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Polarization reversal in thin film lithium niobate on silicon

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Rice University, 1990
RICE UNIVERSITY

POLARIZATION REVERSAL
IN THIN FILM LITHIUM NIOBATE ON SILICON

by

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IN PARTIAL FULFILLMENT OF THE
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MASTER OF SCIENCE

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Abstract

Polarization Reversal in Thin Film Lithium Niobate on Silicon

by

Timothy Alan Rost

The spontaneous polarization of thin film LiNbO$_3$ has been shown to be reversible with the application of an electric field at room temperature. This electric field is applied across the sample in the form of a voltage pulse, and polarization reversal is detected either as a photocurrent reversal, or as a current transient whose integrated area is proportional to the spontaneous polarization. For 80 mil$^2$ size areas of thickness .3 $\mu$m, the fastest switching speed was 500 ns. The samples consisted of a silicon substrate, a thin film of LiNbO$_3$, and a metal contact to form an MFS (metal-ferroelectric-semiconductor) structure. The LiNbO$_3$ film was formed by rf sputtering LiNbO$_3$ onto heated $<111>$ silicon substrates.
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CHAPTER 1

Introduction

Much is known about the ferroelectric LiNbO₃. It has been one of the most intensely researched materials due to its large ferroelectric coefficients. Its spontaneous polarization of 71 μC/cm² [1], is among the highest of all known ferroelectric materials [2]. Despite this attention, relatively little is known about the reversal or switching processes that occur in LiNbO₃ at temperatures much lower than the Curie point. This is somewhat surprising since it is this property that is unique to ferroelectrics and allows LiNbO₃ to be classified as such.

There are several reasons why this investigation has not been pursued until now. The first is the unusually high Curie temperature of LiNbO₃ which is 1210° C [3]. Since the coercive field decreases as the Curie temperature is approached, the electric field necessary for polarization reversal at room temperature is very large. So large in fact that several researchers have termed LiNbO₃ a "frozen ferroelectric" at room temperature [4, 5]. This means that the material will break down electrically before a large enough coercive field can be applied to cause polarization reversal.

The second problem was that until recently there were no methods of preparing high quality thin films of LiNbO₃ [6-8]. Thin films are important in this work because of the large coercive field. One researcher [9], has reported this field to be near 300 kV/cm, which is in the range of electrical breakdown fields in solids. For a
sample of even modest size, say 10 μm, the necessary switching voltage would be nearly 300 V! By reducing the thickness of the material it is possible to bring the voltage necessary for switching to a more reasonable level. If the coercive field remains constant for decreasing thicknesses then a .5 μm film will require 15 volts to switch. These numbers are enticing from a device applications standpoint.

To this end thin ferroelectric films on silicon are even more interesting. "Ferroelectricians" have long recognized the potential for a memory device that uses a ferroelectric as the storage medium. Since data could be stored as a direction of polarization rather than as charge on a capacitor as in conventional dynamic random access memories (DRAM's) the data will still be stored even when the power is turned off. As a bonus, no refresh circuitry is needed and a ferroelectric memory cell is much more radiation hard than a DRAM.

The purpose of this research is threefold. The first problem is to determine the physical and electrical integrity of LiNbO₃ films on silicon. The physical structure is investigated with a variety of surface techniques to evaluate the crystallinity, uniformity, and stoichiometry. For electrical measurements, MFS capacitors are formed, and the current, capacitance, and conductance as a function of applied voltage is examined. The effect of illumination on these values can also reveal some of the ferroelectric properties and resulting band structure of these devices. Next, the switching parameters of the film are examined. This includes basic measurements such as switching speed, coercive field, and repeatability. Finally, an analysis of the switching transient is presented to provide some insight into the kinetics of polarization.
reversal in thin film LiNbO$_3$.

The thesis is organized in the following manner. Chapter 2 discusses the properties of ferroelectrics in general and the techniques for observing polarization reversal. Chapter 3 goes into more depth regarding specific properties of crystalline LiNbO$_3$. Chapter 4 considers the electrical characterization of MFS structures. Chapter 5 looks at previous research of polarization reversal in LiNbO$_3$ and the effects of depolarizing fields and reversal kinetics. Chapters 6 and 7 discuss the fabrication of MFS devices and their characterization.

It should be kept in mind that there are two separate ideas in this thesis. One is the polarization reversal of LiNbO$_3$. The second is the integration of this process into an MFS structure. The marriage of these two ideas gives a device in an MFS structure that can interact with the semiconductor through the polarization reversal process. Thus, it is necessary to discuss both the reversal process in LiNbO$_3$ and the characterization of thin film LiNbO$_3$ integrated into the MFS structure.
CHAPTER 2

Theory

2.1. Definitions

Ferroelectrics are a class of materials that are the electrical analogue of ferromagnets. Rather than a reversible magnetic domain as in a ferromagnet, ferroelectrics possess a reversible electric dipole, or polarization. Polarization reversal in a ferroelectric can be induced with the application of an electric field of the appropriate magnitude, direction, and duration. It is this property that is unique to ferroelectrics and make them worthy of consideration for applications such as electronic memory devices.

Ferroelectrics belong to a more general class of materials known as dielectrics. Dielectrics are materials that produce a polarization under the influence of an applied electric field. This polarization is the result of either of two interactions depending on the material. In non-polar dielectrics an applied field can slightly separate the normally coincident positive and negative charge associated with the unit cell. The applied field has induced a dipole moment in the unit cell and the sum of these moments add to give a macroscopic polarization.

In polar dielectrics this charge separation, or dipole moment, is an inherent part of the material's structure. If these dipoles are randomly oriented, applying an electric field can cause the dipoles to align creating a polarization. The classic example of
this is the H₂O molecule. In this molecule the electrons tend to be shared unequally, leaving the hydrogen atom with a slight positive charge. When a field is applied, this dipole tends to align with the field. When the field is taken away the dipoles randomly orient themselves so no net polarization can be detected.

*Piezoelectric*† materials are a subset of dielectrics that not only produce a polarization with an applied field but also when mechanically stressed. The stress causes the atomic bonds to become stretched or shrunken asymmetrically causing charge separation. Since this charge separation is the same for each cell throughout the

![Diagram](image)

**Figure 1.** The effect of an applied electric field on a dielectric material.

†There are different approaches to these definitions. Generally, each property is a subset of the previous one although it might be impossible to observe it. For example, guanidinium aluminum sulphate hexahydrate (GASH) is a ferroelectric although it decomposes before it reaches its Curie temperature. These definitions are mainly taken from Burfoot and Taylor, chapter 1 [10].
crystal, a macroscopic polarization can be detected.

In some materials a charge separation in the unit cell exists even in the absence of an applied electric field or stress. This charge separation is an inherent part of the crystal structure. These materials are known as pyroelectrics. When the temperature of a pyroelectric is changed, its atomic bonds become stretched asymmetrically, changing the value of the dipole moment. If the temperature change is fast enough, free charge in the crystal will flow to compensate for the change in polarization. This transient effect is known as the pyroelectric effect or pyroelectric current. This current is greatest if measured between the two faces of the crystal’s polar axis.

It is possible to reverse the direction of the spontaneous polarization in some pyroelectrics by applying an electric field that is larger than the structural forces separating the charge. These materials are known as ferroelectrics and the electric field necessary to reverse the polarization is the coercive field. A working definition of a ferroelectric would be a material that has at least two stable directions of polarization and the possibility of electrically switching between those two or more states. From the previous discussion, it should also be noted that ferroelectrics are at the same time dielectric, piezoelectric, and pyroelectric. In fact, ferroelectrics often have the largest figures of merit for these properties since a dipole is an elemental part of the unit cell.

The spontaneous polarization of a ferroelectric can be aligned in a given direction through a procedure known as poling. Poling simply means aligning all the unit dipoles along a a single polar axis. This is usually accomplished by heating the fer-
roelectric up past its *Curie temperature*, applying a relatively small voltage across the material, and cooling down past the Curie point with the voltage still applied. The Curie temperature is the temperature that separates the ferroelectric phase from the *paraelectric* phase. Once again the analogy from magnetic materials is seen by the Curie temperature separating the magnetic phase from the paramagnetic phase. Thus, with a small voltage, the desired alignment can be imposed.

2.2. Reversal Techniques

In theory, the dipoles can also be aligned at temperatures below the Curie temperature with the application of a voltage pulse of the appropriate magnitude, polarity, and duration [10]. This is known as *switching* the ferroelectric. It is different

---

**Figure 2.** Sawyer-Tower hysteresis loop circuit.
from poling in that the sample is usually in thin film form and it is not heated to near its Curie point. Switching characteristics are normally determined by applying a sinusoidal voltage across the ferroelectric as shown in figure 2. This circuit is known as the Sawyer-Tower hysteresis loop circuit after the researchers who first used it [11]. This can be plotted on an oscilloscope as a polarization versus electric field curve (P-E loop). Figure 3 compares a linear dielectric and a polar dielectric.

For a ferroelectric the polarization can completely reverse with increasing electric field so a P-E loop will have some hysteresis as shown in figure 4. In this diagram \( E_c \) is the coercive field, \( P_s \) is the saturation polarization, and \( P_r \) is the remnant polarization. Generally, the term spontaneous polarization (also \( P_s \)) is more commonly used and is used interchangeably with the saturation polarization. If the P-E loop is reasonably square, then \( P_r \) is very nearly \( P_s \). In this case the spontaneous polarization is used interchangeably with \( P_r \) also.

Another way to observe switching in a thin film ferroelectric is shown in figure 5 [10]. Here, the capacitor of the Sawyer-Tower circuit is replaced with a resistor. This resistor is much smaller than the resistance of the sample so that most of the voltage drop is across the sample. If a voltage pulse is applied across the ferroelectric, the resulting charge flow can be recorded as a voltage developed across this current viewing resistor. With this set-up there are three things that can happen: 1) the linear portion of the dielectric can charge without switching, 2) the polarization can switch, or 3) the sample can electrically break down.
Figure 3. Polarization vs electric field characteristics for a) a linear dielectric and b) a polar dielectric.

Figure 4. Polarization vs applied electric field for a ferroelectric.
If the applied voltage is less than the coercive field the system will behave as a linear dielectric capacitor. When the pulse is applied, a voltage spike will appear across the resistor as a result of the charging of the linear dielectric component. This situation is shown in figure 6.a). The size of this spike is proportional to the charge that flowed, which is equal to

\[ Q = CV \]

the capacitance times the applied voltage.

An applied voltage of greater than the coercive field will produce the switching transient of figure 6.c). This transient is the combined charge flow due to the linear
Figure 6. a) applied voltage pulse and b) non-switching, c) switching, and d) breakdown currents.
dielectric component (initial spike), and a hump due to the non-linear (switching) component. The area under the switching transient represents the amount of compensation charge that flowed due to the reversal of the polarization. The integrated current is related to the polarization by:

\[ \int i \, dt = 2P_s A \]

If the sample breaks down, most of the voltage will then appear across the current viewing resistor for the duration of the pulse. This is illustrated in figure 6.d. This is an important parameter as it determines a working range of voltages that can cause switching.

The pulse switching method also provides some other advantages over the Sawyer-Tower method in looking at polarization reversal. Since the applied voltage is constant over the pulse duration, it is possible to accurately determine the coercive field. Pulsing also limits space charge accumulation helping to prevent breakdown. Once the switching transient is recorded it is possible to tell a great deal about the reversal process. The total switching time is easy to determine since it is simply duration of the switching transient.
CHAPTER 3

Properties of Lithium Niobate

3.1. Ferroelectric Properties

LiNbO$_3$ is of interest because it has several useful properties. These properties have been summarized in Appendix A. It has one of the largest values of spontaneous polarization of all known ferroelectrics [2]. It is chemically very unreactive. Its Curie temperature of 1210$^\circ$C [3] is well beyond any normal active use temperature. Most of all, it has been deposited by r-f sputtering techniques and has been shown to retain many of the bulk values necessary for device applications [7,8,12]. Since these films were sputtered on silicon substrates, compatibility with existing silicon technology is also noted.

A diagram of the unit cell of LiNbO$_3$ is shown in figure 7. It is evident from this diagram that the motion of both the Li$^+$ ion and Nb ion are confined to move in a straight line along the c axis of the crystal. Stable positions for the Li$^+$ ion occur at .7 Å above or below the oxygen plane depending on the orientation of the polarization. The potential energy curve for this Li motion is given in figure 8. Since there are two possible orientations for the polarization, both of which lie on the polar axis, LiNbO$_3$ is often termed a "1-d ferroelectric".
Figure 7. Atomic arrangement of LiNbO$_3$ a) before and b) after reversal.
3.2. Optical Properties

Another interesting property of LiNbO$_3$ is the *bulk photovoltaic effect*. When light of the proper wavelength is uniformly incident on a crystal of LiNbO$_3$, an open-circuit voltage develops across the polar faces of the crystal. Even more interesting is the fact that this field can be as high as several hundred volts per centimeter. This effect is obviously different from p-n junction photovoltages or voltages associated with the Dember effect as these effects are limited by the band gap of the material. The voltages associated with the photovoltaic effect can be orders of magnitude higher than the band gap. Glass *et al* [13] have reported a saturated open circuit
photovoltage in excess of $10^5$ V/cm for iron doped LiNbO$_3$ crystals. This curious effect is only exhibited in a few other materials.

One explanation that has been put forth by Amodei [14] is diagrammed in figure 9. The absorption of intense light (such as that conveniently provided by a laser) by the crystal causes some electrons to be photoexcited from donor sites. These donor sites are caused by impurities, which, for the metal dopants such as Fe, are thought to occupy Nb positions. Because this defect occupies an asymmetrical lattice position,

![Diagram](image)

Figure 9. One theory to explain the photovoltaic effect [15].
the excited carriers can be excited with a preferential direction. After drifting or diffusing it is possible for them to become retrapped after a short period of time. Since this leads to charge separation on a macroscopic level, fields will be induced within the crystal.

By taking advantage of this effect it is possible to determine the orientation of the +c axis in LiNbO₃ by shining light on the sample and recording the direction of the photocurrents. This will consist of two components as shown in figure 10. The transient effect is due to the pyroelectric effect and is caused by the light source heating the crystal. As mentioned before, this causes the lattice to stretch, changing the value of $P_s$. Free charge must flow to compensate for this change. The steady-state

![Graph of photocurrents associated with LiNbO₃](image)

Figure 10. Photocurrents associated with LiNbO₃
value is due to the bulk photovoltaic effect. Thus, if a positive current is detected with the structure shown in figure 11, the +c axis must be pointing into the silicon. If

![Diagram](image)

Figure 11. Production of photocurrents in MFS structure.

†Positive current in this case is defined as current flowing from the metal electrode, through the ammeter, to the semiconductor.
a negative current is detected the +c axis must be directed away from the silicon. Thus, in this manner, it is possible to determine the polarity of the voltage necessary to cause reversal.

An effect closely related to the photovoltaic effect is the photorefractive effect. When the sample is illuminated with light of sufficient intensity the fields created in the manner described for the photovoltaic effect can cause changes in the index of refraction of LiNbO$_3$. This is due to the electro-optic effect known as the Pockels effect. This change is given by

$$\Delta n = -\frac{1}{2} r n^3 E + K$$

where $r$ is the electro-optic coefficient, $n$ is the index of refraction with no applied field, and $K$ is the birefringence.

3.3. Electrical Properties

At temperatures below the Curie point, LiNbO$_3$ is an insulator. The value of the resistivity in single crystal LiNbO$_3$ has been measured to be near $10^{18}$ $\Omega$-cm by several groups [16,17]. Very little is known about the conduction processes in LiNbO$_3$ except for evidence of ionic transport at elevated temperatures [18]. At room temperature the electron mobility is very low possibly due to strong electron-phonon coupling as reported by Chanussot and Glass [19]. Perhaps this low mobility and the presence of defects couple to make polarization reversal difficult at temperatures much lower than the Curie point.
A calculation of the electronic band structure of LiNbO$_3$ is shown in figure 12. This band structure was calculated by Kam et al [20] using a plane-wave-Gaussian (PWG) basis set with an $X_h$ exchange. From this they found the smallest valence-conduction gap to be 3.47 eV and an indirect gap of about 2.3 eV. This agrees with the generally accepted value of 3.72 eV as experimentally determined by Clark et al [21]. Also with this calculation it was possible to calculate the spontaneous polarization by assuming that the boundaries of the molecule are represented by minimum surface contours of the electronic charge density. The spontaneous polarization calculated in this manner was 97 $\mu$C/cm$^2$ which agrees remarkably well with the meas-

![Figure 12. Band structure of LiNbO$_3$ plotted along different paths in the Brillouin zone. The shaded area represents the band gap [20].](image-url)
ured value of 71 μC/cm².

Dopants have also been added to LiNbO₃, mainly in an effort to enhance the photorefractive effect. Transition metal impurities have shown to be useful in this regard particularly Fe, Mn, and Cu. It is thought that these metals provide both extra charge carriers and form traps, both of which are necessary to form the internal field. Dopants have also been used to form waveguides in bulk LiNbO₃. One method of doing this is in-diffusing Ti into a LiNbO₃ substrate [22]. This provides a change of refractive index of .04 (extraordinary) and light wave, such as that provided by a HeNe laser (632.8 nm), to be guided. In-diffusion of V and Ni was also successful.

3.4. Chemical Stability

Chemically, LiNbO₃ is very stable. This can be a problem when attempting to delineate areas for devices by etching techniques. There are only a few etches that seem to etch LiNbO₃ at all. This can be an advantage if a selective etch is used to etch the silicon and not the LiNbO₃ film. Transmission electron microscopy (TEM) could then be used to examine the crystallinity of the film. Since our films are very thin (<1 μm) this might prove to be somewhat difficult. Etches have also been used to help identify the orientation of the crystal. Since some etches have differing rates for etching the positive and negative ends of the c axis they can be used to differentiate antiparallel domains.

The first article discussing the etching of LiNbO₃ appeared in 1965 by Nassau et al [23]. They used one part HF and two parts HNO₃ at the boiling point (110º C).
This is a selective etch, etching the negative dipole end faster than the positive dipole end. The negative dipole surface forms pits upon etching that are readily visible under a microscope with x200 magnification. The positive dipole surface does not etch as rapidly and can easily be distinguished from the negative dipole surface after about 10 minutes of etching.

In a subsequent paper [24], Nassau et al discuss some other etches that produce similar results. Molten potassium hydroxide at 400° C was said to darken one of the domains after etching for 15-60 seconds. Presumably, this is similar to the HF-HNO₃ etch in that it causes etch pits to form on the negative surface scattering the incident light better, making it appear darker. Another etch that produced similar results was a combination of two parts 30% hydrogen peroxide and one part 30% sodium hydroxide at 50° C for 15 minutes.

Later papers [25] have generally used the HF-HNO₃ etch for polishing and etching. Since the etching rates are so low the only practical use of this etch is to differentiate domains or for fine polishing. Another use as mentioned before would be to etch through the silicon to be able to use the film for TEM analysis.
CHAPTER 4

Electrical Measurements

Very little work has been done on using conventional MIS (metal-insulator-semiconductor) measurement techniques to characterize the electronic properties of MFS structures. This is surprising since it is possible to tell a great deal about the electrical and ferroelectric properties of the thin film by measuring current versus voltage (I-V), capacitance versus voltage (C-V), conductance versus voltage (G-V), and photocurrents. Ideally it would be nice to completely characterize the film with these techniques, switch the polarization, and characterize the film again. This would help provide insight into the switching process.

Measuring I-V and the photocurrent are straightforward. By measuring I-V one can use the relation

$$\rho = R \frac{A}{l}$$

to find the resistivity of the film. In the expression $R$ is the measured resistance of the MIS capacitor, $A$ is the area, and $l$ is the film thickness. This method relies on the assumption that the majority of the voltage drop will be across the film. For LiNbO$_3$ on silicon, this is a good assumption. In this manner the resistivity of the film can be calculated and compared to the bulk. A good film with no pinholes or other defects should yield a resistivity on the order of $10^{18} \, \Omega \cdot \text{cm}$.
By shining light on the sample it is possible to determine various constants associated with the bulk photovoltaic effect. Lines and Glass [26] have shown that the photocurrent can be found from

\[ J = k \alpha I \]

where \( J \) is the photocurrent due to the bulk photovoltaic effect, \( k \) is the anisotropy constant, \( \alpha \) is the absorption coefficient, and \( I \) is the intensity of the light source. A plot of \( k \) and \( \alpha \) is shown in figure 13. By measuring the photocurrent, calculating these constants, and comparing them with known values of a high quality single domain crystal gives another indication as to the quality of the film.

Figure 13. The absorption coefficient \((\alpha)\), and the anisotropy constant \((k)\), of a single domain LiNbO\(_3\) crystal as a function of wavenumber (from Lines and Glass [26]).
The C-V, G-V measurement has the potential for providing more insight into the electronic processes in this structure. Basic measurements of the dielectric constant can be made by measuring the capacitance and using

\[ C = \varepsilon_r \varepsilon_0 \frac{A}{d} \]

to calculate the relative dielectric constant \( \varepsilon_r \). Here \( \varepsilon_0 \) is referring to the vacuum dielectric constant, \( A \) is the area of the capacitor, and \( d \) is the thickness. Another indication of the film quality is given by calculating \( \varepsilon_r \) and comparing it with known values for bulk LiNbO_3. Since LiNbO_3 is birefringent, the crystallographic orientation of the film must be known.

The previous examples ignore the fact that the ferroelectric film is interacting with the carriers in the semiconductor. This interaction is the result of the face of the

![Diagram of MFS structure](image)

Figure 14. Band diagrams of MFS structure.
ferroelectric attracting compensation charge. Figure 14 shows the resultant band bending at the semiconductor surface which depends on the orientation of the polarization, and the doping type of the semiconductor. For example, if the polarization of the ferroelectric is directed into the semiconductor, negative charge will arrive at the semiconductor surface to compensate this positive bound charge. This will result in an accumulation layer in an n-type semiconductor, and an inversion layer in a p-type semiconductor. If the polarization is switched, the opposite will occur.

The high frequency C-V curve for an ideal MIS structure is shown in figure 15. For p-type material accumulation will occur at negative voltages. As the voltage is increased first a depletion layer, and then an inversion layer will form. Here, the

![Diagram of C-V curve for p-type and n-type MIS capacitors]

Figure 15. High frequency C-V characteristics of an MIS capacitor.
capacitance is reduced due to the series addition of the depletion layer capacitance. The relation between the minimum and maximum capacitance with a ferroelectric material replacing the insulator is given by Wu [27] as

\[
C_{\text{min}} = C_{\text{max}} \left[ 1 + \left( \frac{\varepsilon_f}{\varepsilon_s} \right) \left( \frac{4\varepsilon_s k T \ln(N_D/n_i)}{q^2 N_D} \right)^{1/2} \right]^{-1}
\]

The most important application of capacitance and conductance measurements, however, is the extraction of information regarding the surface states at the ferroelectric-semiconductor interface [28]. These states are the result of 1) the interruption of the periodic lattice, 2) fixed surface charge, 3) mobile ions, 4) ionized traps. Of particular use would be a comparison between the surface charge density, \( Q_{ss} \), and the spontaneous polarization \( P_s \). In the ideal film, these two would be equal. There are various methods of obtaining this information involving capacitance and conductance methods.

Perhaps the easiest method is known as the differentiation procedure. The existence of surface charge will cause a shift in the high frequency C-V curve since

\[
Q_{ss} = C_i (\Delta V) \text{ coul/cm}^2
\]

where \( C_i \) is the insulator capacitance per unit area, and \( \Delta V \) is the shift of the ideal C-V curve along the voltage axis. Graphical differentiation can then be used to find the surface state density per unit energy

\[
N_{ss} = \frac{1}{q} \left[ \frac{\partial Q_{ss}}{\partial \psi_s} \right] \text{ states/cm}^2/\text{eV}
\]
where \( \psi_s \) is the surface potential. By measuring a low frequency C-V and comparing it to the high frequency plot information about the time constant \( \tau \) of these states can be found.

There are two other capacitance type methods that are worth mentioning although they will not be used in this work. The first is the integration procedure whereby \( N_{ss} \) can be obtained directly from a low frequency C-V curve. The advantage with this method is that no graphical differentiation is required to find \( N_{ss} \). The second method is known as the temperature procedure. Here, the temperature is changed as the bias is changed to maintain a flat-band condition. This gives a graph of flat-band voltage versus temperature which can be used to find \( Q_{ss} \) and \( N_{ss} \).

The plot of G-V can also be used to extract information regarding surface states at the ferroelectric-semiconductor interface. This analysis is also due to Sze [28], with a much more involved description found in Nicollian and Brews [29]. An equivalent circuit model of the interface is shown in figure 16. Since a plot of \( G_p/\omega \) vs \( \omega \tau \) will be maximized when \( \omega \tau \) is 1, \( \tau \) can be calculated. At this point \( G_p/\omega \) will be exactly \( C_s/2 \). This is used in the expression

\[
N_{ss} = \frac{C_s}{qA} \text{ states/cm}^2/\text{eV}
\]

to find the surface state density. It is thought that this value will be much higher than an interface such as the Si-SiO\(_2\) system due to the high depolarizing fields present. A model considering interface states is shown in figure 16.
Figure 16. Equivalent circuit model of the interface [28].
CHAPTER 5

Previous Research

5.1. Lithium Niobate Switching

Previous work on polarization reversal in LiNbO$_3$ has been done with the goal of producing optical quality crystals. Since the crystals for optical applications are necessarily large, polarization reversal studies concentrated on trying to switch relatively large samples. Because of the high resistivity and large coercive field, switching LiNbO$_3$ proved to be very difficult. Generally, thinner samples were used to bring the switching voltage down to a more reasonable level.

The earliest and most relevant work was done at Bell Telephone Laboratories in 1967 and 1968. Two papers were published. The first by Wemple et al [2] explained the basic results and a follow-up paper by Camlibel [9] explained the experimental arrangement and procedure. This group was the first to directly measure the spontaneous polarization in LiNbO$_3$ to be $71 \pm 2 \, \mu$C/cm$^2$. This is the generally accepted value for $P_s$ in LiNbO$_3$. They were also successful in determining $P_s$ for other ferroelectrics with large polarizations, most notably LiTaO$_3$.

The experimental setup for this measurement is shown in figure 17. A pulse is applied across the sample through the pulse generator as shown. If the sample switches then there will be a charge flow proportional to switching area $A$ and the spontaneous polarization $P_s$. The capacitor $C_o$ to ground essentially integrates this
charge flow over time and produces a voltage. This voltage is related to the charge flow as:

\[ Q = 2 P_s A + \varepsilon_r \varepsilon_0 \left( \frac{V}{t} \right) A \]

since on the capacitor,
\[ Q_0 = C_o V_o \]

then \( P_s \) can be calculated from:

\[
P_s = \frac{1}{2} \left[ C_0 \frac{V_o}{A} - \varepsilon_r \varepsilon_0 \left( \frac{V}{t} \right) \right]
\]

where \( t \) is the thickness of the sample, \( C_0 \) is the value of the capacitor, \( V_o \) is the voltage measured on the capacitor, \( \varepsilon_0 \) is the vacuum dielectric constant and \( \varepsilon_r \) is the dielectric constant of the sample measured along the axis of polarization. Since it is possible to measure \( V_o \) and \( C_0 \) quite accurately, an accurate measurement of \( P_s \) can be obtained.

The samples were 30-35 \( \mu \)m thick and were contacted using LiCl electrodes. Both metal and liquid electrodes were used but metal electrodes caused a substantial increase in the coercive field. LiTaO\(_3\) was also switched but the polarization did not remain switched when the pulse was removed. It was still possible to calculate the spontaneous polarization since the flow of charge saturated at high fields.

Haycock and Townsend [30] used a novel approach to reverse the polarization in an already poled sample. Since it has been noted that reversal involves the Li\(^+\) motion across the oxygen plane, reversal would occur more readily if the oxygen triangle could be widened. Haycock and Townsend reasoned that a way to accomplish this would be to expose the sample to ionizing radiation. They hoped that the excited oxygen ions would form a metastable molecular ion when relaxing. A diagram of this situation is shown in figure 18. Note the wide area for Li\(^+\) motion. This metastable
state has to exist just long enough for the Li+ to traverse the oxygen plane.

With ionizing radiation, domain reversal was possible with an applied field of 10 V/cm at 600°C. The electron beam had an energy of 1.8 MeV. With this method it was also possible to reverse the polarization in LiTaO₃. For the same beam energy, an applied field of 900 V/cm at 400°C was necessary to cause reversal. These temperatures are much lower than the Curie temperatures for both of these materials.

There have been a number of papers in Soviet literature addressing this issue. Evlanova et al [31] used constant electric fields to induce polarization reversal in 1 mm thick crystals. "Convenient" reversal times were of the order of minutes, resulting from an applied field of 85 kV/cm at 130°C. A plot of reversal current as a function of time for their results is given in figure 19. The exact reversal time was never
given. Their work was interesting in that at each numbered point along the reversal current curve shown in figure 19, they removed the applied field, cut the sample down the middle, and etched to reveal the domain growth between the electrodes. By integrating the charge under the curve they obtained a $P_s$ of $60 \pm 5 \, \mu C/cm^2$.

Another Soviet team reported partial polarization reversal for Fe doped LiNbO$_3$ crystals [32]. Kovalevich and coworkers found that partial ferroelectric switching was occurring in regions of a LiNbO$_3$ crystal illuminated with a He-Cd laser (442 nm) with a power density greater than 10 W/cm$^2$. This reversal was detected by Barkhausen signals which are simply "quantum" jumps in reversal current due to the polarization reversal of a single domain. These domains are thought to be switching due to the high fields ($10^4$ to $10^5$ V/cm) associated with the charge movement brought
about by the bulk photovoltaic effect.

This work was extended by Kamentsev and Rudyak [33] who applied electric fields of the same order of magnitude as the internal photorefractive fields determined by Kovalevich et al to crystals of LiNbO$_3$. By applying voltages on the order of 10 kV/cm they observed a charge flow corresponding to partial polarization reversal. This behavior was not observed in non-ferroelectric cuts (for instance, perpendicular to the c axis) of LiNbO$_3$ at even the highest attainable voltages. This switching was done at room temperature although it was not stated how thick the crystal was or what the contact material was. A typical switching transient is shown in figure 20. As can be seen, the switching time is on the order of tens of minutes. This is attributed to a dielectric viscosity in LiNbO$_3$ that is very large. In order to observe switching either the dielectric viscosity must be reduced or the frequency of the

![Graph](image)

Figure 20. Polarization reversal current in LiNbO$_3$ (Kamentsev and Rudyak).
external field must be decreased.

These groups all claim that it is possible to switch LiNbO$_3$ and the assumptions of early researchers are not true. The other point to be remembered is that these groups were all working with optical size crystals with the exception of Wemple et al who used 30-35 μm platelets. Thus, the idea of looking for switching in thin film LiNbO$_3$ seems well founded.

5.2. Depolarizing Fields

Another issue that must be dealt with when discussing thin ferroelectric films is that of depolarizing fields. It is important to understand how they arise, how their presence can influence the stability of the film, and what effect they will have on the polarization reversal process. To help answer these questions a series of papers [34-39] have been written discussing depolarizing fields in various structures involving thin ferroelectric films and metal and/or semiconducting contacts. It is helpful to analyze their results and apply them to the LiNbO$_3$ system.

From electromagnetic theory it is known that a polarized material will produce polarization charge wherever $P$, the polarization, is nonuniform. Stated explicitly this is

$$\rho_b = -\nabla \cdot P$$

where $\rho_b$ is the bound polarization charge. Such a condition certainly exists at the faces of a ferroelectric crystal as shown in figure 21.a. This can be represented as a parallel plate capacitor with a charge of $P_s$ on the top plate and $-P_s$ on the bottom.
\[ \oplus \ominus = \text{bound charge} \]

\begin{array}{c}
\begin{array}{c}
\text{P}_s \\
\uparrow \\
+ + + + + + + + \\
\downarrow \\
- - - - - - - - \\
\end{array} \\
\begin{array}{c}
a) \text{ferroelectric crystal} \\
b) \text{equivalent capacitor} \\
\end{array}
\end{array}

Figure 21. Parallel plate capacitance representation of the polarization charge.

This situation is shown in figure 21.b. The electric field, \( E \), can be found from

\[ C = \frac{Q}{V} \]

\[ \frac{\varepsilon_0 \varepsilon_r A}{d} = \frac{P_s A}{V} \]

\[ E_d = \frac{P_s}{\varepsilon_0 \varepsilon_r} \]

\[ = \frac{0.71 \text{C/m}^2}{32 \times (8.854 \times 10^{-12} \text{F/m})} \]
\[ = 2.5 \times 10^7 \text{ V/cm} \]

This example shows that the ferroelectric must support an electric field that is larger than the dielectric breakdown strength of most materials! Also recall that Cam-libel used voltages on the order of \(10^5\) V/cm to switch the polarization. This shows that the depolarizing field cannot be ignored since with this simple example this system has been shown to be unstable.

Fortunately, *compensation charge* exists in real crystals to help reduce the effect of the depolarizing field. This charge can arrive at the surface by conduction through the crystal (since it's not a perfect insulator) or by some external means such as a connection between electrodes placed on the polar surfaces of the crystal. It is because of compensation charge that it is not possible to directly measure the polarization, only measurements of the change in polarization can be made. During a switching event, this charge needs to be removed before the polarization can reverse.

With the concept of compensating charge in mind, it is possible to attempt a more realistic examination of depolarizing fields in thin LiNbO\(_3\) films. This theory was first worked out by Mehta *et al* [34]. Consider the film of LiNbO\(_3\) sandwiched between two gold electrodes as shown in figure 22. This arrangement produces the charge distribution as shown in figure 22.b. Using this it is possible to calculate the potential and electric field distribution as shown qualitatively in figure 22.c and 22.d. To find an exact expression for the compensation charge \(q_c\), and the depolarization field \(E_d\), it is necessary to solve Poisson's equation
Figure 22. a) metal-ferroelectric-metal structure, b) charge distribution c) potentials d) electric fields.
$$\frac{dD}{dx} = \rho(x)$$

using the boundary conditions imposed by this structure. This derivation has been done by Mehta et al and the final result for the compensation charge is

$$q_e = -P \frac{1/2\varepsilon_f}{l_s/\varepsilon_e + \varepsilon_f/l_f} = -\theta P$$

with

$$\theta = \frac{\varepsilon_f/l_s}{2 \varepsilon_f/l + \varepsilon_e/l_s}$$

Here, $\varepsilon_e$ is the relative dielectric constant of the electrode, $\varepsilon_f$ is the relative dielectric constant of the ferroelectric, $l_s$ is the electrode screening length, and $l$ is the thickness of the ferroelectric film. The depolarization field can be expressed as

$$E_d = -\frac{P}{\varepsilon_f} \left[ \frac{2\varepsilon_f/l}{2\varepsilon_f/l + \varepsilon_e/l_s} \right] = -\frac{P}{\varepsilon_f\varepsilon_e} \left[ 1 - \theta \right]$$

Plugging in .6 Å for the gold electrode screening length, 32 for $\varepsilon_r$ gives the following results:

$$q_e = 0.996 P_s$$

$$E_d = 9.5 \times 10^4 \text{ V/cm}$$

Also note that for large crystals $l$ will be large making the depolarizing field small. Another way to reduce the depolarizing field is to make $l_s$ very small. This can be done by using electrode materials that have very short screening lengths, ie. metals.
The screening lengths were calculated with the use of the Thomas-Fermi potential as described by Kittel [1]. The results of these tabulations for some common electrode materials are tabulated in table 1. At this point, the screening length of the electrode material seems to be an important factor in the magnitude of the depolarizing field. It essentially is a parameter that determines how close charge can get to the ferroelectric surface. It is this separation of bound polarization charge and compensation charge that determines the magnitude of the depolarizing field in the perfect ferroelectric.

<table>
<thead>
<tr>
<th>Material</th>
<th>Electron density</th>
<th>Screening length</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ag</td>
<td>$5.85 \times 10^{22} \text{ cm}^{-3}$</td>
<td>.59 Å</td>
</tr>
<tr>
<td>Au</td>
<td>$5.90 \times 10^{22} \text{ cm}^{-3}$</td>
<td>.59 Å</td>
</tr>
<tr>
<td>Cu</td>
<td>$8.54 \times 10^{22} \text{ cm}^{-3}$</td>
<td>.55 Å</td>
</tr>
<tr>
<td>Al</td>
<td>$18.06 \times 10^{22} \text{ cm}^{-3}$</td>
<td>.49 Å</td>
</tr>
</tbody>
</table>

Table 1. Thomas-Fermi screening lengths of various metals.

It must be realized that a number of approximations have gone into this theory (see especially Burfoot and Taylor, § 12.3):

1. The ferroelectric film has been assumed to be perfectly insulating. Of course, this is not consistent with such concepts as ferroelectric band gaps and band bending.

2. Interface and surface states have been ignored along with bulk trap states.
3. $P_s$ is assumed to be homogeneous throughout the film. This ignores the existence of domains, whose formation can also help reduce the depolarization field.

4. The one-dimensional approximation is used whereby $P_s$ is assumed to consist of only one component, $P_z$. This precludes any consideration of polarization spreading at the edges of the film.

In general, finding the depolarizing field associated with a body of known polarization is very complicated even for simple geometries. This is partly because $E_d(x,y,z)$ and $P(x,y,z)$ are interdependent. For this reason researchers have looked for geometries in which a uniform polarization creates a uniform depolarization field. A classical example of such a geometry is a spheroid. For this and other similar geometries the depolarizing field can be expressed as

$$E_x = -N_x P_x$$

$$E_y = -N_y P_y$$

$$E_z = -N_z P_z$$

where $N_x, N_y, N_z$ are known as depolarization factors.

Since the actual structure under consideration consists of metal-ferroelectric-semiconductor layers, consideration must be given to the stability of this system due to the much larger screening length of the semiconductor. This creates an asymmetry in the compensating charge distribution which would certainly increase the depolarizing field. It is also thought that since compensating charge needs to be present for the
system to be stable the doping of the semiconductor is important. If the semiconductor is doped n-type, negative carriers are available for compensation. If positive charges are needed for compensation, they would have to be thermally generated, a comparatively slow process. To get around this problem it might be necessary to put a metal layer between the ferroelectric and the semiconductor to get stable switching.

5.3. Reversal Mechanism

The shape of the switching transient is important since it reveals how the charge is flowing as a function of time. This essentially provides a "snapshot" of the reversal process. By examining this transient the kinetics of the reversal process can be understood. This problem was tackled independently by early researchers who were just interested in the reversal processes of their own materials. In 1962, a paper by Fatuzzo [40] examined the switching processes of several different ferroelectric materials and attempted to fit the results with a theoretical model.

The basic premise of this model is that there are two phenomena responsible for the switching transient shape; sideways movement and forward growth. The interplay between these two mechanisms determine the shape of the switching transient. In some materials one mechanism may dominate producing a transient with a shape characteristic to that mechanism. In other materials this shape can be a combination of the two mechanisms.

Forward growth is simply spikes of antiparallel domains progressing from the surface of the applied voltage to the opposite side as shown in figure 23.a. Sideways growth is shown in figure 23.b. Here the forward growth spikes are shown expanding
sideways until they reach another domain with a polarization in the same direction. Typically, a reversal starts as forward growth spikes followed by sideways domain movements which coalesce until the entire area is reversed.

Fatuzzo gives examples of materials such as in BaTiO$_3$ whose switching current is dominated by sideways domain growth. Thus the current at any given time is proportional to the area of reversed domains. The transient for this type of reversal is shown in figure 23.a. The current starts at zero, rises to a maximum, and decays with the same time constant. An example of a material whose reversal is dominated by forward growth is guanadinium aluminum sulfate hexahydrate (GASH). The shape of this transient is shown in figure 23.b. At t=0 a maximum number of domains are nucleated so the transient will rise quickly to a maximum and slowly decay. In materials where both of these mechanisms are involved, the transient will be intermediate between these two shapes such as in tetramethylammonium trichloro mercurate (TTM) or triglycine sulfate (TGS). In general, reversal dominated by forward growth will be faster than that dominated by sideways growth.
Figure 23. Switching transient of material a) dominated by forward growth, b) dominated by sideways growth, and c) intermediate between these two (after Fatuzzo [40]).
CHAPTER 6

Fabrication

6.1. Preparation

A detailed account of the apparatus and method used for preparing thin LiNbO$_3$ films on silicon is given elsewhere [6]. However, for completeness, it is necessary to outline the procedures used and discuss some important deposition parameters. There are basically three steps involved in preparing LiNbO$_3$ films on silicon. These include cleaning the substrate, growing the film, and providing a means to make contact to the wafer. Since a discussion of film growth involves the quality of the resultant film, x-ray diffraction and lattice mismatch calculations are also included in this chapter.

The substrates were single crystal <111> silicon wafers doped either n or p type. The resistivities for both types were .1 $\Omega$/cm, 1 $\Omega$/cm, or 10 $\Omega$/cm. The wafers were 38-40 mils thick, mainly to prevent cracking during handling. They were chemically cleaned prior to deposition with the following procedure:

1. To remove residual organics and metals a solution of H$_2$O-NH$_4$OH-H$_2$O$_2$ (5:1:1) was used. The Electronic grade chemicals were used with concentrations of 29% for ammonium hydroxide and 30% for hydrogen peroxide. The water was deionized and filtered. This solution was placed cold on a hotplate and heated to 75-80° C. The wafers were then placed in the solution and the temperature maintained at 80° C for 10 min. The solution was then overflow-quenched with deionized water for
5 minutes.

2. Next, to strip the thin, native oxide film, a solution of HF-H₂O (1:50) was used. Electronic grade, 49% hydrofluoric acid was used and the water deionized and filtered as before. The wafer was placed in this solution for 15 seconds. When the wafer is removed from this solution it should look very clean and dry as a clean silicon surface repels the HF solution. To remove the HF solution it was agitated in deionized water for 30 seconds.

3. Finally, to remove any remaining atomic or ionic contaminants, a solution of H₂O-HCl-H₂O₂ (6:1:1) was used. This was heated to 80°C and the wafers were placed in this hot solution immediately after step 2 for 10 minutes. After this time the wafer was overflow-quenched as in step 1.

4. The wafer could be left rinsing in deionized water while the vacuum is opened and the system readied for the wafer. At this point the wafer is placed in a spinner and a final deionized water rinse applied and spun until dry. Now it is clean and ready to be placed in vacuum.

6.2. Sputtering

A number of various techniques have been employed in the effort to obtain high quality thin film LiNbO₃. Among these are chemical vapor deposition (CVD) [41], epitaxial growth by melting (EGM) [42], liquid phase epitaxial growth [43-45], and various types of sputtering [46-55]. These techniques were tried mainly to produce films of high enough quality for integrated optics applications. Sputtering was chosen
because earlier work has shown promise for producing good films.

The parameters used to produce the films in this work are outlined in table 2. It is evident from this table that there are a number of parameters that

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Frequency</td>
<td>13.56 MHz</td>
</tr>
<tr>
<td>Sputtering Gas</td>
<td>Ar/O₂ (60%:40%)</td>
</tr>
<tr>
<td>Sputtering Pressure</td>
<td>5 - 10 mTorr</td>
</tr>
<tr>
<td>Forward RF Power</td>
<td>100 - 150 Watts</td>
</tr>
<tr>
<td>Target-Substrate Spacing</td>
<td>6 cm</td>
</tr>
<tr>
<td>Substrate Temperature</td>
<td>550°C</td>
</tr>
<tr>
<td>Target Dimension</td>
<td>4 in</td>
</tr>
<tr>
<td>Growth Rate</td>
<td>5 - 10 Å/min</td>
</tr>
</tbody>
</table>

Table 2. LiNbO₃ sputtering parameters [6].

significantly affect the crystallinity of the film. Previous work (as referenced above), has shown trends in the values necessary in several of these parameters to produce crystalline films. One is the heating of the substrate to near 550°C and the other is the use of a reactive sputtering environment.

The reactive sputter gas mixture was about 3 mTorr argon with 2 mTorr oxygen. The reactive sputtering procedure was used to compensate for the oxygen lost by dissociation in the sputtering process. The target consisted of a water cooled, r-f electrode in which LiNbO₃ powder with 10% Li₂O was placed. These materials were thoroughly mixed and pressed to a depth of about 1 mm. The target to substrate spacing was approximately 6 cm.
The addition of Li$_2$O to the target was necessary to compensate for the loss of lithium and oxygen from the LiNbO$_3$ film during sputtering. Oxygen loss seems to particularly effect film quality as oxygen depleted films had a chalky grey appearance, were not transparent, and were amorphous as indicated by Bragg x-ray diffraction scans. This corresponds with results of previous researchers [46, 53-55].

A stainless steel substrate holder was used to heat the wafer to 550° C during the deposition. This temperature has been empirically shown by Bragg x-ray diffraction to produce oriented, crystalline or polycrystalline films. 550° C seems to give the sputtered atoms that land on the wafer enough surface mobility for them to find their correct lattice positions. Below this temperature the films are not highly oriented.

The orientation of substrate also affects the crystallinity of of the LiNbO$_3$ film. <111> substrates that were heated to 550° C produced films with just the (006) diffraction peak evident whereas <100> films sputtered under the same conditions had several orientations. It is thought that since the <111> direction in silicon, and the c axis of LiNbO$_3$ crystal both exhibit 3-fold symmetry, growth is initiated with the c axis perpendicular to the silicon surface. This condition is ideal for applications involving the ferroelectric surface of the film such as switching.

6.3. Lattice Mismatch

The unit cell of silicon along the <111> axis is shown in figure 26. The dimension of the unit cell in this direction is:

\[ a \sqrt{2} = 5.431 \, \text{Å} \quad \sqrt{2} = 7.681 \, \text{Å} \]
Figure 24. Effect of substrate temperature on crystallinity.
Figure 25. Effect of the substrate orientation on crystallinity.
Figure 26. Unit cell of <111> silicon [29].

To determine the extent of the lattice mismatch it is necessary to determine the corresponding lattice parameter in LiNbO$_3$. Since the hexagonal lattice parameter for LiNbO$_3$ is $a_H = 5.148\,\text{Å}$ then,

$$\Delta = \frac{5.148}{7.861} = 33\%$$

Obviously this is not a good match and indicates that the film has many dislocations and possibly some strain at the interface.

Since it is probably the oxygen bonding that is important it might be helpful to look at the spacing of the oxygen atoms and compare this with available bonding
sites on the $<111>$ silicon surface. The width of a side of the oxygen triangle is shown in figure 27 to be 3.38 Å [56]. Nicollian and Brews have shown [29] that for SiO$_2$ the length of a side of an oxygen triangle is 2.27 Å. The mismatch between these triangles is

$$\Delta = \frac{2.27}{3.38} = 33\%$$

It must pointed out that although the lattice mismatch is important, there is still an oxide layer present on the silicon surface despite efforts to reduce it. In fact, it has been noted by Cholapranee [8] that such a layer is necessary to prevent damage to the silicon during sputtering and a thermal oxide was deliberately grown prior to sputtering. It also should be noted that oxide layers are known to greatly reduce the migration of atoms from the film into the silicon. Perhaps this is another important case to

![Diagram of oxygen triangles](image)

**Figure 27.** Oxygen triangles associated with a) the LiNbO$_3$ molecule and b) SiO$_2$ molecule.
be made for the oxide - it will help prevent the migration of ions (especially Li\(^+\)) into the silicon. This is particularly important when trying to make repeatable measurements.

A thermal oxide as discussed by Nicollian and Brews [29] is amorphous in nature as shown in figure 28. As can be seen the thermal oxide does not possess long range order as does the quartz lattice, but some short range order is evident by the formation of "rings" of five or six silicon atoms. There is still some question as to the role the oxide layer plays in the crystallinity of the deposited film and of the importance of the lattice mismatch when viewed in this context.

The presence of the (006) peak indicates that an ideal condition exists for switching. Since the (006) peak corresponds to the axis of polarization, the films are oriented such that the c axis is normal to the silicon surface. This is fortunate because LiNbO\(_3\) is a 1-d ferroelectric meaning that it has two stable directions of polarization. Since the axis of polarization is perpendicular to the silicon surface the polarization can be directed into or normal to this surface. In theory, it is possible to switch between these two polarizations by applying a voltage between the LiNbO\(_3\)-silicon structure of the appropriate magnitude and polarity.

6.4. Contacts

To insure reliable and repeatable electrical characterization of LiNbO\(_3\) films it is important to be able to make a good contact to the wafer. For this reason it is necessary to choose a material to contact the LiNbO\(_3\) and a means to connect this contact with the measuring instruments. A further constraint is that the film be semi-
Figure 28. a) Quartz crystal lattice and b) the amorphous structure of a thermally grown oxide [29].

transparent to allow photocurrent measurements.

Little is known about ohmic contacts to extremely high resistivity materials. Even less is known about contacts to high resistivity ferroelectrics. There have been two papers published about contacting ferroelectrics. The first by Wemple [57] explained that since oxygen vacancies in these materials served as donors, either blocking or ohmic contacts could be formed depending on the contacts’ affinity for
oxygen. From his criteria it is evident that LiNbO$_3$ is a p-type ferroelectric oxide and thus following his discussion, gold should be used as a contact material.

In another paper, Pulvari and Srour [58] used a "graded" electrode to contact bismuth titanate (Bi$_4$Ti$_3$O$_{12}$). This process involves first making contact to the insulator with a thin semiconducting layer, and then placing the metal layer on top. They reported a substantial reduction in the coercive field and no distortion of the hysteresis loop for a period of more than three years. This is essential from an applications standpoint.

The problems encountered with using gold as a contact is that since it needs to be very thin and does not adhere to the surface of the LiNbO$_3$ film very well, it is easily scraped off. Another important point is that since the resistivity of LiNbO$_3$ is so high, the surface of the wafer must be clean otherwise a path of lower resistance might be found in the "crud" along the surface to the silicon. This would significantly alter any measurement of the MFS structure. Also because the resistivity of the LiNbO$_3$ film is so high the issue of an ohmic contact might be moot since even a back biased diode could provide the small currents being dealt with.

Typically the electrodes were evaporated on the film as soon as possible after deposition. The wafers were placed in the evaporator against an aluminum mask which had an array of holes of known dimension drilled in it. This formed an electrode pattern on the front of the wafer as shown in figure 29. The evaporator was usually pumped overnight or until a pressure of less than $10^{-6}$ Torr was achieved. The current necessary for deposition for gold or aluminum was about 130 amps.
6.5. TI Devices

Texas Instruments has cooperated in this research by fabricating silicon wafers with test devices on them. These are <111>, 10-15 ohm-cm p-type wafers that can be used to fabricate MFS structures by a molybdenum liftoff process. This process is shown in figure 30. A layer of LiNbO$_3$ is first sputter deposited onto the devices, followed by a layer of gold. $\text{H}_2\text{O}_2$ is then used to etch the molybdenum and isolate areas.
of LiNbO$_3$. After this step, capacitors are isolated and can be tested. Of specific interest are MFS capacitors formed by this method that are 80 mil$^2$ and 900 mil$^2$.

Although only the capacitors are used in this work the wafers also have transistor structures on them in which the LiNbO$_3$ replaces the gate oxide. The gates have areas of 25 x 25, 25 x 2, and 2 x 2 microns. To isolate these transistor structures it is necessary to remove the plasma oxide covering the source and drain areas. This is done by etching the oxide with HF until the source and drain probe electrically clear.
Figure 30. Lift-off procedure [6].
CHAPTER 7

Experimental Apparatus

7.1. Probe Station

The probe station consists of a gold coated wafer chuck with connections for a mechanical pump. The mechanical pump holds the wafer in place and helps insure a good contact with back of the silicon wafer. The chuck was isolated from ground so that measurements could be made in a "floating" mode. This simply means that both measuring lines are not connected to ground so the effect of cable capacitance, for example, in a C-V measurement cancel each other out. Care was taken to make sure the measurement cables were of approximately the same length.

The metallic dots evaporated on the surface of the film were contacted by a 5 mil tungsten probe, electrochemically sharpened to a .35 μm point. The probe has a nickel shaft measuring 20 mils that inserts into a Micromanipulator© probe holder (Model 77). This holder is mounted on an x-y-z micropositioner. The shaft of the probe is bent in an "S" shape to provide some flexibility to the probe so it doesn’t punch through the film and also to maintain tension once contact has been made. The fine tip on the probe is necessary to punch through any oxides that may have formed on the metallic contact.

Since LiNbO₃ is photosensitive it was necessary to have a light-tight enclosure. This enclosure was grounded to provide shielding during a measurement. For meas-
urements that required a spot on the wafer to be illuminated, a mirror was mounted above the sample, and the light was directed in through a shutter mounted on the side of the enclosure. For most illuminated measurements a green helium-neon laser (543 nm) was used. As discussed previously, this is because green is absorbed very strongly in LiNbO₃.

7.2. Measuring Equipment

Capacitance versus voltage information was obtained by connecting the sample to an HP4280A 1 MHz C-V meter as shown in figure 31. This instrument can be operated in a floating mode whereby the capacitances associated with the cables can

![Diagram of wafer chuck and probe arrangement]

Figure 31. Wafer chuck and probe arrangement.
be eliminated. The HP4280A is controlled by a Texas Instruments Personal Computer (TIPC) through an IEEE-488 bus. A program was written to access the HP4280A through the bus and plot out the results on an HP7180 plotter. A schematic of this set-up is shown in figure 31.

![Experimental set-up diagram](image)

*Figure 32. Experimental set-up.*
The HP4140B was used to help find current versus voltage. This instrument combines a programmable DC voltage source with a picoammeter. Since the resistivity of LiNbO$_3$ is very high, currents on the order of picoamps will need to be measured. A box was constructed to enable connection to the probe station for I-V measurements. A diagram of this box, and the wiring to the sample are shown in figure 30. This instrument was also controlled by the TIPC through the IEEE-488 bus. Since the time constants when dealing with LiNbO$_3$ are usually quite large, a loop was included in the program to allow data to be taken from 1 to 99 minutes per data point. This allowed a long settling time for a measurement at each voltage. The source code for the programs used to measure capacitance vs voltage and current vs voltage are given in Appendix B.

The photocurrents were measured with several pieces of equipment. A Keithley 601 Electrometer was used as a current amplifier to measure the small currents associated with the photocurrents. The electrometer produced a voltage proportional to the current measured. This voltage was used as input for the HP4145A (Semiconductor Parameter Analyzer) which converted this voltage back to the measured current mathematically and plotted the result as a function of time. Since the screen of the 4145A can be dumped to a plotter, it was relatively easy to obtain decent plots of the photocurrents. The method for obtaining these plots is given in Appendix B.

7.3. Pulse Application

The switching pulses were applied with the apparatus shown in figure 32. This consisted of a pulser (SKL model 503A), which launches a pulse into a 50 Ω coax
cable. Since the impedance of the pulser is 50 Ω and the sample impedance is very large a 50 Ω termination/feed-thru is placed close to the sample. If the polarization is reversed with the pulse a current will flow through the 50 Ω current viewing resistor. This can be monitored with the aid of an oscilloscope with a camera. The oscilloscope is triggered by the pulser and captures on film the resulting current transient.

Another method of delivering pulses to the sample was with a capacitor charging/discharging circuit. The capacitor was charged with a Kepco power supply.
and discharged with a mercury wetted relay. A relatively large capacitor (1 mFd) was used to insure that the applied voltage was constant during a switching event. The only limitation on the magnitude of the applied voltage was the breakdown voltage of the capacitor.
CHAPTER 8

Results

The first hint as to the possibility of reversing the ferroelectric domains in thin film LiNbO$_3$ is found in research by Fabiny in 1985 [12]. In it he discovered a contact, that when illuminated, exhibited a photocurrent in a direction opposite to those previously measured. The only explanation for this is the existence of a reversed ferroelectric domain in the film. This also shows that at least two orientations are possible, one with the +c axis of the film directed primarily into the silicon substrate, and one with it directed primarily out of the substrate.

The first film in which switching was attempted was 16c. The photocurrent was measured prior to switching and is shown in figure 34. Single, 100 ns pulses of increasing magnitude were applied until the photocurrent was reversed. This was observed at 100 volts and is shown in figure 34. It is interesting to note that the magnitude of the photocurrent was slightly larger after the reversal. This indicates that either the number of domains contributing to the photocurrent has increased or that the degree of domain alignment is more perpendicular with the surface.

The next sample in which switching produced a photocurrent reversal was sample 1B. This sample did not switch with single pulses so a series of short pulses were applied. The pulses were supplied by the SKL pulser and were 10 ns in duration with a period of roughly 15 ms. The voltage was ramped from 0 to 150 volts in 15 seconds. This method of pulsing was used mainly in an attempt to prevent
Figure 34. Photocurrent reversal for sample 16c.
breakdown which can occur at lower voltages when DC or pulses much longer than 10 ns are used. After the pulses were applied, the photocurrent was observed to be reversed. The sample was left overnight and the photocurrent was measured again, 12 hours after reversal, and was still opposite of the the photocurrent prior to reversal. The original photocurrent, and the photocurrent measured 12 hours post reversal are shown in figure 35. The post 12 hour measurement showed no change in the magnitude of the photocurrents from a measurement made immediately after reversal.

In an effort to quantify the switching process, more effort was invested in the characterization of the physical structure of the LiNbO₃ films, the electrical properties of the MFS structure, and in switching experiments on MFS structures of known dimensions. The goal was to be able to monitor the charge flow during a switching event and compare it to the amount expected for the ideal case.

8.1. Physical Characterization

In addition to inspection of the films by optical microscopy, a number of other techniques were employed to determine the quality, stoichiometry, uniformity, crystallinity, and grain size of the films. These included Bragg x-ray diffraction, Auger Electron Spectroscopy (AES) depth profiling, Rutherford Backscattering (RBS), Scanning Electron Microscopy (SEM), and Secondary Ion Mass Spectroscopy (SIMS). The tests were all done on one film, sample 3/12/88.

First, the stoichiometry of the film was examined by RBS. This information is shown in figure 36. RBS is a nondestructive method of determining elemental composition throughout the bulk of the film. RBS uses 2.4 MeV helium ions and
Figure 35. Photocurrent reversal a) before switching and b) post switching 12 hours.
measures the energy of the scattered ions. The energy lost by the scattered ions is a
function of the element that it scatters from. The vertical lines in figure 36 mark ener-
gies that backscattering is predicted for O, Si, and Nb. Unfortunately, Li cannot be
detected with this technique. High angle (170°) measurements were made to charac-
terize deeper portions of the film.

The width associated with each peak occurs from energy lost as the particle
travels through the film. It is possible to determine the ratio of oxygen to niobium in
the film by dividing the ratio of the areas for each peak by the square of the atomic
number. In this manner the ratio of oxygen to niobium in the film was determined to
be 3, within the accuracy of the experiment. The thickness was calculated by deter-
mining the energy loss (peak width) and comparing it with the loss in LiNbO₃ which
has been calculated and has units of MeV/(mg/cm²). This allows the calculation of
the areal density, which, for this plot is 76 µg/cm². Dividing this by the mass density
of LiNbO₃ (4.64 gm/cm³) gives a thickness of .16 µm for the film. This agrees with
the value of .14 µm determined with interferometry.

Next, a depth profile of the same film was made with AES. This is shown in
figure 37. With AES it is possible to resolve lithium so a complete analysis of the ele-
mental composition throughout the sample was possible. The depth profile was made
by repeatedly sputter etching the sample with Ar+ ions and acquiring an Auger spec-
trum. To obtain the relative amount of each element in the film, it was necessary to
compare the measured Auger spectra with a single crystal LiNbO₃ calibration spec-
trum. To obtain a true depth profile it is also necessary to correct for the differences
Figure 36. RBS data for LiNbO$_3$ film on silicon
Figure 37. AES depth profile of LiNbO$_3$ film on silicon.
in the sputter rates of LiNbO$_3$ and silicon. These corrections have been taken into account in the plot shown in figure 37.

The composition was relatively constant throughout the film. The film ends abruptly at about 1550 Å where the lithium and niobium signals drop off. The point at which the oxygen level decreases (1800 Å) signifies the transition to pure silicon. It is difficult to completely determine the composition of the interface region due to the depth profiling process. The width of the interface is approximately 500-600 Å. A small amount of carbon was also detected at the surface of the sample, but this has been removed from the plot for clarity.

![SEM photograph of the LiNbO$_3$ film's surface.](image-url)
To examine the surface morphology and to look for ferroelectric domains SEM was used. A photograph of the surface of the film, magnified 50,000 times is shown in figure 38. The majority of the surface is smooth and uniform. If ferroelectric domains of opposite orientation were present, they would show up as light or dark regions since ferroelectric domains exhibit surface charge. No such regions are observed in this photograph, possibly indicating that they are preferentially oriented in one direction.

The white spots in the SEM photograph were examined with energy dispersive x-ray spectroscopy (EDS). With this technique they were determined to have a higher niobium to oxygen ratio than stoichiometric LiNbO$_3$. This information is shown in figure 39. Unfortunately, it was not possible to determine if these were niobium rich or oxygen deficient areas since EDS only gives the relative amounts of the two elements.

It is also noted that it is extremely difficult to resolve domains with SEM due to the high resistivity of LiNbO$_3$. The sample becomes electrically charged and further reduces the imaging capability. Generally, a thin layer of gold was sputtered onto the surface and electrically grounded. This helped eliminate the charging problem, but unless a very thin layer of gold was used, impaired the resolution of the surface structure. For the SEM photograph shown in figure 38 no conducting overlayer was used in order to resolve the ferroelectric domains but this limited the time spent analyzing any one area. It may not have possible to resolve ferroelectric domains in this film due to this surface charging problem.
8.2. Electrical Characterization

Small areas of LiNbO$_3$ on silicon were isolated using the liftoff process as described earlier. Specifically, the electrical properties of the 80 and 900 mil$^2$ capacitors were examined. The initial measurements of the electrical properties are shown on the following pages. Figure 40 shows the photocurrent measurement of the film. This corresponds to the +c axis of the polarization being oriented out of the silicon as shown in figure 11. Thus, in order to achieve reversal, positive pulses need to be applied. The other parameters prior to pulsing are shown in figures 41-43. The resistivity of the device $1.3 \times 10^{12}$ $\Omega$-cm. A correlation between the intensity of the photocurrent and the laser intensity was not attempted as the probe itself blocked a good deal of laser light from reaching the sample. The intensity of the laser was 75 mW/cm$^2$ at 543 nm.
Figure 40. Photocurrent measurement of MFS structure.

Figure 41. Current vs voltage for MFS structure.
When the dielectric constant was calculated using the capacitance of figure 42, with the device in accumulation, the value was much lower than the bulk value of 32, measured along the c axis. By illuminating the device with 632.8 nm laser light a new C-V curve was obtained. This C-V curve is shown in figure 44. Calculations using the value of the capacitance in accumulation, under illuminated conditions, gives a value of 27 for the dielectric constant.

To explain these results it is necessary to postulate the existence of a depletion layer in the silicon. Such a layer could result, in certain cases, from the diffusion of Li+ ions during deposition into the silicon. An oxide layer helps prevent this, but Li+ ions are very mobile, especially at the elevated temperatures used for deposition. During deposition Li+ would enter the silicon substrate by diffusion and form an n-type layer. The effect of this layer could then be lessened during a measurement by flooding the n layer with carriers. This is accomplished by creating carriers with the 632.8 nm laser light.

Using the above information it is possible to draw a band structure of the resulting interface. This proposed band structure is shown in figure 45. Since both the C-V curves and photocurrent measurements indicate that the +c axis is oriented normal to the surface, a layer of positive charge can reside in the semiconductor at the semiconductor-ferroelectric interface. Next to this is an n type region formed by Li+ diffusion. Lithium is very mobile in silicon and can certainly be present due to the lattice mismatch. The diffusion coefficient \( D_o \), is \( 2.5 \times 10^{-3} \) cm\(^2\)/s for lithium in silicon with an activation energy of .66 eV. At 600° C for 6 hours this corresponds to a
Figure 42. Capacitance vs voltage for MFS structure.

Figure 43. Conductance vs voltage for MFS structure.
diffusion length of .57 cm! After this region a transition back to p type material takes place. When this structure is flooded with carriers created by 632.8 nm laser light, the effect of the depletion region is lessened, making it possible to measure the capacitance due primarily to the LiNbO₃ layer.

In order to illustrate this more clearly it is helpful to consider a circuit model of this structure as shown in figure 46. $C_f$ represents the capacitance due solely to the ferroelectric layer and $C_d$ and $R_d$ are the parallel capacitance and resistance of the p-n junction. The resistance of the ferroelectric layer has been ignored since it is extremely high. When 632.8 nm laser light is incident on the p-n junction, $R_d$ is
Figure 45. Proposed band structure to explain the illuminated capacitance shift.

decreased, shunting the capacitance $C_d$. This allows measurement of the capacitance of the ferroelectric layer. When the capacitance measurement is made in the dark, this resistance is not decreased and the series combination of $C_o$ and $C_d$ is measured. If the assumption is made that the doping caused by Li donors is equal to the original
number of acceptors in the bulk Si, then for a linearly graded junction, \( C_d \) is calculated to be 4.4 pF. This corresponds to a depletion width of approximately 1.3 \( \mu \)m. This is in agreement with the dark measured value of 5.3 pF. For this model \( R_d \) was calculated to be \( 2.3 \times 10^5 \) \( \Omega \).

The C-V curves also indicate a positive threshold shift of roughly 4 volts. In conventional MOS devices this shift indicates the presence of negative charge located in the oxide, near the oxide-semiconductor interface. In an MFS structure as discussed in chapter 4, this charge will be present as bound polarization charge. This

\[a)\]

\[b)\]

Figure 46. Simplified circuit model of the device with \( a \) no illumination and \( b \) illuminated with 632.8 nm laser light.
agrees with the photocurrent measurements, which indicate that the +c axis is oriented away from the silicon surface. Using 41 pF as the insulator capacitance and 4 volts as the threshold shift yields a surface charge density of .32 μC/cm². This is much lower than the bulk value of 71 μC/cm². One explanation for this could be in the attraction of compensation charge in the LiNbO₃ film to the negative bound polarization charge. This could lessen the effect of the bound polarization charge on the carriers in the semiconductor.

The 900 mil² capacitors did not fare as well during the liftoff. A common failure mechanism was the partial or complete removal of the gold electrode. As a result, only two 900 mil² devices were available for testing. The results were qualitatively the same as the 80 mil² devices. Table 3 summarizes the electrical parameters of the capacitors.

<table>
<thead>
<tr>
<th>Area</th>
<th>80 mil²</th>
<th>900 mil²</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness</td>
<td>~ .3 μm</td>
<td>~ .3 μm</td>
</tr>
<tr>
<td>Relative dielectric constant</td>
<td>27</td>
<td>21</td>
</tr>
<tr>
<td>Resistivity</td>
<td>1.3 × 10¹² Ω-cm</td>
<td>3.8 × 10¹² Ω-cm</td>
</tr>
<tr>
<td>Photocurrent</td>
<td>-3 pA</td>
<td>-15 pA</td>
</tr>
<tr>
<td>Electrode material</td>
<td>Au</td>
<td>Au</td>
</tr>
<tr>
<td>Electrode transmittance</td>
<td>~ 1-5%</td>
<td>~ 1-5%</td>
</tr>
</tbody>
</table>

Table 3. Physical parameters of MFS structures.
8.3. Switching Data

After the sample had been characterized, pulses from the capacitor pulser of the appropriate polarity were applied. Typically, a 10 volt pulse was applied and the resultant current transient was viewed on the oscilloscope. If no switching occurred, the voltage was increased by 10 volts and the process repeated. A series of such pulses is shown in figure 47. The initial spike is the linear dielectric component and the "hump" is the polarization reversal current. This is twice the amount of the polarization equivalent charge since the polarization has to completely reverse direction. Note that the voltage of the applied pulse is flat during the entire photograph.

For this particular device the polarization switched back to its original orientation after the pulse was removed. This is shown by the fact that the photocurrent did not reverse and that later pulses with the same polarity also caused reversal. Thus, it was not possible to characterize the device after switching the polarization to its second possible orientation (in this case with the +c axis directed into the silicon).

It is thought that this "switch back" might be caused by the asymmetry of the electrodes. In particular, if part of the compensation charge is fixed in the intermediate oxide layer, it may not be possible in this time regime to completely reorganize with compensation charge of the opposite polarity. During a switching event the new orientation would be energetically unfavorable and it would switch back to its original orientation.

The area under the curve in figure 47 for a switching transient is:
Figure 47. Switching transients in LiNbO$_3$. On all plots the scale is 5 V/division vertical, 500 ns/division horizontal. (a) 120 volt pulse, non-switching (b) 130 volt pulse, switching (c) 130 volt pulse, non-switching, and (d) 130 volt pulse, switching.
\[ q = (1.5 \text{ squares})(5\text{V}/50\Omega)(500\text{ns}) \]

\[ = 75\text{nC} \]

Remembering the integrated current

\[ q = 2P_sA \]

gives

\[ P_s = \frac{q}{2A} \]

\[ = \frac{75\text{nC}}{2(0.000516 \text{ cm}^2)} \]

\[ = 73 \mu\text{C/cm}^2 \]

which agrees remarkably well with the accepted value of 71 \mu\text{C/cm}^2.

Another switching event on a second 80 mil\textsuperscript{2} device was recorded and is shown in figure 48. The spontaneous polarization of this device was calculated to be 43 \mu\text{C/cm}^2 using the same method as outlined before. It is possible to more accurately determine the polarization of this device since the time scale was expanded to allow a better calculation of the area under the curve. This device switched at 120 volts. This device also exhibited the switch-back phenomenon. The switching time for this device was longer, nearly 800 ns for complete reversal. For both devices the coercive field was 4 \times 10^6 \text{ V/cm}. 
A total of five 80 mil² capacitors exhibited switching of 8 tested. Of these devices, 2 were captured on film as switching and the rest were observed on the oscilloscope. The three that did not switch broke down at voltages slightly larger than the voltage used to switch. The breakdown field for these samples for this method of pulsing was approximately $6 \times 10^6$ V/cm. The two 900 mil² capacitors available for testing also broke down before switching.
From the data presented in figures 47 and 48 it is impossible to unambiguously determine what the switching mechanism is. It can be argued that for the first 80 mil\(^2\) sample the primary reversal mechanism is forward growth. This is due to the fact that the reversal current reaches a maximum immediately and then decays. This makes sense since LiNbO\(_3\) is a 1-d ferroelectric and the film is very thin. In the second sample however, at least some of the reversal current is due to sideways growth. The shape of the curve and the longer reversal time both agree on this point. Since the spontaneous polarization was measured to be lower for the second sample, perhaps the crystallinity of the film effects the reversal mechanism.
CHAPTER 9

Conclusions

9.1. Perspective

Recently there has been a great deal of interest in the properties of ferroelectric thin films. This interest has been fueled in part by their application in nonvolatile memories. There are a number of other areas that ferroelectric films are making contributions to. The waveguiding and optical processing capabilities of ferroelectric films are of technological importance for various applications in integrated optical devices. These films are generally grown on insulators such as sapphire, quartz, or fused silica. Never before have crystalline films of LiNbO₃ been deposited on conducting or semiconducting substrates. As a result, relatively no research until now has been focused on looking at and understanding the electrical and optoelectric properties of these films.

This work has shown that the spontaneous polarization of thin film LiNbO₃ on silicon is reversible under an applied electric field at room temperature. This is the first look at the possibility of using the switching nature of thin film LiNbO₃ in a device. LiNbO₃ has several attractive properties including chemical stability and large spontaneous polarization. This would provide the best possible signal to noise ratio in a device. Of particular importance is the demonstration of switching on silicon. This allows the consideration of novel devices that use the immediately available fabrica-
tion technology and electronic properties of silicon.

9.2. Possible Devices

Potentially the most useful device would be a transistor in which the gate oxide is replaced with a ferroelectric material. A schematic of this structure is shown in figure 49. This device could operate as a memory element. The simplest mode of operation would be one in which a bit of information is written by applying a voltage pulse of the appropriate polarity to the gate electrode. This would either leave the polarization oriented the same way or reverse it. Depending on which way it is oriented, it will either form an accumulation layer or an inversion layer in the semiconductor. This information could be read out simply by measuring the resistance of the channel.

Another mode in which transistors of this type might operate would be in a "photovoltaic mode". Here voltages caused by the bulk photovoltaic effect could be used to shift the flat band voltage of the transistor. As mentioned earlier these fields can be as high as $10^5$ V/cm for iron doped LiNbO$_3$, so a 1 μm film could, in theory, produce a 10 volt shift. Thus, it might be possible to couple optical signals to electrical signals in a most unique way.

An interesting possibility is using both the switching and photovoltaic nature of the films. Dharmadhikari et al [59] have observed the reversal of the polarity of the photovoltage in BaTiO$_3$ with polarization reversal. This effect has also been observed in several LiNbO$_3$ on silicon films. Thus, barring the presence of any interface effects, light of a certain intensity will shift the transistor's characteristics a certain
amount, and after switching, the same light intensity will correspond to a negative shift of the same amount.

9.3. Future Research

This work has shown that it is possible to switch the polarization of thin films of LiNbO$_3$ deposited on silicon by rf sputtering. There is much more research to be done to fully quantify and understand this process. Part of this is an ongoing process of producing high quality films and understanding which of the myriad of controllable parameters are crucial to film quality. A few of these have been positively
identified such as substrate temperature, deposition rate, target stoichiometry, and reactive gas mixture.

Closely related to the deposition process is the physical and electrical characterization of the resulting films. Each film needs to be fully characterized before any definite conclusions can be reached. Capacitance vs voltage measurements and derivatives of this method can provide much information about the electrical properties of MFS devices, including band structure and interface quality. The band structure can also be investigated with the use of carriers generated by impact ionization of alpha particles. This technique also has the potential to reveal more information about conduction processes in single crystal LiNbO$_3$, especially as it pertains to the bulk photovoltaic effect.

To investigate the switching nature of these films more closely it is necessary to apply a pulse train to the samples and monitor the resulting charge flow. The pulse train would consist of two pulses of one polarity followed by two pulses of the opposite polarity. The width between pulses, the duration of the of the pulse, and the magnitude would all be separately controllable. Then, if a switching event is initiated in the first pulse, the second pulse in the same direction would insure that it is completely switched. The following two pulses in the opposite direction switch the polarization back to the original direction. With this method it is possible to very accurately determine the coercive field, switching speeds, and total charge flow.

There are many other issues that need to be addressed in order to completely understand the switching process in LiNbO$_3$ and its potential for use in device appli-
cations. Some of these are outlined below.

- Measure $P_z$ vs film thickness.
- Try switching films deposited on metal substrates.
- Determine the effect of crystallinity on switching.
- Look at aging and retention in switchable films.
- Try switching other ferroelectrics such as LiTaO$_3$.
- Explore the possibility of a device utilizing both the switching and the photovoltages.
- Determine the effect of dopants (Fe) on the switching process.
APPENDIX A

Physical Constants of Lithium Niobate

General Properties

1. Crystal Structure [60]: Rhombohedral, \( C_3^{\overline{5}} \), (R3c) at 23° C
2. Lattice Constants [60]:
   \( a_H = 5.14829 \pm 2 \text{ Å} \)
   \( c_H = 13.8631 \pm 2 \text{ Å} \)
3. Mass Density [3]: 4.64 gm/cm\(^3\) at 25° C
4. Melting Point [60]: 1260 ± 10° C
5. Coefficients of Thermal Expansion [3]:
   (a) Linear: (i) along a-axis: 16.7 x 10\(^{-6}\)/°C from 20 - 800° C
      (ii) along c-axis: 2 x 10\(^{-6}\)/°C from 20 - 600° C
   (b) Volume: 36.5 x 10\(^{-6}\)/°C from 24 - 1000° C

Polar Properties

6. Curie Temperature [3]: 1210 ± 10° C
7. Spontaneous Polarization [3]: 71\(\mu\)C/cm\(^2\)
8. Ferroelectric Transition [26]: 2nd Order
9. Coercive Field [9]: ~ 300 kV/cm
10. Pyroelectric Constant [61]: \( \lambda = -40 \times 10^{-6} \text{ C/m}^2\text{° C} \)
**Electrical Properties**

11. Dielectric Constants [3]:
   - parallel to c axis: 32
   - perpendicular to c axis: 78

12. Resistivity [16]:
   - $\sim 10^{18}$ $\Omega$-cm

13. Inverse Susceptibility [3]:
   - $\frac{1}{\chi} \quad \text{linear from 300 - 950° C}$
   - Curie-Weiss intercept $- 1080°$ C

14. Mobility [18]:
   - $15 \text{ cm}^2/\text{V-s}$ (extrapolated from 1000° C)

15. Trapping Time [62]:
   - $4 \times 10^{-11}$ sec

16. Band Gap [21]:
   - 3.72 eV ($\sim 350$ nm)

**Optical Properties**

17. Transparency [63]
   - Transparent from 350 nm to 5000 nm

18. Refractive Index [64]:
   - 2.31657, ordinary @ 546 nm, $20°$ C
   - 2.22816, extraordinary @ 546 nm, $20°$ C

19. Electro-Optic Coefficients [65]:
   - $r_{33} = 30.8 \times 10^{-10}$ cm/V
   - $r_{13} = 8.6 \times 10^{-10}$ cm/V
   - $r_{22} = 3.4 \times 10^{-10}$ cm/V
   - $r_{51} = 28 \times 10^{-10}$ cm/V
APPENDIX B

TIPC Source Code and Photocurrent Measurement Techniques

These programs were written to control the HP-IB, IEEE-488 bus with the Texas Instruments personal computer (TIPC). These are revisions of programs originally written by Dr. Bill Wilson.

Capacitance/Conductance vs Voltage

100 REM *************** SET-UP OF BASIC FOR IEEE-488 BOARD ***************
110 CLEAR ,53152!
120 IBINIT = 53152!
130 BLOAD "bib.m",IBINIT
140 CALL IBINIT (IBRD%,IBWR&T%,IBCMD%,IBWAIT%,IBRPP%,IBONL%,IBRSC%,IBSIC%
,IBSRE%,IBRLT%,IBRSV%,IBLPE%,IBPAD%,IBSAD%,IBIST%,IBDMA%,IBEOS%,IBTMO%
,IBEOT%,IBGTS%,IBCAC%,IBIAG%,IBSTA%,IBERR%,IBCNT%)
150 BD%=0                        'BOARD 0 IS HP-IB
160 CALL IBISC%(BD%)            ' SET PC AS BUS CONTROLLER
170 IF IBSTA% < 0 THEN STOP     ' IF ERROR, THEN STOP
180 V%=1
190 CALL IBSCI%(BD%,V%)        ' REMOTE ENABLE
200 REM **************** END OF BASIC SET-UP FUNCTION ******************
210 REM
220 REM ************** BASIC PROGRAM CONTROL LOOP ***********************
230 DIM C(250),G(250),V(250)
240 CLS
250 GOSUB 410 'INITIALIZE DATA TAKING, INPUT HP4140 PARAMETERS
260 GOSUB 500 'ACQUIRE THE FIRST SET OF DATA
270 GOSUB 1190 'PLOT THE DATA ON THE 7470
280 INPUT "WOULD YOU LIKE TO REPLOT? (Y/N)",{QS
290 IF QS="N" OR QS="n" THEN GOTO 332
300 INPUT "CMAX=",CMAX
310 INPUT "CMIN=",CMIN
320 INPUT "CSTEP=",CSTEP
330 GOSUB 1280

95
332 INPUT "WOULD YOU LIKE TO PLOT G VS V? (Y/N)";QS  
335 IF QS="Y" OR QS="y" THEN GOSUB 2000  
340 INPUT "WOULD YOU LIKE TO PRINT OUT THE DATA (Y/N)";QS  
350 IF QS="Y" OR QS="y" THEN GOSUB 820  
360 INPUT "HOW ABOUT ANOTHER RUN? (Y/N)";QS  
370 IF QS="Y" OR QS="y" THEN GOTO 240  
380 CLS  
390 END  
400 REM ******************* END OF CONTROL LOOP *******************  
410 REM  
420 REM *********************** INITIALIZE DATA TAKING **********************  
422 LOCATE 5,5:PRINT"Please enter the following:"  
424 LOCATE 7,8:PRINT"Date: ";DS:LOCATE 7,13:INPUT " ,DS  
426 LOCATE 8,8:PRINT"Wafer: ";WS:LOCATE 8,14:INPUT ";WS  
428 LOCATE 9,8:PRINT"Probe Spot: ";SS:LOCATE 9,19:INPUT ";SS  
430 LOCATE 10,8:PRINT"Conditions: ";CNS:LOCATE 10,20:INPUT ";CNS  
440 LOCATE 12,8:PRINT"Start Voltage: ";SV:LOCATE 12,23:INPUT ";SV  
445 LOCATE 13,8:PRINT"Stop Voltage: ";NV:LOCATE 13,22:INPUT ";NV  
450 LOCATE 14,8:PRINT"Step Voltage: ";DV:LOCATE 14,22:INPUT ";DV  
455 LOCATE 15,8:PRINT"Hold Time: ";HT:LOCATE 15,19:INPUT ";HT  
460 LOCATE 16,8:PRINT"Step Time: ";DT:LOCATE 16,19:INPUT ";DT  
470 RETURN  
480 REM **************** END OF INITIALIZATION ***********************  
490 REM  
500 REM ****************** 4140B I-V DATA ACQUIRING SUBROUTINE ******************  
510 CMD$="1"+CHR$(&H4)  
520 CALL IBCMD%(BD%,CMD$)  
530 CMD$="@1"  
540 CALL IBCMD%(BD%,CMD$)  
550 WRT$="FN1FLIB3"  
560 CALL IBWRT%(BD%,WRT$)  
570 WRT$="PS"+STR$(SV)+";FF"+STR$(NV)+";PE"+STR$(DV)+";PL"+STR$(HT)+";PD"+STR$(DT)+";"  
580 CALL IBWRT%(BD%,WRT$)  
590 WRT$="SW1":CALL IBWRT%(BD%,WRT$)  
600 CMMS="? Q"  
610 CALL IBTMO%(BD%,HT)  
615 FOR I=1 TO NS  
620 NS=2*(((NV-SV)/(DV))+1)  
630 CMAX=0:CMIN=30000  
640 GMAX=0:GMIN=30000  
643 CALL IBTMO%(BD%,HT)  
650 FOR I=1 TO NS  
670 CALL IBCMD%(BD%,CMMS)  
675 CALL IBTMO%(BD%,ST)  
680 CALL IBRD%(BD%,AS)  
690 "READ CURRENT & VOLTAGE IN AS"
690 C(I)=VAL(MIDS(AS,4,15))
695 G(I)=VAL(MIDS(AS,20,29))
697 V(I)=VAL(MIDS(AS,32,36))
700 C(I)=C(I)*1E+12
705 G(I)=G(I)*1000000!
710 IF C(I)>CMAX THEN CMAX=C(I)
720 IF C(I)<CMIN AND C(I)>0 THEN CMIN=C(I)
730 IF G(I)>GMAX THEN GMAX=G(I)
740 IF G(I)<GMIN THEN GMIN=G(I)
760 PRINT USING "WITH ###.## VOLTS, C = ###.## pF , G = ###.## uS";V(I),C(I),G(I)
770 NEXT I
780 CALL IBCMD%(BD%,CMS)
790 CMS="SW0";CALL IBWRT%(BD%,CMS)
795 RETURN
800 REM *************** END OF DATA ACQUIRING *******************************
810 REM
820 REM *************** PRINT DATA ON SCREEN AND LINE PRINTER ***************
830 CLS
840 PRINT
845 LPRINT:LPRINT
850 LPRINT:LPRINT
860 IF SV=1 THEN VOLTS=" Volt" ELSE VOLTS=" Volts"
870 IF NV=1 THEN VOLTS=" Volt" ELSE VOLTS=" Volts"
880 IF DV=1 THEN VOLTS=" Volt" ELSE VOLTS=" Volts"
890 LPRINT TAB(20)"DATE: ";DS
900 LPRINT TAB(20)"WAFER: ";WS
910 LPRINT TAB(20)"PROBE SPOT: ";SS
920 LPRINT TAB(20)"CONDITIONS: ";CNS
930 LPRINT:LPRINT
950 PRINT " Voltage Capacitance (pF) Conductance (uS)"
955 LPRINT TAB(20)" Voltage Capacitance (pF) Conductance (uS)"
960 PRINT " ----- -------- --------" 
965 LPRINT TAB(20)" ----- -------- --------"
970 PRINT
975 LPRINT
976 FOR K=1 TO NS
980 PRINT USING " ###.## ";V(K),C(K),G(K)
985 LPRINT TAB(20) USING " ###.## ";V(K),C(K),G(K)
990 NEXT K
1000 PRINT
1005 LPRINT
1070 RETURN
1080 REM*************** END OF PRINTING SUBROUTINE ***************
1090 REM
1190 REM*************** 7470 PLOTTING SUBROUTINE FOR T.I.PC ***************
1200 REM AN AUTO-SCALING PLOTTING SUBROUTINE
1210 VMIN=SV; VMAX=NV
1230 IF CMAX>=30000 THEN CMAX=30000
1240 CMAX=INT(CMAX)+1
1250 CMIN=INT(CMIN)
1260 CSPAN=CMAX-CMIN
1270 POWER=INT(LOG(CSPAN)/LOG(10))
1272 CSTEP=10^POWER
1273 ENDMAX=1:ENDMIN=1
1274 WHILE ENDMAX <= 0
1275 ENDMAX=CMAX MOD CSTEP; CMAX=CMAX+1:PRINT CMAX; WEND
1276 WHILE ENDMIN <= 0
1277 ENDMIN=CMIN MOD CSTEP; CMIN=CMIN+1:PRINT CMIN; WEND
1278 CMAX=CMAX-1:CMIN=CMIN+1
1280 CMD$=";%"+CHR$(&H4)  "RESET THE 7470; "%;" IS LISTEN ADDRESS
1290 CALL IBWRT$(BD$,CMDS)
1300 CMLS="@%"  "SET LISTEN ADDRESS (SEE 7470 MANUAL P 16)
1310 CMTS=";E"  "TALK ADDRESS IS "E"
1320 CALL IBCMD$(BD$,CMLS) "GET THE 7470'S ATTENTION
1330 CR$=CHR$(3)  'DEFINE ETX AS TERMINATOR
1340 CMS=";IN;SP2"  'INITIALIZE AND SELECT PEN 2
1350 GOSUB 1930
1360 VMINS=STRS(VMIN); VMAXS=STRS(VMAX); CMIN$=STRS(CMIN); CMAXS=STRS(CMAX)
1370 CMS=";IP1700,1500,6700,6500;"  'DEFINE BOUNDARIES
1380 GOSUB 1930
1390 CMS=";SC"+VMINS+","+VMAXS+","+CMINS+","+CMAXS
1400 GOSUB 1930
1410 CMS=";FU"+VMINS+","+CMINS+","
1420 GOSUB 1930
1430 CMS=";PD"+VMAXS+","+CMINS+","+VMAXS+","+CMAXS+","+CMINS+","+CMAXS+","
1440 GOSUB 1930
1450 CMS=VMINS+","+CMINS+"FU;SP1"
1460 GOSUB 1930
1500 VSTEP=1  'CAN CHANGE THIS FOR LARGER SWEEPS
1510 FOR X=VMIN TO VMAX STEP VSTEP  'LABEL X AXIS
1520 CMS=";PA"+STRS(X)+","+CMINS+","+XT";GOSUB 1930
1530 CMS=";CP"+STRS(X)+","+CR$;GOSUB 1930
1540 NEXT X
1550 FOR Y=CMIN TO CMAX STEP CSTEP  'LABEL Y AXIS
1560 CMS=";PA"+VMINS+","+STRS(Y)+","+YT";GOSUB 1930
1570 IF Y=0 THEN PAD=4 ELSE PAD=INT(4+LOG(ABS(Y)/LOG(10))
1580 CMS=";CP"+STRS(PAD)+","+25;LB"+STRS(Y)+CR$;GOSUB 1930
1590 NEXT Y
1596 VS=STRS(V1); CS=STRS(C1)
1597 IF V1=9.000001E-02 THEN VS=".09"; IF V1=8.999999E-03 THEN VS=".009"
1598 IF C(1)=9.000000E-02 THEN CS=".09";IF C(1)=8.999999E-03 THEN CS=".009"
1600 CMS="PU;PA"+VS"+";+CS"+";PD";GOSUB 1930
1610 FOR K=2 TO NS
    "PLOT THE DATA"
1620 IF C(K)>30000 THEN C(K)=C_MAX
1630 IF C(K)<-30000 THEN C(K)=C_MIN
1634 VS=STRS(V(K));CS=STRS(C(K))
1635 IF V(K)=9.0000001E-02 THEN VS=".09";IF V(K)=8.999999E-03 THEN VS=".009"
1636 IF C(K)=9.0000001E-02 THEN CS=".09";IF C(K)=8.999999E-03 THEN CS=".009"
1640 CMS="PA"+VS"+";+CS;GOSUB 1930
1650 NEXT K
1660 CMS="SC;SI.14,23;SP1;PU;PA -70,3950;LB CAPACITANCE (pF)"+CRS
1670 GOSUB 1930
1680 CMS="PU;PA 3500,800;LB VOLTAGE (Volts)"+CRS
1690 GOSUB 1930
1700 CMS="PU;PA 2900,7000;SI .25,5;LB 1 MHz C vs. V"+CRS
1710 GOSUB 1930
1720 CMS="PU;PA 2910,7000;SI .25,5;LB 1 MHz C vs. V"+CRS
1730 GOSUB 1930
1740 CMS="PU;PA 7000,6400;SI;LB Date:"+DS+CRS
1750 GOSUB 1930
1760 CMS="PU;PA 7000,6100;LB Wafer ID:"+WS+CRS
1770 GOSUB 1930
1780 CMS="PU;PA 7000,5800;LB Probe Spot:"+SS+CRS
1790 GOSUB 1930
1800 IF HT=1 THEN SECONDS=" second" ELSE SECONDS=" seconds"
1810 CMS="PU;PA 7000,5200;LB Hold Time:"+STRS(H)+SECONDS+CRS
1820 GOSUB 1930
1830 IF DT=1 THEN SECONDS=" second" ELSE SECONDS=" seconds"
1840 CMS="PU;PA 7000,4900;LB Step Time:"+STRS(D)+SECONDS+CRS
1850 GOSUB 1930
1860 IF DV=1 THEN VOLTS=" volt" ELSE VOLTS=" volts"
1870 CMS="PU;PA 7000,4600;LB Step Voltage:"+STRS(D)+VOLTS+CRS
1880 GOSUB 1930
1890 CMS="PU;PA 7000,4000;LB Conditions:"+CNS+CRS
1900 GOSUB 1930
1910 CMS="SP1";GOSUB 1930
1920 RETURN
1930 CALL IBWRT%(BD%,CMS)
1940 RETURN
2000 REM*************** 7470 PLOTTING SUBROUTINE FOR T.IPC ***************
2005 REM AN AUTO-SCALING PLOTTING SUBROUTINE
2010 IF GMAX>30000 THEN GMAX=30000
2020 GMAX=INT(GMAX)+1
2030 GMIN=INT(GMIN)
2040 GSPAN=GMAX-GMIN
2050 POWER=INT(LOG(GSPAN)/LOG(10))
2060 GSTEP=10^POWER
2070 ENDMAX=1:ENDMIN=1
2080 WHILE ENDMAX <> 0
2090 ENDMAX=GMAX MOD GSTEP:GMAX=GMAX+1:WEND
2100 WHILE ENDMIN <> 0
2110 ENDMIN=GMIN MOD GSTEP:GMIN=GMIN+1:WEND
2120 GMAX=GMAX-1:GMIN=GMIN+1
2130 CMS$="%"+CHR$(&H4) "RESET THE 7470; "%" IS LISTEN ADDRESS
2140 CALL IBWRT%(BD%,CMS$)
2150 CMS$="@c%" "SET LISTEN ADDRESS (SEE 7470 MANUAL P 16)
2160 CMS$="?E" "TALK ADDRESS IS "E"
2170 CALL IBCM%(BD%,CMS$) "GET THE 7470'S ATTENTION
2180 CR$=CHR$(3) "DEFINE ETX AS TERMINATOR
2190 CMS$="IN;SP2" "INITIALIZE AND SELECT PEN 2
2200 GOSUB 1930
2210 VMINS=STRS(VMIN):VMAXS=STRS(VMAX):GMINS=STRS(GMIN):GMAXS=STRS(GMAX)
2220 CMS$="TP1700,1500,6700,6500;" "DEFINE BOUNDARIES
2230 GOSUB 1930
2240 CMS$="SC"+VMINS+","+VMAXS+","+GMINS+","+GMAXS
2250 GOSUB 1930
2260 CMS$="PU"+VMINS+","+GMINS+","+
2270 GOSUB 1930
2280 CMS$="PD"+VMAXS+","+GMINS+","+VMAXS+","+GMAXS+","+VMINS+","+GAXM+","+
2290 GOSUB 1930
2300 CMS$=VMINS+","+GMINS+"PU;SP1"
2310 GOSUB 1930
2320 FOR X=VMIN TO VMAX STEP VSTEP "LABEL X AXIS
2330 CMS$=" PA"+STRS(X)+","+GMINS+";XT";GOSUB 1930
2340 CMS$="CP-1,5,-1;LB"+STRS(X)+CR$;GOSUB 1930
2350 NEXT X
2360 FOR Y=GMIN TO GMAX STEP GSTEP "LABEL Y AXIS
2370 CMS$=" PA"+VMINS+","+STRS(Y)+";YT";GOSUB 1930
2380 IF Y=0 THEN PADIO=4 ELSE PADIO=INT(4+LOG(ABS(Y))/LOG(10))
2390 CMS$="CP"+STRS(PADIO)+"-.25;LB"+STRS(Y)+CR$;GOSUB 1930
2400 NEXT Y
2410 CMS$="PA"+VMAXS+";+STRS(V(1))+";+STRS(G(1))+";PD";GOSUB 1930
2420 FOR K=2 TO NS "PLOT THE DATA
2430 IF G(K)> 30000! THEN G(K)=GMAX
2440 IF G(K)< -30000! THEN G(K)=GMIN
2450 CMS$="PA"+STRS(V(K))+";+STRS(G(K));GOSUB 1930
2460 NEXT K
2470 CMS$="SC;SI .14,.23;SP1;PA -70,3950;LB CONDUCTANCE (us) "+CRS
2480 GOSUB 1930
2490 CMS$="PA;PA 3500,800;LB VOLTAGE (Volts) "+CRS
2500 GOSUB 1930
2510 CMS="PU:PA 2900,7000;SI .25,.5;LB 1 MHz. G vs. V "+CRS
2520 GOSUB 1930
2530 CMS="PU:PA 2910,7000;SI .25,.5;LB 1 MHz. G vs. V "+CRS
2540 GOSUB 1930
2550 CMS="PU:PA 7000,6400;SI;LB Date: "+DS+CRS
2560 GOSUB 1930
2570 CMS="PU:PA 7000,6100;LB Wafer ID: "+WS+CRS
2580 GOSUB 1930
2590 CMS="PU:PA 7000,5800;LB Probe Spot: "+SS+CRS
2600 GOSUB 1930
2610 IF HT=1 THEN SECONDS=" second" ELSE SECONDS=" seconds"
2620 CMS="PU:PA 7000,5200;LB Hold Time: "+STRS(HT)+SECONDS+CRS
2630 GOSUB 1930
2640 IF DT=1 THEN SECONDS=" second" ELSE SECONDS=" seconds"
2650 CMS="PU:PA 7000,4900;LB Step Time:"+STRS(DT)+SECONDS+CRS
2660 GOSUB 1930
2670 IF DV=1 THEN VOLTS=" volt" ELSE VOLTS=" volts"
2680 CMS="PU:PA 7000,4600;LB Step Voltage:"+STRS(DV)+VOLTS+CRS
2690 GOSUB 1930
2700 CMS="PU:PA 7000,4000;LB Conditions:"+CRS+CNS+CRS
2710 GOSUB 1930
2720 CMS="SP0":GOSUB 1930
2730 RETURN

Current vs Voltage

100 REM ***************** SET-UP OF BASIC FOR IEEE-488 BOARD *****************
110 CLEAR ,S3152!
120 IBINIT = .53152!
130 BLOAD "bib.m",IBINIT
140 CALL IBINIT (IBRD%,IBWRT%,IBCMD%,IBWAIT%,IBRPP%,IBONL%,IBRSC%,IBSIC% ,IBSER%,IBRTL%,IBRSV%,IBPTE%,IBPAD%,IBSAD%,IBIST%,IBDMA%,IBEOS%,IBTMO% ,IBEOT%,IBGTS%,IBCAC%,IBDIAQ%,IBSTA%,IBERR%,IBCNT%)
150 BD%=0           ' BOARD 0 IS HP-JB
160 CALL IBASIC%(BD%)  ' SET PC AS BUSS CONTROLLER
170 IF IBSTA% < 0 THEN STOP     ' IF ERROR, THEN STOP
180 V%=1
190 CALL IBSER%(BD%,V%)  ' REMOTE ENABLE
200 REM ***************** END OF BASIC SET-UP FUNCTION *********************
210 REM
220 REM ***************** BASIC PROGRAM CONTROL LOOP **********************
230 DIM C(300),V(300)
240 CLS
250 GOSUB 410 'INITIALIZE DATA TAKING, INPUT HP4140 PARAMETERS
260 GOSUB 500 'ACQUIRE THE FIRST SET OF DATA
262 GOSUB 2000
265 INPUT "Would you like to plot the data? ",QS
270 IF QS="Y" OR QS="y" THEN GOSUB 1190
280 INPUT "Would you like to replot? ",QS
290 IF QS="N" OR QS="n" THEN GOTO 340
300 INPUT "CMAX=",CMAX
310 INPUT "CMIN=",CMIN
320 INPUT "CSTEP=",CSTEP
330 GOSUB 1280
340 INPUT "Would you like to print out the data? ",QS
350 IF QS="Y" OR QS="y" THEN GOSUB 820
360 INPUT "How about another run? ",QS
365 ERASE C,V
370 IF QS="Y" OR QS="y" THEN GOTO 220
380 CLS
390 END
400 REM **************************** END OF CONTROL LOOP ****************************
410 REM
420 REM **************************** INITIALIZE DATA TAKING ****************************
422 LOCATE 5,5:PRINT"Please enter the following:"
424 LOCATE 7,8:PRINT"Date: ",DS:LOCATE 7,13:INPUT ",DS
426 LOCATE 8,8:PRINT"Wafer: ",WS:LOCATE 8,14:INPUT ",WS
428 LOCATE 9,8:PRINT"Probe Spot: ",SS:LOCATE 9,19:INPUT ",SS
430 LOCATE 10,8:PRINT"Conditions: ",CN:LOCATE 10,20:INPUT ",CN
432 LOCATE 12,8:PRINT"Start Voltage: ",SV:LOCATE 12,23:INPUT ",SV
436 LOCATE 14,8:PRINT"Step Voltage: ",DV:LOCATE 14,22:INPUT ",DV
438 LOCATE 15,8:PRINT"Hold Time: ",HT:LOCATE 15,19:INPUT ",HT
470 RETURN
480 REM **************************** END OF INITIALIZATION ****************************
490 REM
500 REM *********************** 4140B I-V DATA ACQUIRING SUBROUTINE *********************
510 CMDS="("+CHR$(6)+CHR$(4)+CHR$(1))" 'RESET THE 4140A
520 CALL IBCM%(BD%,CMDS)
530 CMDS="@" 'GET THE 4140'S ATTENTION
540 CALL IBCM%(BD%,CMDS)
550 WRTS="F1RAI131L1M1" 'SET 4140 PARAMETERS
560 CALL IBWRT%(BD%,WRTS)
570 WRTS="PS"+STR$(SV)+";PT"+STR$(NV)+";PE"+STR$(DV)+";"
580 CALL IBWRT%(BD%,WRTS) 'START,STOP,STEP SET
590 WRTS="W2":CALL IBWRT%(BD%,WRTS) 'PUT 4140 IN MANUAL MODE
600 CMMS="? H" 'GET 4140 TO TALK
610 AS=SPACES(21) "DATA HAS 21 BYTES IN IT"
620 NS=ABS(NV-SV)/DV+1
630 MINUTES=HT:GOSUB 1100
635 PRINT:PRINT
640 CMAX=0;CMIN=0
650 FOR I=1 TO NS
660 MINUTES=DT:GOSUB 1100
670 CALL IBCMD%(BD%,CMMS)
680 CALL IBRD%(BD%,AS) "READ CURRENT & VOLTAGE IN AS"
690 C(I)=VAL(MIDS(AS,4,10))
700 C(I)=C(I)*1E+12
710 IF C(I)>CMAX THEN CMAX=C(I)
720 IF C(I)<CMIN THEN CMIN=C(I)
730 V(I)=VAL(MIDS(AS,16,22))
740 CALL IBCMD%(BD%,CMDMS) "GET IT TO LISTEN AGAIN"
745 IF SV<NV THEN WRTS="W6" ELSE WRTS="W5"
750 CALL IBWRT%(BD%,WRTS) "VOLTAGE STEP"
760 PRINT USING "WITH ##.## VOLTS, THE CURRENT = ####.##### pA";V(I),C(I)
770 NEXT I
780 WRTS="W7";CALL IBWRT%(BD%,WRTS) "RESET THE VOLTAGE"
790 RETURN
800 REM *************** END OF DATA ACQUIRING ********************
810 REM
820 REM *************** PRINT DATA ON SCREEN AND LINE PRINTER ********
830 CLS
840 PRINT
850 LPRINT ""
860 LPRINT ""
870 IF SV=1 THEN VOLTS=" Volt" ELSE VOLTS=" Volts"
880 IF NV=1 THEN VOLTS=" Volt" ELSE VOLTS=" Volts"
890 IF DV=1 THEN VOLTS=" Volt" ELSE VOLTS=" Volts"
900 LPRINT TAB(20)"DATE: ";D$;TAB(55)"START V = ";STRS(SV)+VOLTS
910 LPRINT TAB(20)"WAFER: ";WS;TAB(55)"STOP V = ";STRS(NV)+VOLTS
920 LPRINT TAB(20)"PROBE SPOT: ";SS;TAB(55)"STEP V = ";STRS(DV)+VOLTS
930 LPRINT TAB(20)"CONDITIONS: ";;CNS;TAB(55)"HOLD TIME = "STRS(HT)
940 LPRINT TAB(55)"STEP TIME = ";;DT
950 LPRINT ""
960 LPRINT ""
970 PRINT " ";VA"," CURRENT "
980 LPRINT " ";VA"," CURRENT "
990 LPRINT " -----
1000 LPRINT
1010 FOR I=1 TO NS
1020 PRINT USING " ";V(I),C(I)
1030 LPRINT USING " ";V(I),C(I)
1040 NEXT I
1050 PRINT
1060 PRINT
1070 RETURN
1080 REM ************** END OF PRINTING SUBROUTINE **************
1090 REM
1100 REM ********** TIMED DELAY SUBROUTINE **********
1110 FOR K=1 TO MINUTES
1120 FOR J=1 TO 12425!
1130 T=12345/2335666!
1140 NEXT J
1150 NEXT K
1160 RETURN
1170 REM ************** END OF TIMING SUBROUTINE **************
1180 REM
1190 REM*************** 7470 PLOTTING SUBROUTINE FOR T.I.PC ***************
1200 REM AN AUTO-SCALING PLOTTING SUBROUTINE
1210 IF SV>NV THEN VMIN=NV ELSE VMIN=SV
1216 IF SV>NV THEN VMAX=SV ELSE VMAX=NV
1220 IF ABS(CMIN)>CMAX THEN CMAX=ABS(CMIN)
1230 IF CMAX=30000 THEN CMAX=30000
1240 POWER=INT(LOG(CMAX)/LOG(10))
1250 MANTISSA=INT(CMAX/10^POWER)+1
1260 CSTEP=10^POWER
1270 CMAX=MANTISSA*10^POWER*CMIN=CMAX
1280 CMD$="%;CHR$(&H4)" 'RESET THE 7470; "%; IS LISTEN ADDRESS
1290 CALL IBWRT%(BD%,CMDS)
1300 CM$L="@%;" 'SET LISTEN ADDRESS (SEE 7470 MANUAL P 16)
1310 CMTS="%;" 'TALK ADDRESS IS "E"
1320 CALL IBCMD%(BD%,CM$L) 'GET THE 7470'S ATTENTION
1330 CRS=CHR$(3) 'DEFINE ETX AS TERMINATOR
1340 CMS="IN;SP2" 'INITIALIZE AND SELECT PEN 2
1350 GOSUB 1930
1360 VMINS=STR$ (VMIN);VMAXS=STR$ (VMAX);CMINS=STR$ (CMIN);CMAXS=STR$ (CMAX)
1370 CMS="IP1500,1500,6500,6500" 'DEFINE BOUNDARIES
1380 GOSUB 1930
1390 CMS="SC"+VMINS+","+VMAXS+","+CMINS+","+CMAXS
1400 GOSUB 1930
1410 CMS="PU"+VMINS+","+CMINS+","+
1420 GOSUB 1930
1430 CMS="PD"+VMAXS+","+CMINS+","+VMAXS+","+CMAXS+","+VMINS+","+CMAXS+","+
1440 GOSUB 1930
1450 CMS=VMINS+","+CMINS="PU"
1460 GOSUB 1930
1470 CMS="PU 0,"+CMINS+";PD 0,"+CMAXS
1480 GOSUB 1930
1490 CMS="PU"+VMINS",";PD"+VMAXS",";PU;SP1"
1500 GOSUB 1930
1510 FOR X=VMIN TO VMAX 'LABEL X AXIS
1520 CMS="PA"+STRS(X)+";"+CMINS",";XT":GOSUB 1930
1530 CMS="CP;1.5,-1;LB"+STRS(Y)+CRS:GOSUB 1930
1540 NEXT X
1550 FOR Y=CMIN TO CMAX STEP CSTEP 'LABEL Y AXIS
1560 CMS="PA"+VMINS",";+STRS(Y)+";YT":GOSUB 1930
1570 IF Y=0 THEN PAD=-4 ELSE PAD=INT(4+LOG(ABS(Y))/LOG(10))
1580 CMS="CP"+STRS(PAD)+";-.25;LB"+STRS(Y)+CRS:GOSUB 1930
1590 NEXT Y
1595 IF V(1)<CMIN THEN V(1)=CMIN
1600 VS=STRS(V(1));CS=STRS(C(1))
1597 IF V(1)=0 THEN VS=" .0";IF V(1)=8.999999E-03 THEN VS=" .009"
1598 IF C(1)=0 THEN CS=" .0";IF C(1)=8.999999E-03 THEN CS=" .009"
1600 CMS="PU"+VS",";+CS",";PD":GOSUB 1930
1610 FOR K=2 TO NS 'PLOT THE DATA
1620 IF C(K)>CMAX THEN C(K)=CMAX
1630 IF C(K)<CMIN THEN C(K)=CMIN
1634 VS=STRS(V(K));CS=STRS(C(K))
1635 IF V(K)=0 THEN VS=" .0";IF V(K)=8.999999E-03 THEN VS=" .009"
1636 IF C(K)=0 THEN CS=" .0";IF C(K)=8.999999E-03 THEN CS=" .009"
1640 CMS="PA"+VS",";+CS":GOSUB 1930
1650 NEXT K
1660 CMS="SC;SI:14,23;SP1;PU;PA 75,3950;LB CURRENT (pA) "+CRS
1670 GOSUB 1930
1680 CMS="PU;PA 3300,800;LB VOLTAGE (Volts) "+CRS
1690 GOSUB 1930
1700 CMS="PU;PA 2400,7000;SI:25,5;LB Current vs. Voltage "+CRS
1710 GOSUB 1930
1720 CMS="PU;PA 2410,7000;SI:25,5;LB Current vs. Voltage "+CRS
1730 GOSUB 1930
1740 CMS="PU;PA 7000,6400;SI;LB Date: "+DS+CRS
1750 GOSUB 1930
1760 CMS="PU;PA 7000,6100;LB Wafer ID: "+WS+CRS
1770 GOSUB 1930
1780 CMS="PU;PA 7000,5800;LB Probe Spot: "+SS+CRS
1790 GOSUB 1930
1800 IF HT=1 THEN MINUTS=" minute" ELSE MINUTS=" minutes"
1810 CMS="PU;PA 7000,5200;LB Hold Time: "+STRS(HT)+MINUTS+CRS
1820 GOSUB 1930
1830 IF DT=1 THEN MINUTS=" minute" ELSE MINUTS=" minutes"
1840 CMS="PU;PA 7000,4900;LB Step Time: "+STRS(DT)+MINUTS+CRS
1850 GOSUB 1930
1860 IF DV=1 THEN VOLTS=" volt" ELSE VOLTS=" volts"
1870 CMS="PU:PA 7000,4600;LB Step Voltage: "+STRS(DV)+VOLTS+CRS
1880 GOSUB 1930
1890 CMS="PU:PA 7000,4900;LB Conditions: "+CRS
1900 GOSUB 1930
1910 CMS="SP0":GOSUB 1930
1920 RETURN
1930 CALL IBWRT%(BD%,CMS)
1940 RETURN
2000 REM ***** SCREEN PLOTTING SUBROUTINE FOR TI PC *****
2005 RETURN
2010 CLS
2050 LINE (150,50)-(600,250),6,B
2060 Y1=250-(C(1)*200/(CMAX-CMIN))
2070 LINE (150,Y1)-(150,Y1)
2080 FOR I=1 TO NS
2090 Y=C(I)*200
2100 Y=250-Y
2110 X=150+((I-1)*450/NS)
2120 LINE -(X,Y),5
2130 NEXT I
2200 RETURN
Photocurrent Measurements

Since the electrometer is measuring a current and converting it to a voltage for input to the HP4145A, the 4145A is programmed to convert back to a current for the display. This is done with the help of the menu screens on the 4145A. The method for obtaining plots is outlined below.

1. When the 4145A is turned on the screen will display a main menu.

2. By pressing the NEXT key the first menu will appear. This is CHANNEL DEFINITION screen. This screen basically tells the 4145A which connections are to be used. Use the cursor keys to move the cursor to different entries. Delete all entries except for the V1 and VM1 entries. This is done by moving the cursor to the entry to be deleted and pressing the softkey labelled NOT USE. At the bottom of the screen is an area to create a user function. Type:

   IP enter   - function name
   pA enter   - function units
   VM1*115    - the function

Use the blue key to select the alternate letter by each key.

3. Hit the NEXT key to advance to the SOURCE SET UP screen. Since no sources are needed for this measurement hit the NEXT key again to get to the MEASUREMENT & DISPLAY MODE SET UP screen.
4. This screen is devoted to determining what the parameters of the plot will be.

For a typical photocurrent measurement this is what is typed in:

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>select interval</td>
<td>.02</td>
</tr>
<tr>
<td>number of readings</td>
<td>500</td>
</tr>
<tr>
<td>Display mode</td>
<td>GRAPHICS</td>
</tr>
<tr>
<td>X axis</td>
<td></td>
</tr>
<tr>
<td>minimum</td>
<td>0</td>
</tr>
<tr>
<td>maximum</td>
<td>10</td>
</tr>
<tr>
<td>Y axis</td>
<td></td>
</tr>
<tr>
<td>EXTN (softkey)</td>
<td>IP (softkey)</td>
</tr>
<tr>
<td>scale</td>
<td>LINEAR (softkey)</td>
</tr>
<tr>
<td>minimum</td>
<td>-30</td>
</tr>
<tr>
<td>maximum</td>
<td>30</td>
</tr>
</tbody>
</table>

Now the 4145A can begin taking data. Press NEXT to display the graphics screen and SINGLE or REPEAT to start taking data and plotting it on the screen. SINGLE just takes one screen full of data and stops whereas REPEAT continues taking data and rewrites the old data. When a plot is desired simply hit PLOT and EXECUTE and the screen is dumped to the plotter. It is possible to make comments on the plot by using the COMMENT softkey.
References


