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Experiment Safety Review Form
Review Number: NC-10-2011

PRINCIPAL INVESTIGATOR: Fernando Camino
GROUP: CFN
EXT: 7606 **E-MAIL: fcamino@bnl.gov** **LIFE NUMBER: 23799**

Project Title: Electronic transport in nanostructures
Location(s): 0735
Area(s): 0735-FIRST-1-1L35
Proposed Start Date and Duration: 12/31/2009 - 1 years

SIGNATURES:

Principal Investigator: Fernando Camino	**Date: 2/7/2012**
Experiment Review Coordinator: Robert Sabatini	**Date: 2/7/2012**
Reviewer: Nicole Chiu	**Date: 12/6/2011**
Reviewer: Lorraine Davis	**Date: 12/15/2011**
Approval: Emilio Mendez	**Date: 2/17/2012**
Review/Approval (ERC) Comments: 02/07/2012 4:03 PM	
Walkthrough Signature: Robert Sabatini	**Date: 2/17/2012**
Expiration Date (max 1 yr.): 2/17/2013	
FUA Change Required? No	
Fire Rescue Run Card Changes Required? No	
Has a NEPA Review been Performed for this Project? Yes	
Required Approvals (i.e., IACUC, IBC, etc.): NA	
Project Termination Acceptance Signature: **Comments:**	**Date:**

I. Define the Scope of the Work

A. Description

Nanomaterials are expected to exhibit electrical and optical characteristics not present at macroscopic dimensions. In this laboratory, experiments are carried out to understand the electrical behavior of these novel materials in order to seek applications in areas of interest of the Center for Functional Nanomaterials, such as the improved performance of energy-conversion devices.

All nanostructures under study in Lab 1L-35 are in solid, non-volatile state, fixed or mounted in macroscopic size substrates. Consequently, there is no risk of ambient contamination from nanostructures. When samples are not measured, they are preserved in closed containers, catalogued and stored in lab cabinets. No samples are discarded in this laboratory.

Electrical measurements are performed using conventional low-noise and low-signal electronic instrumentation. Examples are nanovoltmeters, semiconductor parameter analyzers, lock-in amplifiers, etc. Most of the time, the delicacy of the nanostructures under study limits the level of currents and voltages used during experiments to values below milliamperes and few volts, respectively. Accordingly, dangerous situations for users are not expected. Obviously, the instrumentation is capable of creating dangerous signals if not used appropriately. To reduce to a minimum the chance of accidents, users are trained in the correct use of the electronic instrumentation. In addition, each piece of instrumentation has a user's manual, which users are required to read and understand prior to starting their research.

In this laboratory, samples mostly are characterized electrically using state-of-the-art probe stations. Probe stations use micromanipulators to precisely place conductive needles on a sample in an area of the order of 10x10 μm^2. An optical microscope is needed to locate the area of interest. Currently, lab 1L-35 has three probe stations: two room temperature stations (Signatone CheckMate and Rucker and Kolls model 260), and a cryogenic probe station, the LakeShore Model HFTP4, which uses liquid helium and liquid nitrogen to lower the temperature of the sample under study. Because of its design, only the Signatone CheckMate needles present some puncture risk to the user if not operated correctly. To minimize this potential hazard, only authorized users will have access to the probe stations in lab 1L-35. The authorization will be granted by Fernando Camino after users are trained in the correct operation of the probe stations. The needles of the other two probe stations, Rucker and Kolls and LakeShore, present minimal puncture risk to the user.

This laboratory also has the capability of performing optoelectronic characterization of nanostructured photovoltaic devices (i.e. power conversion efficiency and external quantum efficiency) using a probe station (Rucker and Kolls model 260) equipped with a solar simulator and a monochromator. The solar simulator uses a xenon arc lamp to output simulated solar light, and the monochromator selects an specific wavelength. The output spectrum contains ultra-violet (UV) light, which is a potential ozone generator. Thus, in order to reduce this hazard, the following engineering measures are taken: 1) . To prevent users' exposure to ozone and UV light, the lamp housing, where most of ozone is generated, is contained in a closed box, whose exhaust port is connected to the house exhaust, and 2) the light path is covered by light-tight optical components. A designated ES&H coordinator has tested UV and ozone levels of this system to ensure user's safety.

In January 2010, the lab incorporated a high temperature (500 °C) annealing oven (MBE-Komponenten AO500) capable of in-situ electrical measurements performed under vacuum (2 mbar) or under gas flow regulated by mass flow controllers. Current gases available are Argon, nitrogen and hydrogen. Engineering measures to reduce hazards associated with hydrogen gas have been taken, such as leak testing the hydrogen gas regulator and gas supply lines. In addition, we have connected the pump exhaust to the building vent pipe in order to avoid any hydrogen from accumulating in the room.

In December 2011 two new pieces of equipment will be added to the laboratory: A Hall Effect measurement System from LakeShore Cryotronics, Inc. and a Variable Temperature Micro Probe System from MMR Technologies.

Below we briefly describe each main piece of instrumentation of this laboratory.

The Signatone CheckMate Probe Station is an ultra-stable 200mm/300mm analytical probe station with coarse wafer stage and fine microscope xyz movement. It operates at room temperature. The services it requires are house compressed air (80 psi) to move the microscope head, and rough house vacuum to hold samples in place. The probe sits on an optics table that isolates vibrations and that requires house compressed air (80 psi) for its operation.

The Photovoltaic Device Characterization Station (PDCS) mainly consists of a custom-modifed Rucker and Kolls model 260 room probe station, two 150 W solar simulators, a 300 W xenon arc lamp source, and a monochromator. The system can input the simulated sun light from either top or bottom, permitting measurement of both conventional inorganic based *solar cells and more exotic third generation solar cells (organic, dye-sensitized, quantum-dot, hybrid-type etc.) fabricated on transparent bottom substrates. Currently the system can input up to 1.5 SUN light intensity on to a sample with a capability to vary the light intensity using a set of optical neutral density filters.The LakeShore Model HFTTP4 cryogenic probe station is a versatile micromanipulated probe station used for non-destructive magnetic testing of devices at variable temperatures from room temperature down to liquid helium temperature. The maximum magnetic field attainable is 1 Tesla. This field is controlled by a power supply that consumes about 1.6 kW of power, connected to a separate single-phase electrical supply. The stray fields that this probe station generates were mapped with a Gauss probe. The 5 gauss line is 80 cm from outside of the vacuum chamber in the direction down the bore of the magnet. In the direction perpendicular to the bore the 5 gauss line is 40 cm from the outside of the vacuum chamber. Above the vacuum chamber the 5 gauss line is 40 cm above the top of the vacuum chamber. The maximum above the chamber is not in the middle but approximately above each coil. Under the chamber the 5 gauss line is 30 cm below the bottom of the vacuum chamber.*

To cooldown samples, liquid nitrogen and liquid helium cryogens are required. In order to transfer these cryogens into the probe, pressurized N_2 and He gases are needed. An attached file to this ESR contains the recommended cooldown procedure for this probe station.

The MBE-Komponenten AO500 is a high temp (500 °C) annealing oven capable of in-situ electrical measurements. Measurements are performed under vacuum (2 mbar) or under gas flow regulated by mass flow controllers capabale of sourcing up to 5,000 sccm of gas. Current gases available are argon, nitrogen and hydrogen.

The Model 8404 AC/DC Field Hall Effect Measurement System is designed to measure mobilities down to 0.001 cm^2/Vs, which allows the study of low mobility materials such as organic electronic materials and transparent conducting oxides (e.g., ITO, ZnO or IGZO).

MMR's Variable Temperature Micro Probe System allows opto-electrical characterization of samples in the temperature range from 70K to 730K under controlled atmosphere (inert, vacuum etc.) and light condition. The sample cooling is achieved by a Joule-Thompson refrigeration system. Here, flow of high pressure nitrogen or argon (~1800 psi, 0.7-9 scfm) is pumped by a mechanical pump through a capillary stage where cooling action occurs. The system does not exert any mechanical vibration or noise. The pumped inert gas will be exhausted through the building exhaust line, ensuring that the gas does affect the normal laboratory air composition. Currently the system is equipped with four microprobes and a electrical feed-through that can accommodate up to ~20 manual electrical connections to a sample for more customized electrical measurements. The probes can be positioned anywhere on a 10 mm x 12 mm sample to a precision of 50 microns.

Equipment manuals or procedures that are controlled documents:

All documentation regarding electronic instruments and probe stations are located in two places. Thin manuals are stored in a file cabinet organized by instrument name. Manuals that are too thick and heavy are located on a bookshelf by the entrance of the laboratory.

B. Human Performance Factors

Never leave cryogenic probe station unattended while cooling with LN2 or LHe. There are several things that can go wrong, which are easily caught and corrected if User is present and follows cooling down instructions. Training is mandatory in order to closely follow cooldown procedure and avoid accidents

C. Waste Minimization/Pollution Prevention

There will be no waste generated besides pump oil.

D. Materials Used /Waste Generated

Materials Used	Disposal Method	Amount per Use	Amount per Year	Comments
N2 gas	Point Source	1.00 ft3	200.00 ft3	
He gas	Point Source	1.00 ft3	400.00 ft3	
Ar gas	Point Source	1.00 ft3	400.00 ft3	
H2 gas	Point Source	1.00 ft3	400.00 ft3	
liquid N2	Fugitive	50.00 ltr	500.00 ltr	
liquid He	Fugitive	30.00 ltr	300.00 ltr	
metahnol	Fugitive	10.00 ml	500.00 ml	
acetone	Fugitive	10.00 ml	500.00 ml	
2 propanol	Fugitive	10.00 ml	500.00 ml	

II. Identify and Analyze Hazards Associated with the Work

The following hazards were identified:

Physical Hazards:

- Oxygen deficient atmosphere
  (Area: 0735-FIRST-1-1L35)
- Cryogens (any substance or device capable of producing temperatures <= 170K)
  (Areas: 0735-FIRST-1-1035, 0735-FIRST-1-1L35)
- Compressed gases (lecture bottles, cylinders, gas lines)
  (Areas: 0735-FIRST-1-1035, 0735-FIRST-1-1L35)
- Compressed gas-Flammable
  (Areas: 0735-FIRST-1-1035, 0735-FIRST-1-1L35)
- Flammable liquids
  (Areas: 0735-FIRST-1-1035, 0735-FIRST-1-1L35)

Chemical Hazards:

- Chemicals, Hazardous (General)
- Flammable liquids
  (Areas: 0735-FIRST-1-1035, 0735-FIRST-1-1L35)
- Nano-material bound in a solid matrix or fixed substrate
  (Areas: 0735-FIRST-1-1035, 0735-FIRST-1-1L35)

Ionizing and Non-ionizing Radiation Hazards:

- Static magnetic fields >5 G and <600 G (attach exposure form)
  (Areas: 0735-FIRST-1-1035, 0735-FIRST-1-1L35)
- Static magnetic fields =>600 G (attach exposure form)
- Ultraviolet sources >1 W
  (Areas: 0735-FIRST-1-1035, 0735-FIRST-1-1L35)

Biological Hazards:

- None

Offsite Work:

- None

Other Issues (Security, Notifications, Community, etc.):

- None

Significant Environmental Aspects

- Any amount of industrial waste generation (e.g., oils, vacuum pump oil)

- Production or use of, or waste containing, engineered nanomaterials
- spill potential (Other)

III. Develop and Implement Hazard Controls and Assess Risk

A. Physical Hazards, Tasks and Controls

Hazard, Default Controls, Task Specific Info	Risk Level
Hazard: Oxygen deficient atmosphere **Default Controls:** Contact ESH Coordinator for ODH determination and associated controls. Comply with Subject Area "Oxygen Deficiency Hazards (ODH), System Classification and Controls" **Task Specific Info:** ODH 0 training is required.	**Negligible (0-20)**
Hazard: Cryogens (any substance or device capable of producing temperatures <= 170K) **Default Controls:** General Requirements: • Evaluate location oxygen deficiency • Store/transport only in approved containers (i.e. DOT/ASME or BNL LESHC) • Never pour from above chest level • PPE: Long Sleeve Shirt (or Lab Coat), long pants (or skirt covering ankles) and closed shoes Pressurized transfer to open (vented) container; Or-Pouring > 5 liter volumes of LN2 between open containers: • Face shield along with either Safety Glasses (w/side shields) or Goggles • Gloves (Cryo or Heavy Leather) Pouring small (5 liters or less) volumes of LN2 between open containers: • Safety Goggles (face shield recommended if possible) • Gloves (Cryo or Heavy Leather) Work with samples immersed in LN2 in small (~1 liter) dewars: • Use Tongs (tools) to manipulate/handle cryogenic samples (do not touch with gloves)• Use insulated non-absorbent gloves with dexterity (cotton/nylon gloves under disposable nitrile gloves) • Safety Goggles	**Negligible (0-20)**
Hazard: Compressed gases (lecture bottles, cylinders, gas lines) **Default Controls:** • Any systems >15psi must be SME Approved • Transport cylinders using a cylinder cart • Secure cylinders to a fixed object/wall • Use regulator, hoses, and components compatible with gas • Use hoses and clamps rated for maximum regulator output or use pressure relief device • Wear safety glasses with side shields when installing/removing/or adjusting regulator • Label piping/tubing	**Negligible (0-20)**
Hazard: Compressed gas-Flammable	**Negligible (0-20)**

	Default Controls: In addition to compressed gas requirements: • Flash arrestor/backflow device • Separate 20 ft from oxidizers or barrier • Electrically ground lines/equipment • Non-Sparking tools	
Hazard:	**Flammable liquids** **Default Controls:** As for chemicals, plus Store large quantities in Flam. cabinets as required **Task Specific Info:** Only small amounts used for cleaning parts is permitted in this lab.	**Negligible (0-20)**

B. Chemical Hazards, Tasks and Controls

Hazard, Default Controls, Task Specific Info		Risk Level
Hazard:	**Chemicals, Hazardous (General)** **Default Controls:** • All operations with large (>250ml, health hazard 3) quantities of hazardous chemicals (pouring, mixing, evaporation, etc) in hood, or use snorkel when hood is impractical • Register Commercial Chemicals in CMS • Work alone after hours only if permitted by supervisor or ESR • Identify containers so contents are identifiable unless being actively used (ex. 1 shift) • Food, beverage, smoking, and cosmetics are prohibited • Handle glassware properly: no mouth suction, no drinking from labware.	**Negligible (0-20)**
Hazard:	**Flammable liquids** **Default Controls:** Use hazardous chemicals controls. Review large quantity storage with Fire Protection Engineer Note location on fire run card and hazard info. placard for storage of solids, greater than 40 pounds; liquids, greater than 5 gallons; gases, greater than 10 pounds	**Negligible (0-20)**
Hazard:	**Nano-material bound in a solid matrix or fixed substrate** **Default Controls:** PPE Requirements for Handling: Standard PPE required for the work area. No additional requirements. Handling Requirements • No Mechanical abrasion. • No thermal stresses. • No etching Laboratory Posting Requirements - No Posting Requirements Waste Handling: • Solids containing nanomaterials which have a potential to be released must NOT be disposed in the regular trash. See SBMS Hazardous Waste Management for more information. • Collect solid UNP waste in a bag (6 mil thick minimum zip-lock type or J-sealed) or other sealing container (i.e. jar with threaded lid). • Spell out the chemical name (do not use formulas or trade names) on the RED Hazardous Waste Label. • The contents line on the label must contain the chemical composition and the word “NANO”	**Negligible (0-20)**

• A second label, in addition to the Red Hazardous Waste Label, is required on the outside other container/bag stating “CONTAINS NANOMATERIALS” see ES&H Coor. or 90-Day area manager for labels.

C. Environmental Hazards, Tasks and Controls (include on/off site transportation and products/services)

Hazard, Default Controls, Task Specific Info	Risk Level
Hazard: Any amount of industrial waste generation (e.g., oils, vacuum pump oil) **Default Controls:** Engineering Controls • Store only compatible wastes together, in suitable containers. • Provide secondary containment for liquid wastes if potential for environmental release exists. • Keep containers closed and secured unless adding waste to container. Administrative Controls • Use a green industrial waste label, with generator’s name and chemical contents (trade name/formula NOT acceptable). Label oils “Used Oil”. • When full, complete and submit a WCF for pick up. The waste may be stored in the 90-day area. Training: Hazardous Waste Generator (HP-RCRIGEN3) Comply with the SBMS Subject Area: “Industrial Waste”.	**Negligible (0-20)**
Hazard: Production or use of, or waste containing, engineered nanomaterials **Default Controls:** • All waste in contact with nanomaterials must be disposed as hazardous waste e.g., (gloves, lab coats, swabs, Kimwipes, blotter paper, beakers, flasks, tape, sample holders). Chemicals containing nanomaterials must NOT be released to the sink or disposed in the regular trash. See SBMS Hazardous Waste Management for more information. • Liquids must be stored in a rigid leak proof container. • Particulates must be stored in a rigid leak proof containers “OR” =/> 6 mil plastic bags. • Spell o	**Negligible (0-20)**
Hazard: spill potential (Other) **Default Controls:**	**Negligible (0-20)**

D. Radiation Hazards, Tasks and Controls

Hazard, Default Controls, Task Specific Info	Risk Level
Hazard: Static magnetic fields >5 G and <600 G (attach exposure form) **Default Controls:** Administrative Controls • Request SMF survey through SHSD • Complete JAF or Non-Employee Static Magnetic Field Questionnaire for exposed employees/workers • Training: TQ-SMF (supervisors) • Appropriate posting (see Subject Area)	**Negligible (0-20)**
Hazard: Static magnetic fields =>600 G (attach exposure form)	**Negligible (0-20)**

Default Controls:
Administrative Controls
• As above for fields >5 and <600 G
• Training: workers also to complete TQ-SMF
Log Exposures and observe exposure limits.
Engineering Controls
Work with fields de-energized if practical
Block access with barriers
Where practical, use non magnetic tools
Shield sources with high permeability materials (eg Mumetal)

Hazard:	**Ultraviolet sources >1 W** **Default Controls:** Task and source specific. Evaluate below.	**Negligible (0-20)**

E. Biological Hazards, Tasks and Controls
None

F. Offsite Work Hazards, Tasks and Controls
None

G. Other Issues (Security, Notifications to Other Organizations, Community Involvement, etc.)
None

H. Recommended Exposure Monitoring

- None

Description or comments:

I. EPHA Determination

Chemical Name	Quantity (lbs, gal)	Location (Bldg/Room#)

IV. Perform Work Within Controls

A. Recommended Training and Medical Surveillance Summary

- Electrical Safety for Benchtop Workers (TQ-ELECT-BENCHTOP)
- Laboratory Standard (HP-IND-220)
- Cryogen Safety (HP-OSH-025)
- Hazardous Waste Generator (HP-RCRIGEN3)
- Nanoparticles Protocol (OM-MEDSURV-NANO)
- Compressed Gas Safety (TQ-COMPGAS1)
- Nanotechnology for Nano-workers (TQ-NC-HS2)
- Oxygen Deficiency Hazard (TQ-ODH)
- Static Magnetic Fields (TQ-SMF)

B. Personnel Training, Qualification, and Authorization List

Employee/Guest Name	Life/Guest#	Dept	Required Training Course(s)	Signed
Fernando Camino	23799	NC	Cryogen Safety (HP-OSH-025) [EXPIRES: NEVER] Compressed Gas Safety (TQ-COMPGAS1) [EXPIRES: 3/4/2013] Laboratory Standard (HP-IND-220) [EXPIRES:	11/30/2011 4:53:52 PM

			8/24/2013] Nanotechnology for Nano-workers (TQ-NC-HS2) [EXPIRES: 4/29/2012] Static Magnetic Fields (TQ-SMF) [UNASSIGNED: EXPIRED: 9/10/2010] Hazardous Waste Generator (HP-RCRIGEN3) [EXPIRES: 8/17/2012] Nanoparticles Protocol (OM-MEDSURV-NANO) [UNASSIGNED: EXPIRES: NEVER] Electrical Safety for Benchtop Workers (TQ-ELECT-BENCHTOP) [EXPIRES: 8/31/2013]	
Chuck Black	23566	NC	Cryogen Safety (HP-OSH-025) [EXPIRES: NEVER] Compressed Gas Safety (TQ-COMPGAS1) [EXPIRES: 2/12/2013] Laboratory Standard (HP-IND-220) [EXPIRES: 11/15/2012] Nanotechnology for Nano-workers (TQ-NC-HS2) [EXPIRES: 6/4/2012] Static Magnetic Fields (TQ-SMF) [UNASSIGNED: EXPIRED: 12/15/2009] Hazardous Waste Generator (HP-RCRIGEN3) [EXPIRES: 11/15/2012] Nanoparticles Protocol (OM-MEDSURV-NANO) [UNASSIGNED: INCOMPLETE] Electrical Safety for Benchtop Workers (TQ-ELECT-BENCHTOP) [EXPIRES: 1/10/2013]	
Chang-Yong Nam	23659	NC	Cryogen Safety (HP-OSH-025) [EXPIRES: NEVER] Compressed Gas Safety (TQ-COMPGAS1) [EXPIRES: 3/4/2013] Laboratory Standard (HP-IND-220) [EXPIRES: 5/27/2013] Nanotechnology for Nano-workers (TQ-NC-HS2) [EXPIRES: 4/27/2012] Static Magnetic Fields (TQ-SMF) [UNASSIGNED: EXPIRED: 6/11/2010] Hazardous Waste Generator (HP-RCRIGEN3) [EXPIRES: 3/8/2012] Nanoparticles Protocol (OM-MEDSURV-NANO) [UNASSIGNED: EXPIRES: NEVER] Electrical Safety for Benchtop Workers (TQ-ELECT-BENCHTOP) [EXPIRES: 5/24/2013]	12/20/2011 2:52:52 PM
Young uk Jung	z8007		Cryogen Safety (HP-OSH-025) [UNASSIGNED: INCOMPLETE] Compressed Gas Safety (TQ-COMPGAS1) [UNASSIGNED: INCOMPLETE] Laboratory Standard (HP-IND-220) [UNASSIGNED: INCOMPLETE] Nanotechnology for Nano-workers (TQ-NC-HS2) [UNASSIGNED: INCOMPLETE] Static Magnetic Fields (TQ-SMF) [UNASSIGNED: INCOMPLETE] Hazardous Waste Generator (HP-RCRIGEN3) [UNASSIGNED: INCOMPLETE] Nanoparticles Protocol (OM-MEDSURV-NANO) [UNASSIGNED: INCOMPLETE] Electrical Safety for Benchtop Workers (TQ-ELECT-BENCHTOP) [UNASSIGNED: INCOMPLETE]	

C. Emergency Procedures

Folloow the Bldg. 735 Local Emergency Plan. Spill kits located in SE service galley and in the 90-day area. Eyewash and shower in SE service galley.

D. Transportation

None

E. Logistical Interactions

FACILITY PROJECT MANAGER: A. Piper. Extension: 5937, Cell: 631-258-5809 ES&H CORDINATOR: R. Sabatini. Extension: 3509 Cell: 631-294-0778 BUILDING OUTDOOR ASSEMBLY AREA: West Parking Lot by Catch Basin/Stone Wall BUILDING INDOOR ASSEMBLY AREA: Laboratory 1L01 BUILDING SHELTER- IN- PLACE AREA: Laboratory 1L01

F. Termination/Decommissioning

Closeout is not anticipated in the near future. At the time of closeout, guidance from ESH Coordinator, Facility Safety Representative, and Environmental Compliance Rep. will be sought.

V. Provide Feedback

• Cryogenic probe cooldown procedure has been fine-tuned using the experience gained after several experimental runs during the past year. Controls are various. One of them is a low pressure gauge at the vent intake of the LHe dewar in order to monitor the dewar pressure. This helps to perform consistent cooldowns and to avoid dewar over pressurization. • Potential risk for electrical shock in the operation of the optical probe station was discovered and corrected. • Static charge control are being implemented in order to minimize risk of sample and instrumentation damage.

VI. Attachments

Attached Files:

Experimental Equipment in Lab 1L-35.doc

1L35 SMF survey of Lakeshore cryo probe.pdf

1L35 SMF MEMO 12-13-07.doc

1L35_hydrogen_leak_test_20100402_REPORT.pdf

1L35-cooldown-SOP-121510.pdf

20110928 Oxygen doping experiment experimental procedure.docx