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The Effect of Varying Sb Layer Thickness on the Time-Temperature Characteristics of Co/Sb Superlattices

Kazi M. Reaz
Indiana University of Pennsylvania

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THE EFFECT OF VARYING SB LAYER THICKNESS ON THE TIME-TEMPERATURE CHARACTERISTICS OF CO/SB SUPERLATTICES

A Thesis

Submitted to the School of Graduate Studies and Research

in Partial Fulfillment of the

Requirements for the Degree

Master of Science

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Indiana University of Pennsylvania

December 2014
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I performed a systematic study on Co/Sb superlattices varying Antimony (Sb) thickness keeping Cobalt (Co) thickness constant at 1 nm. Here I studied the decay of resistivity of those samples. To make those samples I deposited 10 bilayers of Co and Sb at 500°C on <100> face of silicon substrate in an Edward model E306A e-beam evaporator. After making the sample we measured the decay of resistance using Vibrating Sample Magnetometer (VSM) with Magneto Resistance (MR) option.

We made samples of 8 different thickness of antimony, ranging from 0.5 nm to 5.0 nm. We measured the decay of electrical resistance of our samples as a function of time and temperature. In this thesis we measured the decay of resistance of all of the samples at various temperatures like 320K, 340K, 360K, 380K, and 400K. Then we plot decays of resistances on log time scale in order to inspect the variable time needed for a certain thickness of Sb to decay. We also made Arrhenius plot of our data and analyzed them.
ACKNOWLEDGEMENTS

At first I would like to thank my thesis advisor Dr. Gregory Kenning for his support, cooperation, guidance and criticism. He has always been encouraging, helpful, and caring.

I am very grateful to Dr. Muhammad Numan who is one of my thesis committee members. He has provided me with innumerable comments and guidance. In the course of the last two years I have learnt so many things from him.

I would like thank my other thesis committee members Dr. Majid Karimi and Dr. Vythilingam Wijekumar for their support and advice in every step of my thesis. I would also like to thank Jared Paul Phillips and Steven Paul Hensley for their help on writing my thesis. I am also grateful to our department secretary Tracey Emanuel for her assistance, sincerity and smiles.
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CHAPTER I
INTRODUCTION

Nanotechnology is an emerging technology that enables us to design and build new materials at molecular level. There are many advantages in making devices smaller, as in modern semiconductor electronics. Here the word “Nanotechnology” will be associated with human designed devices in which some essential element, thin film, produced in a controlled fasion, have size of 0.1 nm to thousandths of a nanometer. Thin films are usually created through a deposition process. By modifying four states of matter- solid, liquid, vapor, plasma- we achieve deposition. The deposition process can be executed through various kinds of vacuum processes like physical vapor deposition (PVD), chemical vapor deposition (CVD), and evaporation through vacuum sublimation. Nowadays all kinds of semiconductor devices are made with thin films.

In this thesis the materials that are used to make thin films are cobalt and antimony. We made our thin layer samples using e-beam evaporation technique. I got the motivation of doing this thesis from the thesis of a previous graduate student Christopher Heidt in 2009 and Kenning et al. 2011. We have made multilayer samples of cobalt and antimony keeping the cobalt layer thickness fixed at 1.0 nm. We varied the interceding layer of antimony. We made samples of 8 different thickness of antimony, ranging from 0.5 nm to 5.0 nm. We measured the decay of electrical resistance of our samples as a function of time and temperature. In this thesis we measured the decay of resistance of all of the samples at various temperatures like 320K, 340K, 360K, 380K, and 400K. Then we plot decays of resistances on log time scale in order to inspect the variable time needed for a certain thickness of Sb to decay. We also made Arrhenius plot of
our data and analyzed them. We also studied the normalized resistance variation as we changed the temperature systematically.
CHAPTER II
THEORETICAL BACKGROUND

2.1 Band Theory of Electron Transport of Solids

When isolated atoms are brought together they can form a solid. In a solid the atoms can arrange themselves in a lattice configuration. This arrangement is due to various interactions among the atoms. Individual isolated atoms can have identical electronic structures because there is no interaction of electron wave functions with surrounding atoms. When atoms are brought together in a solid, the spacing between them becomes smaller and the electronic wave functions begin to overlap. Consequently electrons can form extended energy states in the solid. Extended states from many atoms (each given up at least an electron) form bands [1][4][22]. There are two types of bands in the solid; the valence band and the conduction band. The band width depends not only on the number of atoms but also on the atomic spacing. The main parameter in the band structure is the Fermi energy or Fermi level. It directly comes from Fermi-Dirac (FD) statistics which describes a distribution of particles in certain systems containing many identical particles that obey the Pauli Exclusion Principle. [1][4][27] Fermi-Dirac distribution can be written as

\[
f(E_i) = \frac{1}{1 + \exp\left(\frac{E_i - E_F}{kT}\right)}
\]  

Figure- 2.1. Band theory of solids.
Here parameter $E_F$ is called Fermi energy. The FD distribution function, $f(E_i)$ can have maximum value of unity corresponding to the energy at which every state is occupied by a particle. The minimum value of $f(E_i)$ is zero. When energy levels create continuum of energy, we can easily drop the subscript $i$. So eq. 2.1 becomes

$$f(E) = \frac{1}{1 + \exp\left(\frac{E - E_F}{kT}\right)}$$

(2.2)

**Figure-2.2.** Probability distribution function of FD statistics.

The FD distribution function is plotted in the Fig-2.2 for various temperatures. At 0K the distribution is characterized by step function

$$f(E) = 1 \quad \text{for } E < E_F$$

$$f(E) = 0 \quad \text{for } E > E_F$$

The bands which are higher in energy than the Fermi level are called the conduction bands while bands with energies below the Fermi level are called the valence bands. In real life each band consists of a very large number of states. For a mole of atoms this number is the Avogadro’s number. Because of this large number of states, a band is essentially a continuum of energies. The gap between two energy bands is called the “Band Gap”. Every solid has its own energy band structure. Because of this variation of band structure in different materials we observe a wide range of electrical characteristics \[1\][2][22]. By observing the variation in band structure we divide solids into four categories. They are
1) Metals or Conductors

2) Semiconductors

3) Semimetals

4) Insulators

---

**Figure- 2.3.** Comparison of band structure in different solids.

### 2.2 Metals or Conductors

In metals or conductors there is no forbidden gap or band gap (Fig-2.3) between the valence band and the conduction band, they overlap each other. This means that a temperature dependent fraction of the valence electrons (those are in conduction band) are free to move through the materials. In metal, atoms are embedded in a “sea” of relatively free electrons. These free electrons can move freely under the influence of electric field. That is why they are very good conductors of electricity [1][2][4].

#### 2.2.1 Temperature dependence of resistivity of a Conductor

In conductors, the number of electrons does not change as temperature increases or decreases. When temperature increases, the ions that are locked in the lattice vibrate at different amplitudes. The higher the temperature, the larger the lattice vibration becomes. In the
conduction band, electrons can collide with phonon excitations in the lattice, and the probability of that collision increases with the temperature. That is why resistivity increases with

![Figure-2.4. Linear relation between temperature and resistance.](image)

temperature of a metal. We know that the resistance of metals vary linearly with temperature (far above from 0k and far below from melting point) [2][4][16]. The relation between resistance and temperature is

$$R = \alpha R_f T + R_f (1 - \alpha T_f)$$  \hspace{1cm} (2.3)

$R_f$ is the resistance at any reference temperature and $\alpha$ is the temperature coefficient of resistivity. In a metal $\alpha$ is always positive for a given reference temperature. Rearranging eq. 2.3 we get

$$\alpha = \frac{R - R_f}{R_f (T - T_f)} = \frac{1}{R_f} \frac{\Delta R}{\Delta T} = \frac{1}{R_f} \frac{dR}{dT}$$  \hspace{1cm} (2.4)

2.3 Semiconductors

Semiconductors are crystalline materials. The Electrical resistivity of semiconductors is generally higher than metals. There are two types of Semiconductor

1. Intrinsic Semiconductor
2. Extrinsic Semiconductor
2.3.1 Intrinsic Semiconductor

At 0K a semiconductor has an empty conduction band. So there is no free electron to move around at 0K. When temperature increases, for the intrinsic semiconductor like silicon and germanium, a finite number of electrons acquire enough energy through thermal fluctuations to reach into the conduction band. So we get some electrical conductivity at higher temperature. We can model the increase in conductivity with temperature by Fermi function. The Fermi function allows us to calculate the total number of electrons of the conduction band [1][5][25]. In the entire course of my thesis only intrinsic semiconductors are being considered.

2.3.1.1 Electrical Conductivity for Intrinsic Semiconductor

When thermally excited electrons contribute to the conduction then we call it intrinsic semiconductor. When we talk about the conductivity of a semiconductor it means the total conductivity of the valence and conduction band carriers, that means sum of holes and electrons. A Hole is the absence of an electron in an atom or atomic lattice where one could exist. A hole is opposite of an electron. When an electron is thermally excited it leaves a hole in its previous position in the valence band [1][4][28][15].

The conductivity can be expressed as a sum of the contribution of both types of carriers

$$\sigma = e \left( n_e \mu_e + n_h \mu_h \right)$$

(2.5)
Here $n_e, \mu_e$ are electron’s concentration and mobility and $n_h, \mu_h$ are hole’s concentration and mobility.

The mobility comes from the relation of drift velocity, $V_d$ of the charge carriers and applied electric field, $E$. It is known that drift velocity is proportional to electric field.

$$V_d \propto E \quad (2.6)$$

From the above relation can clarify,

$$\mu = \frac{V_d}{E} \quad (2.7)$$

Where $\mu$ is called the mobility. We know number of electrons and holes are equal at intrinsic region. So

$$n_e = n_h = n_i \quad (2.8)$$

So the equation 2.5 becomes,

$$\sigma = n_i e (\mu_e + \mu_h) \quad (2.9)$$

### 2.3.1.2 Temperature Dependence of Conductivity of Intrinsic Semiconductor

Electrical conductivity of a semiconductor is in between a conductor and insulator. Its characteristic properties differ from metals in that as the temperature increases the electrical resistivity decreases. We know that the Fermi level lies in between the minimum conduction band and the maximum valence band in the intrinsic semiconductors. At 0K when no thermal excitations are available the conduction band lies above the Fermi energy level and therefore the conduction band is unoccupied. At 0K, the conductivity is zero and thus the resistance is infinite. When the temperature is increased the conduction band starts occupying with electrons due to thermal fluctuations. As a result of this, the electrical resistivity of the semiconductor starts decreasing [1, 4, 5, 15].

We know electron density as a function of temperature is given by

$$n_i = NT^{3/2} \exp \left(-\frac{E_g}{2kT}\right) \quad (2.10)$$
Where N is a constant.

If we replace $n_i$ in eq. 2.9 the electric conductivity turns out

$$\sigma = Ne T^{3/2} (\mu_e + \mu_h) \exp(-\frac{E_g}{2kT})$$

(2.11)

From the eq. 2.11 it is clear that the electric conductivity is dependent on temperature.

**2.3.1.3 Temperature dependence of mobility of Intrinsic Semiconductor**

From eq. 2.7 we see that the mobility means average of the drift velocity of a charge carrier under the influence of electric field. The drift velocity also depends on temperature. We know a charge carrier has a lot of collisions in the intrinsic semi-conduction region from the scattering of lattice vibration or phonons (Fig-2.6). These scatterings are the result of how strongly the ions vibrate and the scattering amplitude depends on temperature [5, 15].

![Figure-2.6. Cross Section of Effective Scattering [15].](image)

The mean free time ($\tau$) of these scattering events is

$$\tau = \frac{1}{SN_{sc}V_{th}}$$

(2.12)

Where $N_{sc}$ is no. of scatterers per unit volume and $V_{th}$ is the electron’s thermal velocity or mean speed.

We know relation between scattering amplitude $a$ and temperature $T$ is

$$a^2 \propto T$$

(2.13)
From the equipartition of energy theorem the mean kinetic energy of an electron is \((3/2)KT\).

So

\[
\frac{1}{2} m_e V_{th}^2 = (3/2)KT
\]  
(2.14)

This implies that

\[
V_{th} \propto T^{1/2}
\]  
(2.15)

Putting the value of \(V_{th}\) in the eq. 2.12 the mean free time becomes,

\[
\tau \propto T^{-3/2}
\]  
(2.16)

We know from the Drude model the drift velocity is

\[
\mu = \frac{e \tau}{m_e}
\]  
(2.17)

So from the equations 2.16 and 2.17 we get

\[
\mu \propto T^{-3/2}
\]  
(2.18)

### 2.3.1.4 Temperature dependence of Resistivity for Intrinsic Semiconductor

We know resistivity is the inverse of conductivity.

\[
\rho = \frac{1}{N e T^{3/2} (\mu_e + \mu_h)} \exp\left(\frac{E_g}{2KT}\right)
\]  
(2.19)

Where \(T^{3/2} (\mu_e + \mu_h)\) is a constant. Let \(A= T^{3/2} (\mu_e + \mu_h)\)

Eq. 2.19 turns out

\[
\rho = A \exp\left(\frac{E_g}{2KT}\right)
\]  
(2.20)

Taking logarithm both sides we get

\[
\ln \rho = \ln A + \frac{E_g}{2KT}
\]  
(2.21)

Or

\[
\ln \rho = C + \frac{E_g}{2K} \frac{1}{10^3} \frac{10^3}{T}
\]  
(2.22)

If we plot \(\log_{10} \rho\) vs \(\frac{10^3}{T}\) we get straight line. (Fig-2.7)
Figure 2.7. Linear Relation between Temperature and Resistivity.

Using eq. 2.20 we can easily derive the expression for the resistance of the semiconductor which is

\[ R = R_0 e^{\frac{\epsilon}{2kT}} \]  

(2.23)

Here \( T \) is the absolute temperature in Kelvin; \( R_0 \) is a constant and \( \epsilon \) is the energy gap. The temperature coefficient of resistance is given by

\[ \alpha = -\frac{\epsilon}{2kT^2} \]  

(2.24)

Here \( \alpha \) is negative because its resistance decreases as temperature increases.

2.3.2 Extrinsic Semiconductor

When we dope a pure (intrinsic) semiconductor with some doping agent (impurities) to change its electrical properties is called extrinsic semiconductor. Theses impurities are added intentionally after purifying the crystal of the intrinsic semiconductor. These impurities are known as donor or acceptor depending on their effect on the intrinsic semiconductor. When dopant atoms are added in the intrinsic semiconductor, the carrier (both holes and electrons) concentrations change. There are two types of extrinsic semiconductor; n-type semiconductor
and p-type semiconductor. When group V elements are used as impurity in the doping process to create extra electrons in the semiconductor is called n-type semiconductors (Fig-2.8). In the doping process when group III elements are used to create extra holes in the semiconductor is called p-type semiconductor (Fig-2.9). Almost all the semiconductor devices employ extrinsic semiconductor because of their electrical properties [1][25][27].

2.4 Insulators

As we know in solids, there is a forbidden gap between the valence band and the conduction band which we call band gap. In insulators the band gap is usually greater than 4 eV, which means a temperature of more than 4000K is needed for electrons to overcome the band gap. This temperature is far above the melting temperature of all insulators, therefore conductivity is not observed in insulators [1][2][4].

2.5 Semimetals

Semimetals are identified by comparing their band structures with metals and semiconductors. Most of the properties of semimetals differ from metals and semimetals due to their rhombohedral crystallographic structure (Fig-2.12). This rhombohedral crystallographic structure can be explained as a slightly distorted cube. There is a very small overlap between the
conduction band and valence band in the semimetals (Fig-2.10 and Fig-2.11). Because of this small overlap distortion occurs in their crystal structure [3][4][24].

Because of overlapping between valence band and conduction band, there is no band gap and at the Fermi level there is very little density of states in the semimetals. Therefore conductivity of semimetal is higher than semiconductors but lower than conductors. Arsenic, antimony, α-tin (gray tin) and graphite (an allotrope of carbon) are well known as semimetals [3][4][30].

2.5.1 Temperature dependence of resistivity of semimetals

Semimetals have similarities with both metals and semiconductors. When the charge carrier is concerned semimetals shows some similarity with semiconductors. As we know a
metal has one type of charge carrier which is electron and semiconductor has two types of charge carriers – holes and electrons. Semimetals also have those two types of charge carriers like semiconductors [2][30].

The temperature dependence of resistivity of the semimetallic state is similar to the metallic state in that the resistivity increases with temperature. This aspect of metals arises from temperature-independent carrier density. Some semimetals like arsenic and antimony have shown the same characteristics below room temperature. Bismuth shows this temperature-independent carrier density at very low temperature. But when temperature increases the carrier density of bismuth also increases (like semiconductors). At high temperature it shows a semimetal-semiconductor transition. Like metals the conductivity of semimetals at absolute zero temperature is always non-zero because of overlapping between conduction band and valence band, whereas semiconductors have zero conductivity at zero temperature. Because insulators have a wider band gap, there is zero conductivity even at high temperature [5][24][30].

2.3 Arrhenius Law

The Arrhenius law is encountered widely in situations where temperature is the main driving force. Svante Arrhenius proposed this formula in 1889 to describe chemical reaction rates which are dependent upon temperature. Usually the Arrhenius law is used to describe the transition of a system under the influence of temperature from one state to another, separated by an energy barrier. When the kinetic energy becomes sufficiently high, due to the increase in temperature, the system will overcome the energy barrier between states [5][31]. The general expression of Arrhenius law is

\[ k = A e^{\frac{E_a}{k_b T}} \]  

(2.25)
When the state transition is time dependent we use the Neel-Arrhenius law. 1949 by Louis Néel use Arrhenius law to explain time-dependent magnetic phenomena. Under the influence of temperature, the magnetization of small ferromagnetic nanoparticles can flip the direction randomly and average appears to be zero. This is known as the superparamagnetic state, which appears in single magnetic domain. Ferromagnetic nanoparticles with a diameter between 3-50 nm show this behavior. The range of diameter depends on materials. The magnetic moment of ferromagnetic nanoparticles has only two orientations which are anti-parallel to each other. This form of magnetism occurs because of their magnetic anisotropy [10][32][33]. The mean time between two flips is called the Neel relaxation time (\(\tau_N\)), which can be calculated by Neel-Arrhenius equation

\[
\tau_N = \tau_0 e^{\frac{E_a}{k_BT}}
\]  

(2.26)

Here \(E_a\) = activation Energy, \(T\) = temperature in kelvin and \(\tau_0\) = attempt period.

Neel relaxation time varies from nanoseconds to years.

If we take natural log on both sides of the eq-2.26

\[
\ln(\tau_N) = \ln(\tau_0) + \frac{E_a}{k_BT}
\]

(2.27)

In our calculations we will be using Neel Arrhenius law in the following form

\[
\ln(\tau) = \ln(\tau_0) + \frac{E_a}{RT}
\]

(2.28)

Here \(R\) is the molar gas constant. Eq-2.28 is similar to \(y = C - Bx\) which is an equation of a straight line, where \(\frac{E_a}{R}\) is the slope of the line. So we can say eq-2.28 gives us a relation of a logarithmic time value which is linear with reciprocal temperature, 1/T.
3.1 Thin Films

Multilayer films are composed of layers of different materials of different thicknesses deposited sequentially on a substrate. Materials that are used to make thin films can be metals, semiconductors, semimetals or insulators. Although the properties of a bulk material may be well known, the same material in a thin film state can possess completely different properties. A primary reason for this is that thin film properties can be strongly influenced by surface properties. Thin films have a higher surface to volume ratio than a bulk material. The preparation of thin films and their overall structure also play an important role in determining their properties. Thin films are very important elements of modern devices [18][19][20][34].

3.1.1 Thin Film Growth

Condensation, nucleation and growth are the main events in the thin film growth process. In this process, materials are condensed from a vapor phase to a solid phase on the surface of a substrate. This condensation on a substrate can lead to various structures of a material from the vapor phase. Usually they range from two and three dimensional to a continuous layer. Nucleation is the event when absorbed atoms diffuse on the surface of the substrate and form aggregates. The growth occurs when the nucleus size exceeds a certain critical size. Growth of aggregates leads to the change in free energy. One is surface free energy and the other is free energy between the solid phase and the vapor phase. The surface free energy increases as the surface area increases. The free energy due to the phase change is directly proportional to the aggregate volume. These two free energies can characterize the whole system [3][7][18][21].
3.1.2 Thin Film Growth Mode

Two most important growth processes observed in the growth of thin film materials are the

1) Frank-van der Merwe mode

2) Volmer-Weber Mode

3.1.2.1 Frank-van der Merwe mode

Another name for Frank-van der Merwe growth mode is layer-by-layer growth. This is also known as two dimensional growth mode. Frank-van der Merwe growth mode takes place when thin film atoms are more strongly coupled with substrate atoms than with each other. Usually it happens when the combined total surface energy of the deposited layer plus the interface energy between substrate and deposited is lower than the surface free energy of the

![Layer-by-Layer Growth Mode](image)

**Fig-3.1.** Frank-van der Merwe growth mode [21]

substrate layer. In this case without completion of one layer, the growth of next layer never starts (Fig-3.1). That is why this growth is completely two-dimensional [3][7][12].

3.1.2.2 Island or Volmer-weber growth

Island or Volmer-Weber growth is also known as a three dimensional growths (Fig-3.2). When metals are deposited on insulators, this method is common. Because in that case the bonds

![Island or Volmer-Weber Growth Mode](image)

**Figure-3.2.** Island or Volmer-Weber growth mode [21].
between atoms of deposited materials are stronger than the bonds between film atoms and the substrate. Sometimes it is not possible to cover the whole surface completely if a large deposition has not been made [3][7][21].

3.1.3 Sample Deposition

Our samples consist of 10 Co/Sb bilayers on silicon substrates. 99.9% pure Cobalt and 99.999% pure Antimony were used to make those samples. The deposition was made using an Edwards model E306A (Fig-3.3) e-beam evaporator in a vacuum of $\leq 10^{-6}$ Torr. Evaporation in high vacuum is necessary for the film materials to remain uncontaminated by residual gases in the deposition chamber. All of our samples were deposited on Czochralski grown <100> oriented silicon wafers. The pressure should be less than $10^{-6}$ Torr to ensure that the substrate is uncontaminated after cleaning for a reasonable amount of time. In our case we cleaned these silicon substrates with alconox, water and isopropyl (95%) alcohol with an ultrasonic cleaner. It is also essential that the interface between the substrate and the film have specific properties that

Figure-3.3. Edwards model E306A e-beam evaporator.
we will measure, which could be contaminated by outside influence. In this case, the surface of the substrate should remain uncontaminated once it is cleaned until we start deposition [3, 10]. When the pressure reached $\leq 10^{-6}$ Torr we started depositing materials on the substrate. The deposition rate was monitored with a crystal monitor during the entire course of the deposition. The deposition rate was approximately 0.1-0.2 nm/min. The Edwards system has a single e-beam gun but rotating target carousel allows sequential deposition of the films. The multilayer samples were produced by evaporating antimony (Sb) target to make the Sb layer, then rotating Co to the gun to produce Co layers. We waited approximately 5 minutes after the Co was deposited and 2 minutes after Sb was deposited to maximize time for possible Volmer-Weber growth mechanism to take effect [3][10].

Kenning et al. [10] have reported Co produces nanoparticles in the solid state within a semi-metallic environment. At 300K the bulk resistivity of Co is 62.7 n$\Omega$-m and its melting temperature is 1768.15 K. In contrast, Sb is a semimetal and has a bulk resistivity of 417 n$\Omega$-m; almost seven times higher than that of Co. The melting temperature of Sb is 903.78 K. In all of our samples we deposited the layers at a substrate temperature of 48-52°C. From Kenning et al. [10], we have seen that 50°C is the optimum deposition temperature. In order to avoid confusion, we monitor the deposition temperature in °C and we measure annealing temperature in K. By the process outlined above, we made 10 bilayers samples. In this thesis, all of our samples were 10 bilayers of Co/Sb. It takes almost two and half hours to make one set of samples. In our experiment we made in total 8 sets of samples [3][10].

In order to minimize any aging of the samples by the outside environment, immediately after deposition, the samples were placed in sample holders and submerged in a liquid nitrogen Dewar where the temperature was maintained at 77K. When needed, we removed a particular
sample and warmed it to room temperature in a desiccator. Then we placed it into an apparatus for resistance measurements. All of our resistance measurements were made using a Lakeshore model 7307 Vibrating Sample Magnetometer (VSM) with Magneto Resistance (MR) option. Lakeshore MR uses 4-probe method for resistance measurement. For our experiment we modified the 4-probe to 2-probe by disabling its two inner probes so that current and voltage were both measured by the outer probes. We will discuss 4-probe and 2-probe method in the section 3.2.1 and 3.3.1. The probes are made of nickel-coated tantalum and they pierce through the multilayers of the sample. In all of our resistance measurements we inserted the sample and MR probe into a preheated oven set at the measuring temperature. All of our resistances versus time measurements were made at 0.01T magnetic field [3][10].

### 3.2 Sheet Resistance

Thin films are considered two dimensional entities. When we talk about sheet resistance-which is applicable to the two dimensional systems- we mean that the current is travelling along plane of the sheet. No current passes perpendicular to it [3][18]. Electrical Resistance of a plane sheet (fig-3.4) of length L, thickness t, and width w is given by

$$ R = \frac{\rho L}{wt} \quad (3.1) $$

![Figure-3.4. Different types of resistance elements.](image)

Where R is in ohms, \( \rho \) is ohms-m, and L, t, and W are in m. When plane sheet is a square L=W, we get a quantity \( R_s \) which is called sheet resistance.
\[ R_s = \frac{p}{t} \]  

(3.2)

The unit of \( R_s \) is ohms per square (\( \Omega/\square \)). We frequently use this, \( R_s \) to measure the resistance of circuits and chips. Here \( R_s \) is independent of the size of the square. The total area of conductor is divided into a number of squares. That number is the multiplied by \( R_s \) and we get the total resistance.

The two most common techniques for measuring the sheet resistance of thin films are the four probe method and the two probe method. I will explain these two techniques in further detail within the following two sections.

3.2.1 Four Probe Method

The Four probe resistance technique is the most popular and widely used apparatus for measuring resistivity of semiconductors. The fig-3.5 can demonstrate the most common four probe arrangement. Experimentally the concentration of carriers is determined by measuring the sheet resistance of the diffused layer at a definite temperature using four probe technique.

![Figure-3.5. Four probe arrangement][15]

3.2.1.1 APPARATUS

The apparatus consists of an oven (0K-450K), the four probe arrangement, fixed current generator, power supply and digital meter to measure voltage and current. In our experiment we
use Lakeshore model 7307 Vibrating Sample Magnetometer (VSM) with Magneto Resistance (MR) option. Lakeshore MR uses 4-probe method for resistance measurement. In four probe method the sample is in the form of a thin wafer. The oven is connected with a heater to heat up the sample so that we can study the behavior of the sample resistivity with increase in temperature. Four equally spaced tantalum metal tips with finite radius are used in the four probe arrangement. To minimize sample damage each tip is supported by spring on the top of it. These four tips can travel up and down in mechanical way so that we can easily set the sample underneath of those four probes. We supply current through the outer two probes. Here we supply high impedance current. Across the inner two probes we measure voltage using a voltmeter. The voltmeter has the high input impedance; the inner two probes supply almost zero current in the circuit. In the 4 probe method voltage drop is eliminated from the potential measurement caused by contact resistance between probes and samples. The electric field is created when current has passed through the outer two probes (Fig-3.6) and the inner two probes measure the potential difference across them [3][14][15][18].

**Figure-3.6.** Electric field creation and measurement in four probe [15].
3.2.1.2 Resistivity Measurement by Four Probe Method

The resistivity across the entire area of the sheet is uniform. If any minority carriers are injected into the semiconductor, then most of the carriers recombine near the electrodes. As a result of that their effect on conductivity is negligible. There should be no surface leakage when the probes are in rest on the surface. The points lie in a straight line on the surface of the four probes that are used for resistivity measurement (Fig-3.7). Distance between the probes should be much larger compared to the diameter of the contact between metallic probes and the semiconductor [14][15][18]. For the bulk samples where thickness of the samples, \( w >> s \), (where \( s \) is the probe spacing) the resistivity \( \rho \) is given

\[
\rho = \left(\frac{V}{I}\right)2\pi s
\]  
(3.3)

Here \( V \) is the measured potential difference between the inner two probe, unit: volt, \( I \) is the current through the outer two probes, unit: Amp, \( s \) is the spacing between point probes, unit: meter, \( \rho \) is the resistivity, unit: ohm-meter.

![Figure-3.7. Resistivity Measurement by Four Probe Method [15].](image)

For a thin slice and non-conducting bottom surface where \( s >> w \), the resistivity \( \rho \) is given by

\[
\rho = \frac{\rho_0}{f\left(\frac{w}{s}\right)}
\]  
(3.4)
The value of \( f(w/s) \) is found from a table. For a smaller value of \((w/s)\), \( f(w/s) \) becomes the case of an infinitely thin slice where \( f(w/s) = (2s/w)\ln2 \)

So resistivity becomes

\[
\rho = \frac{\pi w V}{\ln2 I}
\]  

(3.5)

Using equation 3.3 we get sheet resistance, \( R_s \) which is

\[
R_s = \frac{\pi V}{\ln2 I}
\]  

(3.6)

### 3.2.2 The Two Probe Method

Typically when we measure resistivity \( \rho \) for a long parallelepiped shaped sample of uniform cross section or many times for a long thin wire-like systems of uniform cross section we use two probe method [15]. Here we measure the voltage drop with a voltmeter (V) across the subject resistor due to known current passes through the sample (Fig-3.8). A battery supplies current through the sample which is measured by ammeter (A). Let \( a \) is the cross section area and \( l \) is the area of the length of the sample then \( \rho \) is given by

\[
\rho = \frac{V a}{I l}
\]

(3.7)

![Figure-3.8. Two Probe Method [15.]](image)
Or resistance, $R$ is

$$R = \rho \frac{1}{a} = \frac{V}{I} \quad (3.8)$$

In our experiment we do all the measurements using two probe method.
CHAPTER IV
DATA AND ANALYSIS

4.1 Data and Graph

We have produced multilayer samples of cobalt and antimony keeping the cobalt layer thickness fixed at 1.0 nm. We varied the interceding layer of antimony. We made samples of 8 different thickness of antimony, ranging from 0.5 nm to 5.0 nm. We measured the decay of electrical resistance as a function of time and temperature for all of our samples. All of the measured resistances of our data are normalized resistances. The normalized resistance is the measured resistance divided by the maximum resistance ($R/R_{max}$). The temperature was held constant during this aging process. We measured the decay of resistance at various temperatures varying from 320K to 400K.

4.1.1 Co(1.0nm)/Sb(0.5nm)

![Figure-4.1](image)

**Figure-4.1.** Change of resistances for Co(1.0nm)/Sb(0.5nm).

Our first 10 bilayer sample consisted of Co(1.0 nm) and Sb (0.5 nm). We measured the decay of normalized resistance at 400K, 360K, and 340K (Fig- 4.1). As we can see the resistance increased with time. The initial resistances of the samples that we took for our measurement were very high. They ranged from 2.5 kohm to 4 kohm.
4.1.2 Co(1.0nm)/Sb(01.0nm)

The second 10 bilayer sample we made for our measurement was Co (1.0 nm) and Sb (1.0 nm). At 400K, 360K, and 340K we measured the resistance decay (Fig-4.2). As we can see in the graph resistances did not change with time. The initial resistances were around 1 kohm which were much lower than the Co(1.0)/Sb(0.5).

4.1.3 Co(1.0nm)/Sb(1.5nm)

Figure-4.2. Decay of resistances for Co(1.0nm)/Sb(1.0nm).

Figure-4.3. Decay of resistances.

Figure-4.4. S(t) graph for Co(1.0nm)/Sb(1.5nm).
In our next sample the antimony thickness was 1.5 nm. Here for the first time we saw the significant decay in the time and temperature dependent resistance (Fig-4.3). We measured decays at 400K, 380K, 360K, 340K, and 320K. All graphs demonstrate a sharp drop followed by plateau in resistance value. At 400K we got the total decay in 2000 seconds. As the temperature was decreased the decay time increased. The sample aged at 320K took approximately 3 and half days to fully decay. From the graph (fig-2.3) we see the decays to around 25% to 30% of the initial value. Initial resistance in this case is also much lower than our previous samples. In our five measurements the initial resistance averaged a value of approximately 300 ohms. The actual value for the initial resistances ranged from 270 ohms to 350 ohms. We also find the inflection points for all the decays in logarithm time scale as shown in the fig- 2.4. To find the inflection point we took the negative time derivative of normalized resistance (-[dR/dln(t)]) and plotted it in logarithm time scale. Here we see that inflection point increases with time as temperature decreases.

4.1.4 Co(1.0nm)/Sb(2.0nm)

The next sample we measured was 10 bilayers of Co(1.0 nm)/Sb(2.0 nm). We measured

![Graph](image1.png)

**Figure-4.5.** Decay of resistances Graph.

![Graph](image2.png)

**Figure-4.6.** S(t) graph for Co(1.0nm)/Sb(2.0nm)
the decay of normalized resistance at 400K, 380K, 360K, 350K, 340K, and 320K (Fig-2.5). Here we also saw a sharp drop followed by a plateau in resistance value. The sample aged at 400K took approximately 1000 seconds to fully decay. As the temperature was decreased the decay time increased. The sample aged at 320K took approximately 2.5 days to decay fully. In this sample the total resistance decays are larger than in the Co(1.0)/Sb(1.5). The full decays were around 45% of the initial value. Initial resistances of these measurements were varied from 300 ohms to 400 ohms. From the fig-3 we also can see how inflection points were shifted at various temperatures in logarithm time scale.

4.1.5 Co(1.0nm)/Sb(2.2nm)

In our next sample the thickness of antimony was 2.2 nm. We measured time and temperature dependent resistance decay at 380K, 360K, 340K, and 320K (Fig-4.7). Time taken for fully decay at 360K was almost 30,000 seconds and at 340 it was almost one and half day.

![Graph](image)

**Figure-4.7.** Decay of resistances Graph.

**Figure-4.8.** S(t) graph for Co(1.0nm)/Sb(2.2nm).

Decays were almost 45% for this set of sample from their initial value. From the figure 4.8 we can see distribution of inflection points for various temperatures.
4.1.6 Co(1.0nm)/Sb(2.5nm)

Figure-4.9. Decay of resistances.  

Figure-4.10. S(t) graph for Co(1.0nm)/Sb(2.5nm).

The thickness of antimony for our next sample was 2.5 nm. We recorded the declining of the resistance every 10K, started at 330K and concluded at 400K (Fig-9). Here we see at 400k time taken for fully decay is about 1000 seconds. At 360K it took almost 10,000 seconds and at 330K it took almost 100,000 seconds. The decays were almost 50% for all the temperatures of the initial values. Fig-4.10 shows us how inflection points are distributed in the logarithm time scale at various temperatures.

4.1.7 Co(1.0nm)/Sb(4.0nm)

We made our next sample keeping Sb thickness at 4 nm. We measured the decay of time and temperature dependent resistance at 400K, 360K, and 340K (Fig-4.11). In this case we saw almost 3000 seconds were taken for fully decay at 400K temperature and at 360K decayed time was almost 2 days. At 340K temperature we didn’t see the full decay after 5 days. The decays were almost 40% of the initial resistances. The fluctuation in the initial resistance was around an
average value 260 ohms in the range of 240 ohms to 300 ohms. We also plot negative time derivative (-[dR/dln(t)]) versus logarithm time as shown in the fig-4.12 to find the inflection points for those temperature.

4.1.8 Co(1.0nm)/Sb(5.0nm)

Our last sample that we measured was a 10 bilayer of Co(1.0nm) and Sb(5.0nm). Here at
first we saw some resistance decay then it started going up as shown in fig-4.13. The drops were around just 10% to 20% of the initial resistances. Initial resistances of these measurements varied from 320 ohms to 400 ohms.

4.2 Drop of Initial Resistances

As I said earlier we have observed the drop of initial resistances. In the following table 4.1 I list all the initial resistances and final resistances of my samples. Then I take the average of both initial and final resistances of every sample. Then I plot them in resistance vs thickness graph (fig-4.14 and fig-4.15).

**Table - 4.1.** List All the Initial Resistances and Final Resistances at Different Temperatures of My Samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>Temp. (K)</th>
<th>Initial Resistance(R$_0$)</th>
<th>Final Resistance(R$_f$)</th>
<th>Avg of R$_0$</th>
<th>Avg of R$_f$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Co(1.0nm)/Sb(0.5nm)</td>
<td>400</td>
<td>3313.285</td>
<td>5007.83</td>
<td>3273.257</td>
<td>4399.06466</td>
</tr>
<tr>
<td></td>
<td>360</td>
<td>4075.988</td>
<td>4947.695</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>340</td>
<td>2430.498</td>
<td>3241.669</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Co(1.0nm)/Sb(1.0nm)</td>
<td>400</td>
<td>947.794</td>
<td>988.785</td>
<td>972.895</td>
<td>1009.386</td>
</tr>
<tr>
<td></td>
<td>360</td>
<td>999.2233</td>
<td>1020.952</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>340</td>
<td>971.6678</td>
<td>1018.421</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Co(1.0nm)/Sb(1.5nm)</td>
<td>400</td>
<td>306.2892</td>
<td>225.3707</td>
<td>302.7843</td>
<td>231.42554</td>
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<tr>
<td></td>
<td>380</td>
<td>366.1452</td>
<td>262.8014</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>360</td>
<td>255.2624</td>
<td>198.1502</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>340</td>
<td>313.3967</td>
<td>250.8049</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>320</td>
<td>272.8282</td>
<td>220.0005</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Co(1.0nm)/Sb(2.0nm)</td>
<td>400</td>
<td>337.4796</td>
<td>194.197</td>
<td>340.1619</td>
<td>212.9826</td>
</tr>
<tr>
<td>---------------------</td>
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<td>-------------</td>
</tr>
<tr>
<td></td>
<td>380</td>
<td>380.8016</td>
<td>244.4097</td>
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<td>360</td>
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<td>184.0332</td>
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<td>244.1775</td>
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<td>315.719</td>
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<tr>
<td>Co(1.0nm)/Sb(4.0nm)</td>
<td>400</td>
<td>240.2005</td>
<td>140.0415</td>
<td>265.3566</td>
<td>181.192</td>
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<td>222.5803</td>
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<td></td>
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<tr>
<td>Co(1.0nm)/Sb(5.0nm)</td>
<td>400</td>
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<td>267.7089</td>
<td>359.0826</td>
<td>318.6109</td>
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<td>366.5822</td>
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<tr>
<td></td>
<td>340</td>
<td>360.3834</td>
<td>321.5416</td>
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**Figure-4.14.** Avg initial Resistances vs Thicknesses Graph.

**Figure-4.15.** Avg Final Resistances vs Thicknesses Graph.
4.3 Resistance as a function of Temperature

With sample of Co(1.0nm)/Sb(2.0nm) we also measured the normalized resistance variation as we changed the temperature systematically of the sample. In the first trial, depicted in the figure-4.20 (blue line), we started from a temperature of 100K and in steps of 5K went up to 360K and without any delay returned to a temperature of 100K. In subsequent trials (black and red line, fig-4.20) we waited for 20,000 sec at 360K before retracting out the path to 100K temperature. We also measured the decay of normalized resistance by cycling temperature back and forth from 340K to 360K as shown in the fig-4.21 (red line) to see how it compared with the graph of 360K and 350K temperature.

4.4 Arrhenius Plot

We plot all of our logarithm time of inflection points in 1/T (K⁻¹) scale as shown in the figures below (Fig-4.16,4.17,4.18,4.19,4.20) for all of our samples. This graph is well known
Arrhenius plot. In the fig-21 we show them in combined. From this graph we can easily find the activation energy. To find the activation energy ($E_a$) we measured the slope of the graph and
multiplied with Boltzmann Constant ($K_B$). In the table-4.1 we have listed activation energy ($E_a$) and $\ln(\mu_0)$ of our samples.

**Figure-4.23.** Combined Arrhenius plot.

**Table-4.2.** List of Activation Energy ($E_a$) and $\ln(\mu_0)$ of Our Samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>Activation Energy ($E_a$)</th>
<th>$\ln(\tau_0)$</th>
</tr>
</thead>
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<tr>
<td>Co(1.0nm)/Sb(1.5nm)</td>
<td>$6.548\times10^{-17}$ KJ/K</td>
<td>2</td>
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<tr>
<td>Co(1.0nm)/Sb(2.0nm)</td>
<td>$5.685\times10^{-17}$ KJ/K</td>
<td>2.2</td>
</tr>
<tr>
<td>Co(1.0nm)/Sb(2.2nm)</td>
<td>$8.814\times10^{-17}$ KJ/K</td>
<td>2.4</td>
</tr>
<tr>
<td>Co(1.0nm)/Sb(2.5nm)</td>
<td>$6.44\times10^{-17}$ KJ/K</td>
<td>2.5</td>
</tr>
<tr>
<td>Co(1.0nm)/Sb(4.0nm)</td>
<td>$7.62\times10^{-17}$ KJ/K</td>
<td>2.65</td>
</tr>
</tbody>
</table>
CHAPTER V

CONCLUSION

We performed a systematic study on Co/Sb superlattices varying Antimony (Sb) thickness while keeping Cobalt (Co) thickness constant at 1 nm. We studied the decay of electrical resistance in our samples as a function of time and temperature. The primary goal of my thesis was to find the optimal thickness of Antimony (Sb) for which the decay of electrical resistance at various temperatures is maximum. We observed nice decays of electrical resistance with thicknesses ranging from 1.5 nm to 4.0 nm of Sb. At 5.0 nm there is some initial decay of resistance but as time goes on the resistance increases. At 1.0 nm we did not see any change in the initial resistances over time. At 0.5 nm thickness of Sb we observed increases of resistance.

In our analysis of resistance as a function of temperature (Fig-4.14) we have seen a very interesting property. With the sample of Co(1.0 nm)/Sb(2.0 nm) we started with an initial temperature of 100K and in increments of 10K increased to 360K. We then immediately returned to a temperature of 100K with decrements of 10K. In subsequent trials we waited for 20,000 sec at 360K before retracing the path back to 100K. Later we did conducted another experiment where the waiting time was 56,000 seconds. In the first two cases we observed a negative slope all the way to the end which suggests our samples exhibit semiconductor behavior. In the third study from 100K to 360K we witnessed the same semiconductor behavior. However, when we were retracing the path back to 100K, after the waiting time of 56000 sec at 360K, we observed a positive slope from 260k to 100K. That positive slope suggests a metallic behavior in our sample. That means aging period plays a very important role in our Co/Sb superlattices. Our samples likely go through a semiconductor to metal transition as a function of aging period.
Another interesting observation is that the decrease of initial resistance in our samples appears to coincide with a decrease in their Sb thickness. In our Co(1.0nm)/Sb(0.5nm) sample initial resistances ranged from 2.5 kohm to 4 kohm. But in our next sample, Co(1.0)/Sb(1.0nm), the initial resistances were around 1 kohm, which were much lower than the Co(1.0)/Sb(0.5). In our next sample, with an antimony thickness of 1.5 nm, we observed a decay in initial resistance. The initial resistances of that sample ranged from 270 ohms to 350 ohms in our five measurements. The average value of these initial resistances is approximately 300 ohms which is three times lower than the Co(1.0)/Sb(1.0nm) sample and approximately 10 times lower than the Co(1.0)/Sb(0.5) sample. In our next four samples, Co(1.0)/Sb(2.0nm), Co(1.0)/Sb(2.2nm), Co(1.0)/Sb(2.5nm), Co(1.0)/Sb(4.0nm), the initial resistance was on average 300 ohms with a range of 240 ohms to 370 ohms. This observation suggests that current might be going through the Antimony (Sb). As we know Antimony is a semimetal, but in the nanoscale it acts like a semiconductor.

We have also studied Arrhenius plots of our data and haven’t observed any significant difference among the various thicknesses of Sb. In the Co(1.0)/Sb(4.0nm) sample we observed a slightly steeper line that suggests that the decay of resistances for that sample took longer than our other samples.
References


10- Kenning, G. G., Heidt, C., Barnes, A., Martin, J., Grove, B., & Madden, M. Thermally activated magnetization and resistance decay during near ambient temperature aging of


