISKS TO THE PROJECT TIMELINE

Our biggest obstacle and risk is how long it will take us to acquire parts. Unfortunately, after we order the parts we need, we will be stuck waiting on those materials to arrive before we can move forward. These kinds of issues would be less dire if we weren't constrained to only two semesters to complete the project. To combat this issue, we plan to identify our materials needs early and order from reliable suppliers. Many of the required components in Table 1 have already been ordered.

PHYSICAL DANGERS:

The main physical risk regarding this project is the possibility of frostbite. While the outside of the outer cage doesn't get particularly cold, there will be an inherent risk if one doesn't let the sample heat up before it is removed. The biggest danger when working with cryogenic liquids is the buildup of pressure due to evaporation. These pressures can cause unexpected explosions and ruptures of containers if proper procedures are not followed. For this reason, the Dewar flask system includes two separate pressure release valves which must remain open at all times to avoid any such events.

The only other danger to be considered is that we will be working with a source meter unit (SMU), which can operate as a current source in several modes. In order to source a determined amount of current, the SMU has the capability of delivering very high voltages. While the source meter is on, care must be taken not to touch the leads or disconnect anything, because the system will automatically increase the voltage to dangerous levels.

MARKET/LITERATURE SURVEY

The project we are trying to accomplish is very specialized and is not intended for a market. There are also no existing products which fulfill our needs, but we will need to assemble a few materials. The first issue we researched a new method of measuring temperature. The original system was using a simple thermistor which was only rated to a low temperature of 200 K. We considered thermocouples, silicon diodes, and platinum resistive temperature detectors (RTDs), and chose the platinum RTDs as the best option. We will continue to examine the performance of the RTD devices throughout the course of the project.

The other main issue in this project is thermal conductivity between the sample and the cryostat head. There are many thermal interface materials available, including greases, conformable pads, and encapsulating epoxies, but very few of them have been shown to perform well at cryogenic temperatures. In order to find some good solutions, we contacted several companies, including Dow Corning, 3M, Parker Chomerics, MasterBond, and Stycast, and we will be evaluating our materials continually.

Beyond those material requirements, we also searched through some of the research literature and engineering textbooks on the topic of cryogenic vacuum chambers and trap-level spectroscopy. Below are the sources which we found helpful.

T. Benanti & D. Venkataraman. "Organic solar cels: An overview focusing on active layer morphology". Photosynthesis Research, Vol. 87, 2006, pp. 73-81.
J. Ekin. Experimental Techniques for Low-Temperature Measurements: Cryostat Design, Material Properties, and Superconductor Critical-Current Testing. New York: Oxford University Press, 2006.
T.W. Kerlin. *Practical Thermocouple Thermometry*. North Carolina: Instrument Society of America, 1999.
M. Kim. Understanding Organic Photovoltaic Cells: Electrode, Nanostructure, Reliability, and Performance. Doctoral Dissertation. University of Michigan, 2009.
V.I. Mikla, V.V. Mikla. Trap Level Spectroscopy in Amorphous Semiconductors. London: Elsevier, 2010.
J. Moore, C. Davis, M. Coplan, S. Greer. *Building Scientific Apparatus, Fourth Edition*. New York: Cambridge University Press, 2009, pp. 600-621.
R.G. Scurlock. *Low Temperature Behavior of Solids*. New York: Dover Publications, 1966.
S. Sun, N.S. Sariciftci. *Organic Photovoltaics: Mechanisms, Materials, and Devices*. Boca Raton: CRC Press, Taylor & Francis Group, 2005.