

# **Laboratory Instruction Manual**

## **Physics 133/219**

**Senior Advanced Undergraduate Laboratory  
Condensed Matter Physics-Material Science  
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# **I. INTRODUCTION**

## **A. Goals and Organization**

The purpose of the Senior Advanced Undergraduate Laboratory in Condensed Matter-Materials Science is to give you a feeling for the excitement of research in a modern condensed matter-materials science laboratory. To accomplish this purpose, the course is structured in such a way as to allow you much of the freedom you will encounter in a future research career. With guidance, you will formulate goals and methods of an experiment, design its specific aspects, perform the experiment and report on your results. If you are willing to invest the necessary time and effort, your work may lead to publication in a professional scientific journal. How far you carry your research will only depend on your dedication and inclination.

In this laboratory there will be no preset experiments or stations where you will repeat experiments done by other students before you. This will give you a feel for the environment of a graduate student or researcher in today's laboratory environment.

To perform this type of research you will have to:

1. Understand the basics of the problem
2. Research your problem in the library to learn the fundamental concepts needed to pursue the research
3. Define initial goals for your experiments
4. Learn the basics and the operation of the instrumentation you will use
5. Perform experimental measurements of relevant parameters
6. Develop the theoretical methodology to analyze your data
7. Keep a detailed research notebook
8. Write a paper in the format of a scientific letter
9. Give a 20 minute talk in front of a committee

In the pursuit of your research goals you will be given help in a variety of forms:

1. This manual will orient you and start you thinking about an experiment. Note that this is not a complete well-defined write-up where every detail has been presented.
2. You will have access to teaching assistants who are familiar and knowledgeable in the field. However, this does not mean that they know everything. The teaching assistants will be available in the laboratory Warren Lecture Hall 2133 and in their offices.
3. You will have access to Prof. Maple, who is an investigator in the field in which you are performing experiments. However, he may not know everything either. He will be available in the laboratory from time to time and by appointment in his office at 2310 Mayer Hall.
4. The lab will be open and staffed at the times scheduled on the syllabus. Students are prohibited from using the lab without the appropriate supervision of at least one TA or professor.
5. You may wish to discuss your problems, research results, etc., with anybody outside the formal course instructors. This includes other members of the university, your classmates, friends, etc.

You are expected to work in groups of two or three, which will be arranged at the beginning of the quarter. You are expected to collaborate on all aspects of the research including definition of the problem, using the equipment, maintaining proper research records, analyzing the data and writing the final paper. The best choice of a research partner is somebody that matches your schedule, lives close by so you can work together outside class, and perhaps more importantly somebody with whom you get along. It is expected that you will need to spend at least 8 - 12 hours a week for this course.

There will be a periodic evaluation of your research, the quality of your research records, and the speed at which your research is advancing by the teaching assistants together with the instructor. This is to be used as feedback information to improve your research. There will be an evaluation of your paper before the end of the quarter, the ninth week, by a number of referees, as is done in the real research world. Based on this report you may want to change or improve your manuscript, or respond to the criticisms of the referees. The final grade will be based partly upon your final report.

# **I. INTRODUCTION**

## **B. Equipment inventory**

All the facilities necessary to make polycrystalline high-T<sub>c</sub> samples are here, including chemicals, an electronic balance, mortars and pestles, a hydraulic press, a dc electromagnet, cleaning supplies, and furnaces. We have access to X-ray machines for characterizing samples. A tube furnace allows annealing of samples under flowing gas, such as oxygen. Soldering irons, silver paint, a diamond wheel saw, and a microscope allow samples to be prepared for measuring resistivity.

The Quantum Design SQUID MPMS2 magnetometer measures the magnetic moment of samples as a function of temperature and applied magnetic field (see below). Electrical resistivity can be measured in the closed cycle refrigerator to 10 K, or in the MPMS2 down to 1.8 K.

The magnetron sputterer can produce thin films of various metals. These films can be measured in the SQUID as well as under X-ray diffraction. Two guns are available for sputtering two targets (one at a time, provided they both work).

Computing facilities with plotting programs (Excel, Origin, and Kaleidagraph) are available for data analysis, but papers must be written using either your home computer, or one at a campus facility.

# **I. INTRODUCTION**

## **C. Safety Guidelines**

A modern laboratory requires the usage of equipment that may be potentially hazardous to your health or even lethal. It is impossible to set up modern equipment and pursue interesting research without any potential hazards. Because of this, you are required to familiarize yourself with all potential hazards, and to ALWAYS STRICTLY OBEY ALL THE SAFETY RULES. Violations of the safety rules may lead to termination of laboratory privileges as in a real research laboratory environment. You will be required to sign an agreement that you have read, understand and will obey all the safety rules. Without this agreement you will not be allowed to work in the laboratory.

Three general safety rules, which apply to all experiments, are:

- ° **Understand your equipment !!!**
- ° **Never work alone !!!**
- ° **Never disable or by-pass safety devices !!!**

### 1. HIGH VOLTAGE

All electrical power supplies are potentially dangerous, especially for some high voltage supplies present in the laboratory. The supplies may be dangerous even if they are turned off, since power supplies may have capacitors that take a while to discharge. When testing electronic circuits always keep one hand in your pocket so that if you get a shock it will not be across your chest. Never go barefoot in the laboratory. Always unplug all equipment if you need to test inside or change connections. Never poke or probe with any objects into electrical supplies.

**Remember current can kill !!!**

# **I. INTRODUCTION**

## **C. Safety Guidelines (con't)**

### 2. CRYOGENICS

Cryogenic liquids and cold surfaces may be potentially dangerous. Liquid helium and nitrogen are chemically inert, however may cause severe frostbite or asphyxiation if there is inadequate ventilation. Wear safety glasses and gloves when handling any cryogenic liquids or surfaces. Never leave caps of cryogenic dewars open, since air may condense in the neck of the dewar, leading to a build up of pressure, and then an explosion.

**Remember some cryogenic liquids burn and may explode!!!**

### 3. X-RAYS

You will perform X-ray diffraction ALWAYS together with a teaching instructor and NEVER ON YOUR OWN. During data acquisition, the X-ray apparatus is configured so as to avoid exposure of the operator to dangerous X-rays. The safety interlocks should never be bypassed. Never stick any part of your body in the X-ray beam. Always wear safety badges, dosimeters, and monitors.

**Remember radiation exposure is cumulative and can cause permanent damage!!!**

# **I. INTRODUCTION**

## **C. Safety Guidelines (con't)**

### **4. CHEMICALS**

Some of the chemicals you will be dealing with are highly toxic. Before using any new material, look up its properties in *Dangerous Properties of Materials* by N. Irving Sax or a similar text, or look up the Materials Safety Data Sheets (MSDS) which can be found on the EH&S website (<http://www-ehs.ucsd.edu>). Most of the chemicals you will be using are in powder form, which can be readily inhaled and can stick to your skin and clothing. Avoid creating airborne dust when handling the powders and avoid contact with your skin. The powders can cause irritation of the eyes, nose and respiratory tract and cause serious side effects if ingested. Whenever you handle these materials it is extremely important that you:

1. Wear gloves
2. Wear a dust mask
3. Do not touch your eyes, nose or mouth until hands have been thoroughly washed with soap and water
4. Dispose of wastes properly
5. Never eat or drink in areas where the chemicals are handled
6. Wash your hands and forearms after work, even if you were wearing gloves.

When using acetone, avoid breathing the vapors. Some people have been known to experience headaches and/or dizziness from inhaling acetone fumes. Should you experience dizziness at any time, remove yourself to fresh air. Read the attached sheet on the effects of lead poisoning and realize that these dangers apply to all heavy metals.

When you are finished working with chemicals, be sure to always clean up after yourself. Remove all traces of dust so that the next person using the lab is not unwittingly exposed to hazardous chemicals.



# **I. INTRODUCTION**

## **C. Safety Guidelines (con't)**

### 5. FURNACES

The furnaces you will be working with operate at extremely high temperatures (up to  $\sim 1200^\circ\text{C}$ ), so you will need to take some common sense precautions when dealing with them. Never leave flammable materials (such as Kimwipes, paper or acetone) near the furnaces. Always use tongs to remove hot objects from the furnaces, and always place hot objects on the fire bricks. *Never* place them directly on the table top! Also note the location of the nearest fire extinguisher in case of a fire.

### 6. HYDRAULIC PRESS

The hydraulic press can subject materials to extremely high pressures. Whenever you pressurize a material, *always have the protective shield closed and latched!* If the material should give, pieces of metal can shoot out at high speeds and cause serious damage. Always be sure that the pressure is completely released before opening the door.

### 7. EMERGENCIES

In case of an emergency or accident, contact one of the following people:

Campus police

Kevin Huang	534-2493	Mayer Hall 2110	khuang@physics.ucsd.edu
Colin McElroy	534-2493	Mayer Hall 2118	cmcelroy@physics.ucsd.edu
M. Brian Maple	534-3968	Mayer Hall 2310	mbm@physics.ucsd.edu

If there is a problem with the lab facilities and the TA's are not available then contact Jeff Patterson (jpatters@physics.ucsd.edu) at 822-5031.

## **II. Getting started**

In this laboratory, we have the capability to make ceramic oxide materials, such as high-Tc superconductors, and thin metallic films. You will need to do some research in the library to get a feel for what kind of experiments are done, what interests you, and what you have the capability to do in this laboratory in a 9 week class.

Start by getting a book on superconductivity, then find some review papers - there are several by Prof. Maple which are very interesting and relevant to this class. Ivan Schuller has written many review papers on thin films that may be quite relevant, and you may go to ask his assistance in choosing a project. Physica C is a good journal of superconductivity, Physica B and Physical Review B are condensed matter journals, Physical Review Letters is the most prestigious journal, but you may not find it very useful for this purpose. There are also journals of magnetic materials and materials science that may help. The more time you spend choosing your project carefully in the beginning, the better your final product will be.

### **III. Some example projects**

The most common experiments to do on the high-Tc's are chemical substitutions into well known superconductors, such as  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ ,  $\text{La}_2\text{CuO}_4$ , etc. You may substitute an element for one of these elements, for example, substitute Pr for Y, or Zn or Fe for Cu. You can control the amount of "impurity" that you substitute in, as well as the oxygen content, both of which affect the superconducting properties. Your project may emphasize making a lot of samples, or making a few samples with many measurements.

You may also explore "conventional" materials that have interesting magnetic properties such as iron oxide (although preparation of a good sample may be difficult in this particular case). One of the hot topics in recent years has been the giant magnetoresistive (GMR) materials, which include  $\text{LaMnO}_3$ . These materials exhibit a relatively large change in their electrical resistance when a magnetic field is applied, which makes them potentially useful for magnetic recording, or as a magnetic field sensor. You may also consider making a device out of one of these materials, such as a magnetic field sensor or superconducting transistor, but the primary emphasis should be on the material itself.

There are many interesting phenomena in the thin film realm as well. The magnetron sputterer can make thin films of metals. It is difficult to sputter semiconductors, and more so for oxides. It is possible to sputter a high-Tc oxide, but it can be difficult. At present, we have Nb, Cu, Co, and Fe targets, but we can order whatever targets you like. You may also study the effects of oxide layers, different sputtering conditions, and film thickness. Follow a similar research path as outlined above. It is possible to sputter a superconducting metal such as Nb, but this may require a cleaner environment than our chamber offers - see what the literature says.

There are also other preparation facilities available that require special TA assistance. These include arc furnaces for metallic polycrystal growth, a pulsed laser deposition facility for thin films, and facilities for single crystal growth from flux. If you are interested in a material requiring any of these methods, ask your instructor for more information.

## Example Projects:

1. **Transport measurements of various materials:** Study the electrical resistivity of a number of substances: ordinary metal, semiconductor, insulator, conventional superconductor, high Tc ceramic superconductor. Make at least one sample of each of these, measure their electrical resistivity as a function of temperature, and compare your results to those of other researchers. Compare and contrast the results and discuss the different microscopic mechanisms responsible for the electron transport in these systems.
2. **The role of oxygen in  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ :** Make some ceramic samples by reaction of barium carbonate, yttrium-, and copper-oxide, then systematically vary the oxygen content by annealing the samples under flowing gas. Use X-ray diffraction to determine the crystal structure and lattice parameters, and measure superconducting properties using the SQUID magnetometer and/or the closed cycle refrigerator as a function of oxygen content.
3. **Chemical substitution in YBCO:** Study the superconducting properties of YBCO as a function of chemical doping, either on the yttrium site or on the copper site. What effect do different elements, magnetic or nonmagnetic, have on the magnetization, lattice parameters?
4. **Effect of film thickness on transport and magnetic properties.**

## A. Magnetic properties of superconductors

Although there are many interesting properties of superconductors one could investigate, magnetic properties of superconductors are particularly unique and one can potentially obtain a lot of information about the superconductor from them. The equipment in this lab will enable you to make a high  $T_C$  superconductor (HTSC) and measure its magnetic properties using a state-of-the-art SQUID magnetometer (which itself utilizes some of the unique magnetic properties of superconductors for its operation).

Many materials are superconducting, including many of the elements of the periodic table - such common metals as tin, lead, aluminum, and mercury. Literally thousands of superconducting alloys have been discovered; yet there probably remain many more to be discovered. At present there is no way to predict what materials should be superconducting - the materials are just too complex. There even exist organic superconductors and, of course, oxide superconductors which include the high- $T_C$  copper-oxides.

There are many avenues your research project could take. One of the most basic properties you could investigate initially is the Meissner effect, which is unique to superconductors. Below the superconducting critical temperature  $T_C$ , a superconductor will completely expel a low applied magnetic field, resulting in a perfect (volume) diamagnetic susceptibility ( $\chi = -1/4\pi$ ) which you could measure in the magnetometer. No other property of matter will yield such a large diamagnetic signal. Yet even such a basic property as the Meissner effect is not as straightforward as some textbooks might lead you to believe. For example, you might try to measure the magnetic susceptibility  $\chi$  in the following way. First cool the sample in zero applied magnetic field  $H_a$  to a temperature below  $T_c$ . Then apply a low field  $H_a$  (say, 10 Oe), and collect data as the temperature  $T$  is increased to above  $T_c$ . The resulting curve is referred to as the zero-field-cooled ZFC or screening susceptibility. In this case the field is excluded from the sample due to screening currents on the surface of the sample. But if you then cool the sample from above  $T_c$  to low temperatures note that data yields a smaller diamagnetic signal. In this case, the field must be expelled from the interior of the sample. This is called the field-cooled FC or Meissner signal. How would a perfect conductor behave? How would a superconductor with a hole in the middle behave?

There are other phenomena that can cause irreversible magnetic behavior in superconductors. For Type II superconductors such as  $PbMo_6S_8$ , flux pinning is such a phenomenon. You might want to measure the Meissner effect for a Type I superconductor like Pb or Sn, or for a HTSC. Is  $\chi(T)$  irreversible for these materials?

Because the superconducting signal is so large, the demagnetization factor cannot be neglected - the magnetization depends on the shape of the sample. Information on the

demagnetization factor can be found in Cullity, Shoenberg, and Kittel (see reference list). In the figure, if the sample weighed 0.2 grams and was measured in a field of 5 Oe, what is the demagnetization factor if you assume the sample is 100% superconducting? (Assume also that the volume of the unit cell is  $800 \text{ \AA}^3$  and contains 3 formula units.) What factors might broaden the transition just below  $T_C$ ? What if the size of the particles you are measuring are on the order of the magnetic penetration depth  $\lambda$ ?

Another useful type of measurement one can make is a magnetization isotherm. If you perform a  $M(H)$  run for a HTSC, you will find that it is highly irreversible. Again, this is due to flux pinning. The irreversible contribution to  $M(H)$  can be understood in terms of Bean's critical-state model (see Tinkham). Using this model, one can extract the critical current density  $J_C(H)$  from the  $M(H)$  hysteresis. How do you think this value will compare to the transport critical current density obtained by passing an external current through the sample?

Studying the problem further, you might notice that under certain conditions, the magnetization in the superconducting state has a time dependence. This occurs in the mixed state of Type II superconductors due to thermal activation of pinned magnetic flux vortices, which leads to *flux creep*. In the fully critical state, the relaxation is logarithmic in time and one defines a relaxation rate  $S$ , which depends on the flux pinning energies (within a thermal activation model). Prior to HTSC, little research was done in this area. But because giant flux creep in the HTSC's limits their usefulness in practical applications, a large amount of experimental and theoretical work is currently being done on flux vortex dynamics. Much, if not most, of the magnetic relaxation measurements have been done using commercial SQUID magnetometers like the QD MPMS in this laboratory.

One must be careful in interpreting data taken on a machine deemed as a "black box". You should become familiar with the basic principle of operation of the SQUID magnetometer. (See handout or manuals of the QD MPMS.) Of special concern are (1) finite remnant magnetic fields of the superconducting magnet; (2) cycling the sample in an inhomogeneous field; (3) transverse magnetic moments; (4) external noise sources; (5) magnetic impurity contributions.

So, there are many avenues one could take simply by making different materials and measuring various magnetic properties. What we have presented to you so far are only suggestions on how to get started in this fascinating field.

## REFERENCES

For general introduction to Solid State Physics, including superconductivity, magnetism, and x-ray diffraction:

1. C. Kittel, Introduction to Solid State Physics, 5th ed. (Wiley, New York, 1976).
2. N. W. Ashcroft and N. D. Mermin, Solid State Physics, (Saunders College, Philadelphia, 1976).

#### MAGNETIC PROPERTIES OF SUPERCONDUCTORS:

3. M. Tinkham, Introduction to Superconductivity, (Krieger, Malabar, Fl., 1985).
4. D. Shoenberg, Superconductivity, 2nd ed. (Cambridge University Press, New York, 1960).
5. E. A. Lynton, Superconductivity, (Wiley, New York, 1962).

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6. "Superconductivity in Copper Oxide Compounds - Prospects and Perspectives", M. B. Maple, in Superconductivity and its Applications, ed. H. S. Kwok and D. T. Shaw (Elsevier, 1988), p. 478.
7. " Superconductors Beyond 1-2-3", R. J. Cava, *Sci. Am.*, Aug. 1990, p. 42.
8. "Demonstrating Superconductivity at Liquid Nitrogen Temperatures", E. A. Early, C. L. Seaman, K. N. Yang, and M. B. Maple, *Am. J. Phys.* **56** (7), 617 (1988).
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#### MAGNETISM:

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#### X-RAYS:

11. J. Als-Nielsen and D. McMorrow, Elements of Modern X-Ray Physics, (John Wiley & Sons, New York, 2001).

#### LOW TEMPERATURE MEASUREMENTS:

12. F. Pobell, Matter and Methods at Low Temperatures, (Springer, New York, 1996).

## B. Magnetic flux relaxation in $Y_{1-x}M_xBa_2Cu_3O_7$ ; ( $M = Pr, Ho, Ca$ )

The discovery of high temperature superconductivity in copper oxide ceramic materials has generated a great deal of interest in the scientific community. These materials have many unusual properties that have yet to be completely understood. Among these are incredibly high superconducting critical temperatures  $T_c$  as high as 160 K (exceeding the boiling point of liquid nitrogen  $LN_2 = 77$  K), and extremely large upper critical fields  $H_{c2}$  estimated to be as high as 150 tesla at  $T = 0$ . These two properties in particular give these materials the potential to profoundly impact technology in the future in both low and high current applications.

Perhaps the most formidable obstacle preventing the widespread use of these materials in high-current applications is their relatively low transport critical current density  $J_c$ , especially at higher temperatures and in the presence of high magnetic fields. For polycrystalline samples,  $J_c$  is typically  $10^2 - 10^3$  A/cm<sup>2</sup> at 77 K in zero magnetic field. Current densities are limited by thermally activated flux motion due to low flux pinning energies; in polycrystalline materials, intergranular current flow is further limited by Josephson-like weak links at the grain boundaries. Flux pinning in superconductors is caused by local variations in the superconducting parameters, which cause certain sites to become more energetically favorable for flux lines. The critical current density can be increased by the introduction of suitable flux-pinning centers, such as defects and twin boundaries. In this research project, the effects of chemical doping on flux pinning in  $YBa_2Cu_3O_7$  will be investigated.

Initial efforts will be focused on the system  $Y_{1-x}Pr_xBa_2Cu_3O_7$ . Pr is the only rare-earth element that forms the orthorhombic  $RBa_2Cu_3O_7$  crystal structure (R is any rare-earth element, except for Ce, Tb, and Pm which so far do not form this structure) and yet does not yield superconductivity. As Pr is substituted for Y in  $Y_{1-x}Pr_xBa_2Cu_3O_7$ , there is a monotonic decrease in  $T_c$  with increasing Pr concentration  $x$ , and superconductivity is completely destroyed for  $x > 0.56$ . However, the depression of  $T_c$  is only slight for  $x < 0.1$ , and the substitution of Pr for Y could introduce flux-pinning centers without significantly degrading superconductivity. Preliminary magnetic relaxation measurements indicate that the pinning energies do, in fact, increase with the Pr concentration  $x$ . These results may also have implications about the



mechanism of  $T_c$  suppression in this system, which is still a matter of controversy in the scientific community.

The effectiveness of chemical impurities other than Pr as pinning centers will also be investigated in order to elucidate the aspect of Pr that causes the observed increase in pinning energy. Initially, Ca, which is divalent and nonmagnetic, and Ho, which is trivalent and magnetic, will be substituted for Y (trivalent). Thus, this project involves both basic and applied research of  $R\text{Ba}_2\text{Cu}_3\text{O}_7$  high- $T_c$  superconducting materials.

### Experiments:

The approach we suggest is the following:

(1) Make polycrystalline ceramic samples of  $\text{Y}_{1-x}\text{M}_x\text{Ba}_2\text{Cu}_3\text{O}_7$ . The resulting cylindrical pellets will be cut into various specimens for the measurements.

(2) The samples will be characterized by measuring X-ray diffraction spectra and low field magnetic susceptibility. These measurements utilize state-of-the-art equipment that is presently being used in research.

(3) Various measurements can be made to determine  $J_c$  and the flux pinning energies of the samples. These include magnetic hysteresis loops  $M(H)$  at constant temperature  $T < T_c$ , and magnetic time relaxation  $M(t)$  at constant  $T$  and  $H$ .

## C. Thin Films

Thin film techniques form the basis of all modern microelectronic technology and science. A number of thin film techniques are used to prepare integrated circuits, X-ray mirrors, optoelectronic devices, etc. These include Molecular Beam Epitaxy (MBE), Chemical Vapor Deposition (CVD), and sputtering. The thin film deposition technique used in the lab will be by magnetron sputtering. Given below is a brief description of the sputtering process and experimental procedure. Later in the section is a description of the Researcher 101 sputtering system, and its basic operation instructions.

For a more detailed description of the sputtering process see Thin Film Processes edited by Vossen and Kern.

### Structural Characterization

The first problem in any thin film experiment is to understand the structure of the film. The two main structural parameters that determine the physics of a material are the crystal structure (atomic ordering) and the film thickness. Because sputtered films are not grown in thermodynamic equilibrium, the structure depends sensitively on the growth conditions. X-ray diffraction is an excellent technique for determining structural parameters. X-ray diffraction is commonly used to study the crystal structure and is described in any solid state book (i.e. Kittel, Introduction to Solid State Physics, Ashcroft and Mermin, Solid State Physics) or any introductory X-ray diffraction book. A very good book on the basics of X-ray diffraction is by B. D. Cullity, Elements of X-Ray Diffraction. Low angle X-ray diffraction can also give an accurate measure of the film thickness and the flatness of the film as described in the Chapter 8 of Thin Films From Free Atoms and Particles, edited by Kenneth J. Klabunde. This chapter also includes information on determining the crystal structure of thin films.

The first step is to deposit some thin film and determine the crystalline structure. The easiest parameter to change in the sputtering procedure is the Ar pressure during sputtering. The Ar pressure in the chamber will change the kinetics of growth considerably. To understand why, see K. Meyer, I.K. Schuller, and C. M. Falco J. Appl. Phys. **52**, 5803 (1981). By changing the growth conditions, it is possible to optimize the properties of the films. Some aspects of the structure can be determined. Suggestions are given below in order of increasing complexity.

- 1) crystal structure and lattice constants ( bcc or fcc ?)  
crystal orientation?  
film thickness ?

By determining the film thickness, the sputtering rate of the gun for a given power can be determined which allows you to sputter films of specific thickness in future experiments. Under similar conditions (Ar pressure, substrate to target distance, target, etc. kept constant) the sputtering rate should be proportional to the sputtering power, although the best way to control the film thickness is to determine the rate for a given sputtering condition and vary the sputtering time.

- 2) crystalline coherence length?  
are there grains? (size and orientation?)  
standard deviation of film thickness?
- 3) surface oxide layer or film-substrate interaction?  
lattice strains?  
in-plane orientation?  
diffuse scattering?

To determine many of the structural parameters of the film requires comparing the measured x-ray spectra to model calculations. An example of a low angle spectrum of a sputtered Nb film is shown in figure 3. Low angle spectra can be simulated by using an optical formalism described by Underwood and Barbee, *Appl. Opt.* **20**, 3027 (1981). This formalism can be extended to include layers that are not flat, by calculating the low angle spectra for all the possible thicknesses and averaging the intensities to determine the final intensity scans. The lab is equipped with a 386 SX PC computer, which has enough power to do most of these calculations. By adjusting the distribution of layer thicknesses and comparing to the measured spectra, an estimate of the film flatness can be determined. For an example see J.M. Baribeau, *Appl. Phys. Lett.* **57**, 1748 (1990).

### Superconducting properties

The targets in the sputtering guns are presently Nb and Al. The lab has a SQUID magnetometer that can be used to measure the superconducting transition temperature of Nb films. For relevant work on the superconducting properties of Nb films, see, S. A. Wolf, J.J. Kennedy, and M. Nisenoff, *J. Vac. Sci. Technol.* **13**, 145 (1976) or A. F. Mayadas, R.B. Laibowitz, and J.J. Cuomo, *J. Appl. Phys.* **43**, 1287 (1972). The effect of film structure on the

superconducting properties can be studied, in particular how does grain size and film thickness affect the superconducting transition.

### Multilayered structures

There are two sputtering guns in the chamber, which allows the preparation of layered structures. Once a single film is understood, the easiest extension is to study the structure and superconducting properties of a Nb/A/Nb sandwich where A is another material. There are a number of possible materials that can be chosen for A including Al, Cu, Ag, W, Si, Ge, and Ni. For a complete list of available targets, check with the instructor. The main question is - how does the spacer layer affect the growth of the films and the superconducting properties of the Nb layers? Will a metallic or semiconductor layer affect the superconductivity differently? If a small amount of magnetic material is included in the spacer layer, how is the superconductivity affected?

## **IV. EQUIPMENT GUIDELINES**

### **A. Quantum Design SQUID Magnetometer: Magnetic Property Measurement System (MPMS2)**

1. Principles of operation: The MPMS2 is a commercial SQUID (Superconducting QUantum Interference Device) magnetometer that measures the magnetic moment of a sample at a controlled temperature (between 1.7 K and 400 K) in an applied magnetic field (up to  $\pm 7$  tesla). Most operation steps are performed through a user-friendly, menu-driven software package that runs on a personal computer, which interfaces to the electronic equipment that drives the hardware. The operation of this machine can be broken down into various parts: sample transport, temperature (T) control and gas handling system, magnetic field (H) control, and signal detection electronics. The details can be found in the fairly comprehensive manuals provided by QD, but the basic principle of operation is as follows.

The sample is dragged through superconducting (SC) pickup coils, which are wound in a second derivative arrangement. Magnetic flux that passes through a SC coil will induce a persistent current in the coils. This current drives a separated small SC coil, which produces a magnetic field that is sensed by the SQUID. In conjunction with the RF circuitry, the SQUID acts as a magnetic flux transducer, producing an output voltage signal proportional to the magnetic moment  $\mathbf{m}$  that produced the flux. The absolute value of  $\mathbf{m}$  can be calibrated by measuring a standard sample of known moment (such as Pt or Pd). By cycling the sample through a second derivative coil arrangement, contributions from electronic drift are greatly reduced, as are any constant external field (such as the earth's magnetic field) and any constant gradient field contributions. A stepping motor raises the sample from below the pickup coils, to above them, and the voltage is digitally recorded at a specified number of intervals as the sample passes through the coils. Due to the second derivative arrangement, a plot of voltage vs height yields a characteristic shape (see Fig. 4 on pg. 16 in the User's manual). The center peak occurs at the center of the coil arrangement that consists of 2 windings wound in the positive sense. Each of the two minima on either side of the center peak occurs near a single winding, wound in the negative sense and placed in series with the center windings. The total voltage is the sum of the flux contributions from each winding. The measured "scan" is then fit to a theoretical curve for an ideal dipole moment to determine the magnetic moment of the

sample. Samples that are long compared to the pickup coil diameter ( $\sim 1$  cm) will yield an error in the calculated moment. Samples that are not spherical will have higher multipole moments. You should convince yourself that a second derivative coil type arrangement will not give a voltage for a uniform field and/or constant gradient field.

During the measurements, the sample tube (9 mm ID) contains  $\sim 2$  torr (1 atm. = 760 torr) of He exchange gas, providing good thermal contact with the outer cooling annulus. The cooling annulus is cooled by cold ( $\sim 4.5$  K) He gas flowing through it from the bottom. The power to a resistance heater is altered to control the temperature at a constant value. The temperature is measured by a four-wire ac resistance technique using either a platinum resistor ( $T > \sim 40$  K) or a germanium resistor ( $T < \sim 40$  K), which are isolated from the magnetic field due to the SC magnet, but still are in good thermal contact with the sample tube. To reach temperatures below  $\sim 4.5$  K, liquid He (LHe) is collected at the bottom of the cooling annulus and subsequently pumped by the mechanical pump, which reduces the helium vapor pressure and therefore the boiling point of the LHe. There is a vacuum space surrounding the cooling annulus, which isolates it from the LHe bath (dewar) which keeps the entire cryostat (insert) cold, including the superconducting magnet. An outer vacuum jacket isolates the LHe bath from room temperature, and is superinsulated with aluminized mylar which acts as a thermal shield.

The superconducting magnet is wound in the shape of a solenoid and can attain fields as high as 7 tesla (70,000 gauss). The field is sufficiently uniform only in a small region about the center of the pickup coils. Field homogeneity is of particular concern when cycling hysteretic superconducting samples. The magnet is energized by a 50 amp power supply. When the desired current to the magnet is reached, the "persistent switch" is closed; that is, the magnet is short circuited by a SC wire, and the power supply current is ramped down. The current in the magnet is maintained because of its zero resistance and high inductance ( $\sim 5$  henries). Special care must be taken with SC magnets to prevent a "quench". If any part of the SC magnet goes into the normal state (due, for example, to an insufficient amount of LHe in the dewar), creating a region of finite resistance, the high current will generate more heat and the entire magnet will be driven into the normal state in a small amount of time. The current will then decay to zero and, due to the large inductance of the magnet, large voltages on the order of kV can potentially be induced across the windings of the magnet. This can cause arcing and destruction of the expensive magnet. There are, however, protective diodes across the magnet that should limit the voltage to a safe value. In any case, a large amount of expensive LHe will

evaporate very quickly. To prevent a quench, you should not exceed the recommended maximum field for the various levels of LHe. Above 50%, you can go to the maximum value of 7 T. You should also program each run to ramp the field to zero at the end.

2. Basic operation:

\* Check the LHe level to make sure you can reach the fields you need:

<u>LHe level (%)</u>	<u>Max. recommended field</u>
> 50	70 kOe
40 - 50	10 kOe
30 - 40	1 kOe
20 - 30	100 Oe

Normally, the TA will transfer helium as needed, but if the level is below 50%, you should mention it to the TA. Do not apply a field greater than the above specified limits, or the magnet could quench, potentially causing irreparable damage to the SQUID.

a. Log into the lab notebook and record:

1. Your name.
2. Filename. This is the date you start the sequence plus a letter (a, b, c, etc.) indicating the order of that run on that date. This will assure that every data file will be stored (on the harddisk) with a unique filename. Be sure to change the filename on the computer.
3. Liquid helium level. (It automatically updates every hour)
4. Max field you will apply. Indicate if you demagnetize the magnet by oscillating the field to zero from a high value > 5 kOe.
5. Sample being measured - its chemical formula and code which identifies it.
6. Any unusual behavior.

b. Mount the sample:

When making magnetic measurements, you should be aware that virtually every solid material has a finite magnetic susceptibility, although some are larger than others. The sample holders will contribute to your signal, impurities within and on the surface of your sample will also contribute. At the very least there is a small Landau diamagnetism; metals also have Pauli paramagnetism. Of special concern are paramagnetic or even ferromagnetic impurity contributions, which become large at low temperatures.

Therefore you should handle samples and sample holders with gloves and keep them clean. Measurements of samples with small moments are often difficult because of these other contributions.

Place the sample in a gelatin capsule with some cotton or a kimwipe piece to hold it in place. To prevent it from opening while purging, puncture a small hole in the top of the capsule with a needle or scalpel. The gel cap is then inserted into the center of a straw and held in place with 2 small pieces of a similar straw. The magnetization due to a uniform rod which is longer than the span of the pickup coils (such as the straw) will produce a constant flux, and does not contribute to the calculated magnetic moment.

Stick the straw onto the end of the sample rod, making sure that there is enough teflon tape that the straw will not come off. Puncture another hole near the top of the straw to allow gas to enter easily into the region above the capsule. Then slide the quartz tube assembly over the straw.

c. Load the sample (see pg. 15 of User's manual):

The manually controlled airlock valve should be closed - make sure that it is. Vent the airlock. Then remove the blue top fitting on the cryostat. If you cannot remove the top blue fitting easily, there is probably a vacuum in the airlock and you must go to the computer menu to vent helium gas into the airlock.

Be very careful with the sample rod, especially at the joint where the stainless steel and brass are welded, which is extremely fragile. Carefully place the sample holder in the airlock, turning the black clips and tightening them with an allen wrench. Make sure that the white dot on the blue piece faces forward - it does matter! Tighten the black screws to the sample transport assembly, and LOOSEN the steel knurled nut.

Once the sample holder is in place, purge the top chamber by pressing the black purge button. Repeat this three times. This prevents air from being introduced into the sample tube. One wants especially to minimize the amount of oxygen contamination (which is paramagnetic), which can significantly contribute to the signal at low temperatures. Also, ice could accumulate and cause problems. If the sample tube does become contaminated (or if a sample falls to the bottom of the sample tube), it can be heated to  $\sim 320$  K and purged with helium, i.e. with the airlock valve open.



Upon completion of the purging, open the airlock valve and lower the sample support rod slowly into the sample tube until ~2 cm of the rod is left. Monitor the temperature while you do this; it will increase slightly.

d. Center the sample

Apply an appropriate field (~10 Oe – I always use 1 T) to get a moment large enough to allow you to center the sample; then center the sample using the instructions in the software. At this point, the sample is ready for measurement.

e. Set T and H to initial conditions

f. Program the sequence of the run

While the temperature is settling, you can program the sequence of the run. Complete instructions can be found in the User's manual. At the end of the sequence, set the temperature to ~10 K (to save LHe) and the field to zero (to prevent possible quench).

g. Set all experimental parameters

Here are some suggestions:

MPMS EXECUTIVE MENU:

1. Use magnetic mode OSCILLATE unless you are measuring hysteretic samples. The field is more stable in time when you oscillate to the final value. Also, the remnant field (due to trapped flux in this hard, Type II superconductor) will be smaller.
2. Use temperature mode UNDERCOOL ON for faster cooling, unless you are concerned about hysteretic effects.
3. MAG HIGH-RESOLUTION (ENABLE)

COLLECT DATA MENU:

1. Use iterative regression mode algorithm for determining the moment.
2. Use 3 cm scan length
3. Take 32 data points per scan & record 32 readings per point.
4. Average 3 scans.

5. Longitudinal autorange ON
6. If there is plenty of printer paper, Hardcopy ON & Plot after final scan.
7. Make sure you change the filename.

h. Start the run

Although the second derivative coil arrangement will cancel constant  $H$  and  $dH/dx$ , it is very sensitive to external magnetic fields which change in time. Therefore, while a measurement is being made, activity near the QD MPMS should be minimized. You especially do not want to be moving large magnetic objects like LHe storage dewars or gas cylinders. You can verify the effect yourself by monitoring the voltage signal  $V$  vs time  $t$  on a sufficiently sensitive scale. Go into the PLOT&DISPLAY menu from the MPMS EXECUTIVE menu. Plot  $V$  vs  $t$  in real time and roll a gas cylinder near the MPMS. What moment does the increase in  $V$  correspond to?

Be aware that external RF sources can also produce noise in the system. Although we do not have one for the MPMS, a magnetic shield would greatly reduce these types of problems.

- i. After the run is finished: Set  $T=10$  K and  $H = 0$  to conserve helium and as a courtesy to the next user. Loosen the knurled steel nut on the sample rod and pull the rod up slowly. When you feel resistance, do not force the rod up - wait just a few seconds for the slide seal to warm up, then you can pull up some more. When the rod is all the way up, you can see your sample in the airlock. CLOSE THE AIRLOCK VALVE, then remove the sample rod. Replace the blue plug.
- j. Retrieve your data: copy the data file with the \*.DAT extension to a floppy disk. This produces data in a column format that you can then analyze with any spreadsheet program.

## B. Researcher 101 Sputtering Unit

### 1. Principles of operation

The material that is going to be sputtered is placed in the target position in the sputtering gun. The vacuum chamber is evacuated, and then filled with an inert gas, which in most cases is Ar. The Ar pressure used is typically 3 mTorr. A large negative voltage, of the order of -1000 kV, is then applied to the target. This voltage will accelerate the few positive Ar ions present in the gas towards the target. When the ions strike the target, atoms of the target material are knocked off the target along with secondary electrons. The electrons are accelerated away from the target and spiral about the magnetic field produced by the permanent magnets in the gun. The electrons collide with the neutral Ar atoms causing more ions that accelerate towards the target causing more electrons and so on. This process avalanches and a plasma or glow discharge is formed over the target where the number of electrons produced is sufficient to produce enough ions to generate the same number of electrons. When this occurs, the voltage required to maintain the plasma drops to approximately -300 kV and a current flows into the target to replace the emitted electrons. The plasma is confined by two things: the magnetic field which confines the emitted electrons, and the ground shield around the gun which limits the sputtering process above the target. Atoms from the target are ejected by this ion collision, pass up from the target and condense on the substrate above the gun, hence producing a thin film with the composition of the target.

The sputtering rate (the amount of target material deposited on the substrate per second) depends on many parameters including Ar pressure, substrate distance, target power and target material, but is typically in the range of 2 - 20 Å/sec. It is impossible to determine the sputtering rate directly from the sputtering parameters but instead must be measured. This system is not equipped to measure the rates directly so the guns need to be calibrated. Determining the rates will be discussed later.

Magnetron sputtering works best for depositing non-magnetic metallic materials. If a material is an insulator, then it is impossible to replace the emitted secondary electrons and the plasma will not form. Semiconducting materials will sputter, but tend to run with high voltages, low currents, and low deposition rates. Magnetic materials also tend to cause problems in the sputtering process. The magnetic field from the magnet in the sputtering gun is distorted by the magnetic target. If the target is too thick, then the magnetic field will be completely contained

within the target. Magnetic materials are best sputtered by removing the center set of magnets and making the target very thin (app 1/16").

## 2. Components

Shown in figure 4 is a top view of the sputtering system showing most of the major components, pressure gauges and valves. Most of the components are controlled from the control panel at the front of the machine. The main exception is the gate valve, which is opened and closed by hand using the handle on top of the valve.

### a. Pumps

1. Mechanical Pump (also called the roughing pump): Power to the mechanical pump is controlled from the front panel. The mechanical pump works in pressure ranges from atmosphere down to  $\sim 10^{-2}$  Torr. It serves two purposes; (1) to rough pump the main chamber to a pressure below  $10^{-1}$  Torr and (2) to back the turbo pump which runs at lower pressures. This is why the mechanical pump can pump on either the chamber or the back of the turbo pump.
2. Turbo Pump: The turbo pump works in pressure ranges of  $10^{-1}$  to  $10^{-8}$  Torr. The mechanical pump is used to keep the pressure behind the turbo pump below  $10^{-1}$  Torr. The turbo pump should not be exposed to pressures above 1 Torr. Power to the turbo pump is controlled by the front panel. To start the turbo pump requires hitting the start switch on the turbo pump control box.

b. Valves: All valve switches on control panel are closed when the light is off and open when the light is on.

1. Gate valve: The manually controlled gate valve separates the turbo pump from the main chamber. This valve should be closed whenever the pressure in the chamber is going to be above 100 mTorr. (see venting chamber below)
2. Turbo back valve and Chamber rough valve: These valves control where the mechanical pump is directed. These valves should not be open simultaneously. The turbo back valve can only be closed when the gate valve is closed and has to be open when the gate valve is open.

3. Chamber vent valve: used to vent the main chamber and bring it to atmosphere. The gate valve has to be closed before this is opened (see vent chamber below)
4. Turbo vent valve: This opens the turbo pump to atmosphere. It is used to slow the turbo pump down when the power is switch off to the pump. NEVER use this valve when the turbo pump is running or you will destroy the pump.

c. Pressure Gauges:

1. Ion Gauge: reads pressures from  $10^{-3}$  -  $10^{-10}$  Torr. The gauge will shut off if the pressure in the chamber is too high. Hitting the ion gauge 1 button on the front control power turns the power on the ion gauge.
2. Pressure Gauge A and B: reads pressures from  $10^{-4}$  to 10 Torr. Gauge B measures the pressure in the chamber and gauge A reads the pressure behind the turbo pump. The zero position of Gauge B will drift from zero with time, so it should be checked relative to the ion gauge from time to time. See the TA on how to zero the gauge.

d. Hoist: lifts the top of the chamber and is controlled from the front panel.

e. Chamber door: has an O-ring seal that allows quick access to the chamber.

3. Basic Operation:

This Researcher 101 is a high vacuum system, so always wear gloves when working inside of the chamber. Never work on the sputtering guns without disconnecting the power supplies! These instructions are meant as a reminder of how to operate the system. The first few times that you use the system, do so with the TA present.

- a. Venting Chamber: Do not vent the chamber if the sputtering guns have been run in the last 30 minutes. The guns can be quite warm and need time to cool after operation.
  1. Close gate valve (0%)
  2. Turbo back switch should be open and chamber rough switch closed.
  3. Turn off ion gauge.

4. Open chamber vent switch. Sound of air entering the chamber should be heard. Venting the chamber takes approx 2 minutes to come to atmosphere.
  5. Open chamber door to retrieve sample.
  6. To work on the sputtering gun, the top of the chamber can be lifted:
    - a. Remove the bolts holding the chamber top.
    - b. Turn on power to hoist.
    - c. Lift chamber top using the hoist control.
- b. Changing targets: The targets have a limited lifetime so the targets need to be checked periodically to see if they need to be replaced. A majority of the sputtering occurs in a ring. It is possible to sputter through the target and start sputtering into the gun. To change target:
1. Disconnect power supplies from sputtering guns at power feed-through into the chamber.
  2. Lift up the top of the chamber.
  3. Move the shutter so you can access the gun. (shutters are controlled at front panel)
  4. Remove the outer grounded shield. This is held by friction to the side of the sputtering gun and can sometimes be very difficult to remove. First try to pull the shield off by hand. Pull up on the shield. Do not twist the shield, which can damage the feed-through. If the shield does not come off, then use a fine tipped standard screwdriver to gently pry between the target and shield. This should loosen the shield enough so that will come off easily.
  5. Unscrew the bracket holding the target
  6. Replace target and put the bracket back in place.
  7. Put the shield back in place. Only push down on the shield enough so it is held in place. If the shield is pushed down too hard, the top of the shield will get too close to the target and short the gun or cause arcing when the sputtering plasma is ignited. Before closing the chamber, the gun should be checked for shorts. Disconnect the power cords from the sputtering gun under the chamber. The resistance between the target and ground should be in the MegaOhms range.
- c. Pump down system from atmosphere:
1. Put mechanical pump exhaust tube out door so that fumes from the pump don't fill the room.

2. Lower chamber door.
  3. Close door on side of chamber.
  4. Make sure chamber vent switch is closed.
  5. Close turbo back switch: this isolates the turbo pump. Check pressure behind the turbo pump (gauge A) which should be below  $10^{-1}$  Torr. If the pressure rises above that pressure, then check that the gate valve is completely closed, then open turbo back switch to pump out the back of the turbo pump.
  6. Open chamber rough switch (only if turbo back is closed): starts rough pumping on main chamber. To check if there is a vacuum in the chamber, try to open the door on the side of the chamber. The door should be sealed by the vacuum. If the door opens easily then there is a major leak from either the door, the chamber vent, or the top plate. Close chamber rough switch and check for leaks (tighten bolts on top chamber and reseal door) and then try again.
  7. Wait until the pressure in the chamber is approximately  $5 \times 10^{-2}$  Torr as determined by gauge B. This should take about 4 minutes.
  8. Close chamber rough switch.
  9. Open turbo back switch.
  10. Open gate valve manually to about 20%.  
Never open gate valve if chamber is above 1 Torr. Check the pressure behind the turbo pump (gauge A). It should be less than 1 Torr - if it is too high then close the gate valve slightly.
  11. Wait until the pressure in the chamber is in the  $10^{-3}$  Torr range, then open the gate valve to 50%. The pressure in the chamber should drop quickly below the range of gauge B ( $10^{-4}$  Torr)
  12. When the pressure  $< 10^{-4}$  Torr open gate valve to 100 %.
  13. Turn on Ion Gauge. The pressure of the chamber should drop quickly into the  $10^{-5}$  Torr range and takes approx. 3 hours to reach the  $10^{-7}$  Torr range.
- d. Operating the Sputtering Guns: Operation of the sputtering guns should be done only when the chamber has reached the  $10^{-6}$  Torr range and preferably in the  $10^{-7}$  Torr range. For details of the sputtering process see the attached chapters on sputtering.
1. Record base pressure
  2. Turn off ion gauge
  3. Close gate valve to approx. 25%. This slows the pumping speed of the turbo pump to allow the introduction of the sputtering gas.

4. Record new pressure
5. Check that the Ar tank is open and pressure is approximately 10 psi.
6. Open the Ar leak valve slowly until the pressure in the chamber reaches 4 mTorr (approximately 30 on the leak valve)

NOTE: Pressure behind the turbo pump will increase but should not be above 1 Torr. If it is too high then close the gate valve some more to slow the pumping speed.

7. Check that the power supply controls are set at standby
8. Turn power supply on.
9. Before turning on the sputtering gun, check that the sample is not located over one of the sputtering guns
10. To start the sputtering gun, switch the power supply control to WATTS.  
The output should read the power being applied to the gun. (typically 80-120 Watts). Look inside the chamber to see the plasma. If the power reads 0 and no plasma is observed then check the voltage and current of the power supply. If the voltage is small and the current reads  $>700$  mA then there is a short in gun (which is quite common) and the chamber will need to be vented and the gun repaired. If the voltage is high  $> 1000$  kV and the current 0, then the Ar pressure is too low to ignite a plasma or the power supply is not connected to the gun.
11. The guns should be presputtered for a minimum of 5 minutes before making a film. The shutters should be closed when presputtering. These are controlled by the shutter switches on the front panel.
12. For a given power also record the current and voltage.
13. Adjust the leak valve to maintain the Ar pressure at 3 mTorr. This combination of pressure with a power of 80 Watts gives a sputtering rate of about 120 Angstroms per minute, but this changes for different targets. You should adjust the gun power until the plasma becomes the most stable, and then make a plot of the film thickness versus sputtering time for the material that you are sputtering.
14. To deposit a film, close the shutter and move the substrate over the sputtering gun. Open the shutter for a fixed time and then close shutter and move substrate away from the sputtering gun.
15. Allow sputtering gun to cool for approx 1 hour before venting system to remove the samples.



4. Problems with the sputtering gun: Most the problems associated with the sputtering unit are found in the guns.
  - a. Sparks during sputtering: If the ground shield is too close to the target, then sparks will occur when there is arc between the target and the shield.
  - b. Shorts: Shorts usually occur when flakes of metal fall between the target and ground. The characteristic of a short is that the current to the target is high and the voltage is very low. Remove the ground shield and clean the sputtering gun of all metal flakes. Replace ground shield and check for shorts with the multi-meter (measure resistance between the target and ground).

## C. Instruction for Rigaku X-ray Diffractometer

X-ray Diffractometer is not to be used without TA supervision!!

1. Turn on cooling water by opening both valves at the same time. Valves are located behind diffractometer on right hand side.
2. Turn on chiller power by switching circuit breaker on. Chiller is behind diffractometer on left hand side.
3. Log the beginning usage of the X-ray time (hour meter located behind door of diffractometer) into the notebook. We can also load the sample on platform before turn on X-ray power.
4. Turn X-ray machine power on.
5. Start computer program by typing "menu" at the prompt. C:\menu
6. Choose "Manual operation program" to ramp the X-ray power and check the sample position.
7. Choose "Datum and HV/PHA setting program"  
choose "DXG ramp" for digital X-ray generation  
select program number "3"  
X-ray power: ON  
KV: 30 (on) [20 (off)]  
MA: 30 (on) [2 (off)]  
'Ctrl+Z' to execute the command
8. Choose "Display system status" 'Enter'  
choose "View DXG status" 'Enter'  
to check whether DXG finish ramping
9. After X-ray finish ramping, use "manual scan program" to check whether the X-ray beam hitting the sample.
10. "Qualitative Program"  
"Qualitative data collection program"  
"scan condition" to set the parameters  
select "18" PHY133 high angle scan to set the scanning window.  
[example] Step scan, 2theta/theta reflective, from 15° to 95°, 0.02°/step, and 1.4 sec/dwell time each step.  
Regular powder diffraction slit size "0.5, 0.5"  
'Ctrl+'Z' to accept all the settings.
11. Choose "Measurement" to start the data collection.  
No printer  
Save data on C drive

12. Choose “Real time data collection display” to show the plot of Counts vs  $2\theta$ .

♠ Do not open X-ray machine’s door when the shutter is open and voltage is high. Push “Door open” button to swap sample.

## **V. GUIDELINES FOR WRITING A PAPER**

The presentation of experimental results is one of the most important aspects of research. In most cases you have to present a large amount of work in a few concise pages. Moreover, since your potential audience must be able to understand your paper, it must be as clear and concise as possible. It is very important to give proper credit to related previous work, since your scientific audience will consist of your peers who may have done some work in the field. A well-written paper will have the following ingredients:

1. Title: This is the "hook" which will call attention to your work. The title should be concise, although descriptive of the research. Hopefully, it will not involve much jargon.
2. Abstract: This will be a short section of perhaps 5-15 lines which describes in a very concise manner the main results and the uniqueness of the work.
3. Introduction: A short section, perhaps one typed page, describes the nature of the problem, some background and prior work, theoretical background and a description of what was done in this work.
4. Experimental: This is a description of experimental facilities used, uniqueness of the experimental approach, what samples were prepared, range of experimental parameters explored, etc. There is no need to describe trivial experimental aspects. This section will be, at most, one typed page long.
5. Results: This will be the main body of the paper. It will include a description of the data, including graphs, tables, or any other results. It should include ideally 3 graphs that summarize the data, and perhaps one concise table. Trends in the data and tables will be discussed in this section. This section should be, at most, 5 typed pages.
6. Conclusions/Discussion: In this section, a comparison with previous work will be performed. The uniqueness of the results will be highlighted and a comparison with theoretical aspects will be performed. Again, there is no need to dwell on trivial aspects or describe obvious facts. This section will have at most 3 typed pages.

7. Summary: Here again the problem and the results are restated in a concise fashion. This should be done at most in 1/2 typed page.
8. Acknowledgements: Any acknowledgements to discussions with others, ideas supplied by others, or financial help will be included in this section of 3-5 lines .
9. References: A complete set of references in the American Institute of Physics format will be collected. These references should be referred to in the body of the paper in sequential fashion.

As an example of well written papers and to get an idea of what type of papers you are supposed to write, you should look through Physical Review Letters or look at the following papers included with this manual.