

Making Tunnel Junctions Possible: Design and Implementation of an Angular Deposition Device and Oxidation Process

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Abstract

Modifications to an automated thermal evaporator were made: A device for angular evaporation was fabricated and a protocol for in vacuum oxidation was developed. The new features allow for versatile fabrication of metal-tunnel junction-metal thin film samples. A discussion of the usefulness of tunnel junctions is provided.

I. Introduction

Recent advances in lithographic techniques has broadened the possible experiments in "mesoscopic" physics. The use of optical lithography with high vacuum coating techniques allows for fabrication of highly pure, thin film samples with a minimum feature size of 0.5 micrometers [6]. Fabrication of feature sizes smaller than this resolution limit requires the use of electron beam lithography. The quiver of condensed state experimental activity at Michigan State University makes use of both techniques to fabricate thin film samples with progressively smaller feature size. (Experiments in electron transport and magnetoresistance are a few among the bunch.) These experiments require the growth of metal on metal in a controlled environment. However, the need for various combinations of metal/insulator/metal samples has prompted the demand for a fabrication process not yet installed at the Center for Material Sciences. (Here the term "insulator" refers to an oxidized metal layer that separates two or more metals.) Thus my efforts during this internship were devoted to the design of a angular deposition device and oxidation process that allowed the in vacuum versatility of growing any combination of metal and insulator. The cover plate device provides physical vapor deposition at controlled angles and sample spin rates. An oxidation process allows the growth of insulating barriers between metals. For project motivation, I will provide an qualitative overview of three items of experimental interest made possible by implementing the angular deposition device and oxidation process. A brief overview of the existing vacuum deposition system is given followed by a detailed explanation of the two new features.

II. New Experimental Capabilities

All three new capabilities rely on the electron tunneling phenomena. In each case a thin insulating barrier, which from now on will be referred to as the tunnel junction, joins two conductors. When joined by a sufficiently thin tunnel junction, an electron has a finite probability to tunnel from one from an energy state in one metal to a state of comparable energy in the other metal provided that the destination state is unoccupied. Therefore a *tunneling current* can be measured for the conductor/tunnel junction/conductor system. The availability of destination states can be modified by introducing a potential difference across the system. If we consider

the conductors in each case to be in equilibrium with each other, i.e electron distribution is governed by the fermi function, then we can illustrate the discussion with Figure 1, a one dimensional representation of energy states in a normal metal.

Figure 1: Energy States diagram for NIN system. States below the Fermi level, E_F are occupied. In (a), no voltage applied \Rightarrow no tunneling. In (b), an applied voltage \Rightarrow tunneling

Figure 1a depicts an unbiased normal metal-tunnel-junction-normal metal (NIN) configuration in equilibrium. No tunnel current will flow since there are no available energy states in N_2 . However, biasing N_2 positive with respect to N_1 effectively lowers N_2 's energy states exposes an available destination states for electrons in N_1 . As shown in Figure 1b, a tunneling event can occur since free electrons in N_1 "see" an available energy state in N_2 . We can expect an ohmic behavior since further biasing will create more destination sights and therefore increase the tunneling probability.

A similar argument is applied to the NIS system where one conductor, S, is a superconductor. Figure (2 a,b,c) shows the progression from zero biasing of the superconductor to positive biasing which results in tunneling from normal metal to superconductor.

Figure 2: Energy States diagram for NIS system. When the superconductor is positively biased,(b), with a voltage greater than $\frac{\Delta}{e}$, tunneling will occur. The opposite tunneling route occurs for the negatively biased situation, (c).

Part (c) illustrates the superconductor to normal metal tunneling resulting from a negative bias. Because of the energy gap, labeled Δ in Figure (2), no tunnel current is measured for voltages ranging from $-\frac{\Delta}{e} < V < \frac{\Delta}{e}$ where e represents the fundamental unit of charge. A plot of the IV characteristics for the NIN and NIS systems is shown in Figure (3). It is quite obvious from Figure 3 that tunneling effects provide a direct method for measuring the superconductor's energy gap.

Figure 3: IV characteristics for the NIN system, I_{NIN} , and NIS sytem, I_{NIS}

The second new capability, the ability to probe a superconductor's density

of states, manifests itself through the equations of tunneling current. In the NIS system, the net tunneling current can be shown to be [7]

$$I_{NS} = \frac{G_{NN}}{e} \int_{-\infty}^{\infty} \frac{N_{1S}(0)}{N_1(0)} [f(E) - f(E + eV)] dE \quad (1)$$

where G_{NN} is the conductance for the NIN system, $G_{NN} \equiv \frac{I_{NN}}{V}$. $f(E)$ and $f(E + eV)$ correspond to the electron distributions in each metal. $N_{1S}(E)$ represents the superconductor's density of states below the superconducting critical temperature. $N_1(0)$ refers to the superconductor's density of states in above the critical temperature. Hence the ratio $\frac{N_{1S}(0)}{N_1(0)}$ represents a normalized density of states. From the current equation we obtain the differential conductance, G_{NS}

$$G_{NS} \equiv \frac{dI_{NS}}{dV} = G_{NN} \int_{-\infty}^{\infty} \frac{N_{1S}(0)}{N_1(0)} \left[-\frac{\partial f(E + eV)}{\partial eV} \right] \quad (2)$$

In the limit $T \rightarrow 0$, expression reduces to [7]

$$[G_{NS}]_{T=0} = \left[\frac{dI_{NS}}{dV} \right]_{T=0} = G_{NN} \frac{N_{1S}(e|V|)}{N_1(0)} \quad (3)$$

In the experimental setting, we can measure $\frac{dI_{NS}}{dV}$. It is also quite evident from Figure (3) that when $V \gg \frac{\Delta}{e}$ we obtain a measurement of G_{NN} . Lastly, the electron distribution is a fermi function with known temperature (the chamber temperature.) Thus, via curve fitting methods, we are able to deduce the normalized density of states from expressions 2 and 3. Currently, this method is the only available experimental procedure for probing the density of states [4].

III. System Overview

The system for which the cover plate and oxidation process was designed is an Edwards Auto 306 Vacuum Coater. The fully automated system can obtain an ultimate chamber pressure of 10^{-7} torr via a two stage pump down process. A mechanical rotary pump operates as the first stage by pumping the chamber from atmospheric pressure system to 10^{-2} torr. At this point

the system switches to a turbomolecular pump that drives the chamber pressure eventually to its ultimate value. Typical operating pressures for sample fabrication occur over the range of 10^{-5} to 10^{-6} torr. The exponentially decaying time dependence of depressurizing a chamber makes the 10^{-7} range a timely endeavor (over an hour of pump down time is required to achieve the 10^{-6} range.) To induce a timely pump down process, a liquid nitrogen trap lines the intake valve of the turbomolecular pump. When filled, the liquid nitrogen trap enhances the intake's gettering properties and hastens the pump down process.

For multi-metal depositions without breaking vacuum, the system contains a rotating source tray located at depth 15 inches directly below the sample holder. The tray is equipped with four material holders with specific design for optimal evaporation and emission of the source material. A variable power dial provides the necessary current supply for evaporating the source material. The source to sample path is regulated by an open/close shutter that is operated manually. This feature permits the user to stabilize the deposition rate before exposing the sample to the source material.

Other convenient features include a processing function that allows some versatility chamber pressures during operation. Thus it is possible to devise a multi-step process where the deposition parameters are varied at each step. The system contains an air admit valve that vents the chamber with a desired gas. In addition to the air vent valve, the system has a gas inlet line coupled to a needle valve control. This allows a controlled entrance for pressurized gases during operation. Finally, the turbomolecular pump contains a throttle valve for manual control of pumping speed.

IV. Cover Plate Design Considerations

1. Why *in vacuo*?

To design a functionally sound cover plate with the desired sample spin/tilt features, a basic understanding of vacuum logistics was necessary. The motivation for fabricating thin films in high vacuum is simple—a thin film sample with nanometer to micrometer feature sizes is easily contaminated by particles common in normal atmospheric conditions. As with any experiment, a contaminated sample does not render the desired measured properties as that of pure samples. By removing particles from a chamber via, vacuum pumping techniques, we radically reduce the possibility of contamination.

This point is easily demonstrated by calculating the particle's mean free path. According to kinetic theory, a particle's mean free path, λ , when it is at room temperature is

$$\lambda(mm) = \frac{6.6}{P} \tag{4}$$

where λ is measure in millimeters and $P = pressure$ measured in pascals [2]. For air at atmospheric pressure the molecular mean free path is $\approx 10^{-8}$ meters. However if we decrease the pressure to $\approx 10^{-6}$ torr the molecular mean free path increases to ≈ 6 meters! Hence the likelihood of a contaminating molecule interacting with a source molecule en route to the sample sight (over a 15 inch span) is statistically unfavorable. Furthermore, the contaminating particle is less likely to strike the sample area. The result is a highly pure thin film.

2. Design Overview

The overall cover plate design is quite simple. Both features use rotary feedthroughs to allow externally controlled motion in vacuum. Each rotary shaft is coupled to a worm/spur gear configuration which transfers motion from the vertical to horizontal plane. The feedthrough locations were chosen to provide a coaxial horizontal axis of rotation. For the tilt feature, a set screw couples the rotating plate to its horizontal shaft. The spin feature's horizontal shaft extends through the rotating plate to the cover plate's centerline. To obtain the desired sample rotation, a miter gear configuration, on center, transfers motion back to the vertical plane. The two features are not completely independent-an angular displacement in the rotating plate will result in an equal sample angular displacement. For manual operation, the tilt feature uses a vernier dial precise to $\pm 0.05^\circ$. A variable speed stepper motor drives the spin feature. The stepper motor will be discussed a latter section.

3. In Vacuum Components

Of primary concern is maintaining vacuum integrity. Subtle leaks due to component malfunctions can cause irreversible damage to the pumping systems [2]. Since the sample spin/tilt features two mechanical feedthroughs, the method for transferring rotary motion from the atmosphere to vacuum need to have low leakage ratings. To accommodate these needs, o-ring sealed shaft mechanical feedthroughs were used. The feedthroughs are rated for leakproof operation at speeds of 500 rpm in up to 10^{-7} torr.

Vacuum integrity can also be compromised due to the gas released from chamber surfaces. The effect known as *outgassing* is the primary cause for lengthy pumpdown times. Outgassing occurs as a result of four main processes: vaporization, thermal desorption, diffusion, and permeation. Holland reports that among the various metals considered for design of vacuums, stainless steel and aluminum have the lowest combined outgassing rates [1]. Indeed, the Edwards Vacuum coater system uses stainless steel and aluminum in the fabrication of their vacuum chambers. Thus all necessary mechanical components for the sample spin/tilt features were built from stainless steel and aluminum. To prevent further outgassing from foreign materials, no powders or liquids were introduced into the vacuum chamber. Hence the working gears are not lubricated. This should not constitute a major problem for device performance since the spin feature has a low maximum rotation speed of 12 rpm.

4. Tilt Specifications

To fully accommodate the experimenter's needs, the tilt design allows a $\pm 90^\circ$ maximum deposition angle for a 3" sample holder. This versatility summons one fundamental question-what deposition angle should be used in angular evaporation? This experience has taught me that the lithographic and deposition processes vary with the experimenter's preferences so the answer boils down to "who is the sample for?" But to get a flavor for the answer, we use the geometry of Figure 4 with some practical thin film parameters.

Figure 4: Deposition Angle Geometry

Here, the ballast layer plus the suspended mask layer, h , and desired metal lead separation, d , dictate the deposition angle, θ . The deposition angle, is given as

$$\theta = \arctan \frac{d}{2h} \quad (5)$$

Typically, the ballast and mask layers combine to form a layer of ≈ 700 nanometers [4]. Currently the research, performed at the Michigan State facility, fabricates thin film samples with lead separation to ranging from 200-1000 nanometers [4]. These values correspond to a minimum deposition angle of $\approx 8^\circ$ and a maximum deposition of $\approx 35^\circ$.

5. A Tilt Angle Correction

Some attention should be given to the off axis sample displacement which, when of comparable length to the sample-source distance, can distort the simple 1:1 ratio of tilt angle to deposition angle. Figure 5 represents the a more accurate geometric depiction where L represents the off axis tilt displacement.

Figure 5: Geometry of the angular deposition device

Tilting the sample through a given angle, ϕ , results in a source to sample distance, H . (The angle created at the source is neglected since it does not

appear in the calculation.) The distance from source to sample when $\phi = 0^\circ$ is defined as h . Thus for any value of ϕ , H can be expressed as

$$H = \sqrt{[h + (L(1 - \cos \phi))]^2 + [L \sin \phi]^2} \quad (6)$$

Using the law of sines and expression 6, we achieve an implicit correction for the tilt angle ϕ ,

$$\frac{\sqrt{[h + (L(1 - \cos \phi))]^2 + [L \sin \phi]^2}}{\sin \phi} = \frac{L + h}{\sin \theta} \quad (7)$$

Notice that for $h \gg L$, $\sin \phi = \sin \theta$ which is the 1:1 angle approximation. Those feeling pedantic may proceed with further manipulations but I assert that the end result is not for the faint of heart. In trying to reduce the effective correction, the off tilt displacement was minimized while the source to sample distance was maximized. The physical limitations allowed L to be $\frac{1}{10}$ th h so the approximation can be used until fabrication problems require the necessary correction.

5. Use of Stepper Motor

The need for a constant sample spin rate prompted the spin feature mechanization. An eight lead stepper motor with a motor drive unit, purchased from *Applied Motion Products*, was already in our possession so it was put to use. Among its nice features is a step resolution of 1.8° , a 44.4 oz-in holding torque and parallel/series coil configuration. The latter provides some torque versatility where the series configuration provides high torque at low rpm and the parallel configuration provides just the opposite. Despite these capabilities, this system does not warrant the step angle option and the anti-backlash worm/spur gear assumes the holding torque role. However, should future experiments require the step angle feature, the modification is easy.

The drive unit, an *Applied Motion Products* 2035 0, contains a full and half step phase sequence, two optoisolated circuits, an idle motor current reduction option, variational current DIP switches, an internal oscillator, and internal acceleration/deceleration trim pots. A 24VDC source powers the drive through the primary circuit. Of the secondary circuit features, I used external speed, the run/stop, and the direction controls. It is standard practice to isolate circuits that draw considerably different current loads-it effectively reduces the electromagnetic interference resulting from a shared power source. However, use of the run/stop and direction switches only

requires driving the secondary circuit low with respect to a power source. And since no current drawing devices were to be used on the secondary circuit, I choose not use separate power supply-a voltage drop was taken from the $24VDC$ power supply. Sinking the secondary circuit enables the external speed pot upon switching to "run". Figure (6), pg 11, illustrates the working circuit diagram.

Figure 6: Stepper motor drive circuit

When assembled, I found the motor to run best in half step mode (the rotor transition is small so it can more easily find its next position.) I limited the motor to $0.875A$ (its rated for $1.2A$) but the DIP switches allow a current maximum of $1.5A$. The power supply, motor drive, and switches are housed in an aluminum desktop console and attached to the Vacuum Coater unit. For ease of cover plate transfer, the motor and drive are not hard wired. The circuit is connected via an inline connector. Twisted/shielded wire is used for all in console connections.

V. Cover Plate Limitations

When completed, the spin feature was found to have some limitations. Essentially all its limitations directly result from the motor driven components. The first issue is maximum shaft speed. Although the motor renders an adequate max shaft speed of 12 rpm, the motor tends to bind at speeds just beyond this. Near its limit speed, the rotor loses its step phase with the stator which results in loss of electromagnetic driving force. Also at speeds near maximum, the motor binds when switching direction-another manifestation of phase loss within the motor unit. I mentioned earlier that no lubricant was applied to the worm/spur gearing to suggest some long-run limitations. Since the gears grind metal on metal, I predict that the gearing will need service or even replacement after lengthy usage. For a convenient time table, one might choose to replace the gearing when the systems begins binding at speeds successively lower than the 12 rpm motor limit. Lastly, in time, the teflon bearings on the motor driven side will need replacement after sufficient usage.

VI. Process Protocol

Use of an automated thermal evaporator has the advantages of programmability. I was able to make use of its pre-programmed functions to stabilize chamber pressure after inletting the mixed gas. Since oxygen is a reactive gas, a 90% argon 10% oxygen mixture was chosen for the chamber gas. The reduced oxygen content allows for overall safety at the expense of a timely oxidation process. As a reference pressure, I used Pierre's data that reports that appropriate sized tunnel junctions ($\approx 100 \times 100 \text{ nm}^2$) were achieved after 30 oxidation period in a 10^{-1} torr chamber [?]. Since these values fall just above the turbomolecular pump's minimum operating pressure, we can only use them as a rough time estimate for processing times at lower gas pressures. We can safely inlet gas to the pump's threshold of $\approx 10^{-2}$ torr but caution must be taken not to exceed this limit. Protocol for one oxidized layer is as follows (protocol is specific to the Edwards Auto 306 Coater):

- After depositing a metal seal the chamber using *Seal* function and let the chamber cool for 15 minutes
- Using the *Needle Valve* inlet the mixed gas until the target pressure is reached-not to exceed 10^{-2} torr. Close the needle valve
- Let the chamber sit to allow oxidation-expect at least 30 min
- Pump down the system using the *Cycle*.

VII. Conclusions

The thermal evaporator modifications have provided methods for such superconductor experiments as energy gap determination and probing the density of states. However, some further considerations need attention. In particular, due to time restrictions, I could not acquire data relating the tunnel junction thickness to allowed oxidation time for given chamber pressures. Since no thin film fabrication occurs under like conditions, a database of the varying parameters must be developed as a general guideline for fabrication protocol. I leave this task to future system users-perhaps another REU student project?

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