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Terahertz Testing Fixture

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Abstract

Nanowires are currently being developed for use in next-generation solar cells. In order to produce and improve these solar cells, improved nanowire synthesis methods and non-contact measurement techniques are needed. The goal of this project was to design, build, and test a fixture for measuring both axial and radial conductivity of nanowires by terahertz spectroscopy. In order to measure conductivity along both directions, an adjustable rotating fixture for holding nanowire samples was designed and manufactured. The samples prepared for testing consisted of aligned and unaligned zinc oxide nanowires grown on both quartz and silicon substrates.
1. Introduction and Motivation

This Major Qualifying Project (MQP) was created for the purpose of testing the conductivity of nanowires for future use in solar cells. Nanowires are long, thin structures that can be grown vertically from a substrate, and have an incredibly high surface area to volume ratio. By implementing them in the field of solar energy, nanowires offer many solutions to common problems, but also introduce novel issues. The primary benefit of using nanowires in solar cells is that the efficiency is improved because the nanowire geometry reduces the distance that excited electrons must travel within the semiconductor. This enables wider choice of semiconductor materials for solar cells, and could enable low-cost, non-toxic, economically-processed semiconductors to achieve high efficiency. Another benefit is increased light absorption due to decreased reflection of light. The unique geometry, material advantages, and synthesis methods of nanowires opens up a new field of research that could one day lead the way in solar power generation, as well as flexible and thin solar cells. However, in order to select the best materials and synthesis methods for growing nanowires, their electrical conductivity must be determined. The radial and axial conductivity of crystalline nanowires may differ.

Measuring nanowire conductivity is important for quantifying their usefulness in solar cells. Finding the conductivity along the vertical axis of the wires is more important for determining the usefulness of nanowires, and our goal was to enable this axial measurement. In order to accomplish this, we designed a stage for rotating samples at various angles, which will allow for both radial and axial conductivity to be measured. Our team fabricated ZnO nanowire samples, designed and prototyped a testing fixture for the samples, and measured the conductivity of the nanowires using terahertz spectroscopy, a form of non-contact measurement.
The primary focus of this project was the design of a rotating testing fixture for any samples that require terahertz measurements. The purpose of the fixture is to hold a sample in place so that a terahertz beam can be aimed at a specific portion of the sample. There were various design constraints that guided the design of the fixture, many of which related to the physical space available within the existing THz measurement apparatus, into which the stage needed to fit. Some other design constraints arose from the way in which the beam is aligned and focused, material selection, ease of manufacturing, need for stable angular position of the sample during measurement, method of holding samples in place, and desire for simplicity. The entire design process, including the various choices that were made, designs that were produced, modified, and selected, prototyping, and further work has been documented as the primary learning aspect of this project.

After developing, analyzing, and improving upon multiple preliminary designs, we manufactured a final device that consists of a rotating stage with a slot-shaped cutout for the terahertz beam. The stage rotates within a fixed frame, and the angle is controlled by a knob with angle markings. When the stage is angled, the sample is held in place by an adjustable clamp.

To effectively demonstrate the effects of angled terahertz measurements of nanowire samples, we needed to synthesize aligned and unaligned nanowires. Our group synthesized Zinc Oxide (ZnO) nanowires on quartz and silicon substrates. Various papers were reviewed on synthesis of nanowires, and successful experiments in literature were followed to the best of our ability. Although the team had little experience in this field, synthesis methods evolved over the course of the MQP and resulted in more consistent growths, as was shown in Scanning Electron Microscopy (SEM) images of each sample batch.
2. Background & Literature Review

2.1 Terahertz Spectroscopy

In terahertz spectroscopy, a terahertz (THz) pulse is used to determine properties of a material. THz radiation is in the far infrared region of the electromagnetic spectrum, between infrared radiation and microwaves, and covers a frequency range from 0.1 THz to 20 THz, where $1 \text{ THz} = 10^{12} \text{ Hz}$ (Baxter & Guglietta, 2011). There are two common methods for generating THz pulses in experimental setups (Lanzani, 2006). One way is using a photoconductive switch, known as an Auston switch. With an Auston switch, a laser pulse with a time on the order of 100 femtoseconds illuminates a substrate with two voltage-biased electrodes. A current is generated between the two electrodes, and the resultant acceleration of charge carriers produces a THz pulse out of the other side of the substrate. Photoconductivity can also be used to detect THz radiation. The THz pulse is focused in between two electrodes on a semiconductor substrate. When the semiconductor is illuminated with a laser pulse, photo carriers generate a current which will flow on the semiconductor in the direction of the THz electric field. The second method of generating THz pulses, optical rectification, is used in this project. It involves illuminating one side of a nonlinear optical crystal, such as ZnTe, with a femtosecond laser pulse. Two particular types of THz spectroscopy are of interest for this project: terahertz time-domain spectroscopy, where intrinsic properties of materials are determined, and optical pump-terahertz probe spectroscopy, which yields changes in material properties such as conductivity due to photoexcitation.
2.1.1 Terahertz Time-Domain Spectroscopy

In terahertz time-domain spectroscopy, or THz-TDS, the original laser pulse is split into two beams; one beam generates the THz pulse, while the other is used for detection of the THz pulse. The THz pulse beam passes through a series of off-axis parabolic mirrors which collimate in and then focus it onto the sample. The arrival of the THz pulse at the detector is systematically varied so that the THz electric field can be measured with respect to time. Charge carriers exhibit strong absorption in the THz range, and the pulse becomes attenuated and delayed (Lanzani, 2006). The amplitude and phase of the THz pulse probe is compared to a reference pulse that probes a bare substrate, which allows the complex-valued, frequency resolved conductivity of the sample to be obtained. Free carriers present in the semiconductor material, due to either doping or photoexcitation, exhibit strong absorption in THz range - therefore attenuating the signal. This makes THz spectroscopy an ideal tool to probe the conductivity of nanowires.

Figure 1: Schematic of THz Spectroscopy Measurement
2.1.2 Optical Pump-Terahertz Probe

Another form of terahertz spectroscopy, optical pump-terahertz probe (OPTP), is similar in principle to THz-TDS, but the sample itself is photoexcited using a ~ 100 fs optical pulse. The original laser pulse is split into three beams: one for THz pulse generation, one for THz pulse detection, and one for excitation of the sample. Because the sample is now photoexcited, properties that are direct effects of photoexcitation can be measured, such as recombination velocities and lifetimes of photoexcited charge carriers, which are on the scale of femtoseconds to nanoseconds. These measurements are useful, as carrier lifetime is particularly important in solar cells because it is desirable for excited carriers to reach the charge collector before recombining.

The sample photoexcitation induces a change $\Delta E$ in the terahertz pulse. The spectrometer
measures the transmitted electric field, calculating the conductivity of the sample by comparing E of the terahertz probe and E-ΔE, the reading from the spectrometer.

2.2 Nanowire Solar Cells

In solar cells with nanowire arrays, the charge carrier efficiency is improved because the nanowire geometry reduces the distance that excited electrons must travel within the semiconductor (Law et al., 2005). Additionally, materials commonly used in thin-film solar cells can be costly, toxic, and scarce (Hao, 2016). The primary motivation for nanowire solar cell research stems from the increased performance that nanowire geometry can bring to semiconducting materials. Nanowires would allow materials that are lower cost and in greater abundance to be used in solar cells without a reduction in efficiency (Peksu & Karaagac, 2015). Since the nanowires are used as conductors, electron mobility and concentration within the nanowires are important to achieving a high conductivity. Nanowires made with semiconducting materials, such as zinc oxide (ZnO), have electrical and optical properties that make them desirable for use in solar cells (Unalan et al, 2008). These beneficial properties derive from nanowire geometry. The large surface area of nanowires relative to their volume allows for a higher absorption of solar energy compared to bulk samples of the same material (Lee et al., 2013).

Another potential application for nanowire solar cells is photoelectrochemical water splitting (Rao et al., 2014). In photoelectrochemical water splitting, or PEC, solar energy is converted into chemical energy by the splitting of water molecules into oxygen and hydrogen (Shi et al., 2015). Conventional methods of producing hydrogen for fuel sources release large amounts of greenhouse gases into the environment. PEC is of interest because it has the potential
to supply hydrogen fuel with no greenhouse gas emissions.

2.3 Terahertz Measurement of Nanowire Solar Cells

Multiple studies have analyzed solar cells with terahertz spectroscopy. One such study used THz-TDS to find intrinsic conductivity in both thin film samples of zinc oxide and zinc oxide nanowires (Baxter & Schmuttenmaer, 2009). In this particular study, THz-TDS was used to find that the thin film samples displayed higher electron mobilities. The study also compared using THz-TDS to using Hall-effect measurements as experimental methods. It was found that Hall-effect measurements only allow low-frequency conductivities to be obtained, whereas THz-TDS allows the frequency-dependent, complex conductivity to be measured. Additionally, unlike THz-TDS, Hall-effect measurements require electrical contacts with the samples, which has the potential to affect the readings.

Another study used terahertz spectroscopy to evaluate core-shell nanowires (Zhou et al., 2016). A nanorod core, used for conducting electrons, is coated with another material which absorbs solar photons. This study examined nanowires consisting of an Sb-doped SnO₂ core covered with a thin layer of the semiconductor electrode BiVO₄. THz-TDS was used to evaluate the conductivity of the nanowires.

The samples that will be used in the rotating test fixture and probed with both THz-TDS and OPTP consist of a quartz substrate coated with nanowires on one side of the substrate. Several orientations of nanowires are possible, including randomly oriented nanowires, vertically aligned nanowires, and branched vertical nanowires. The samples vary in size (see Figure 3, below), although they are roughly rectangular with a typical length of 1 inch and width of 0.5 inch.
2.4 Test Fixtures

2.4.1 Current Laboratory Setup

In the Ultrafast Terahertz Physics laboratory at Worcester Polytechnic Institute, THz time-domain spectroscopy (THz-TDS) is used to characterize intrinsic conductivity in solar cell materials, and optical pump – THz probe spectroscopy (OPTP) is used to probe transient changes in conductivity induced by optical excitation. A single near-IR (800 nm) visible laser beam is split into multiple beams for the generation of the terahertz pulses, the detection of terahertz pulses, and in the case of OPTP, the excitation of the sample. The terahertz beam is generated using optical rectification with a ZnTe crystal. Off-axis parabolic mirrors collect and focus the generated THz beam onto the sample. The transmitted beam is then collected and focused onto a ZnTe detector crystal using another set of off-axis parabolic mirrors. Once it reaches the ZnTe detector crystal, the electric field waveform of the pulse is sampled by free-space electrooptic
sampling. The arrival time of the sampling beam relative to the arrival time of the THz pulse at ZnTe detector is controlled by the motorized translation stage.

The current fixture for holding the nanowire samples in place on top of the flat sample stage consist of flexible metallic clips attached to a rounded rectangular aluminum base. The rounded rectangular base is 6 ¾ inches long and 5 ⅛ inches wide, with a thickness of ¼ inches. The sample is placed flat over a circular hole in the base, and the clips are positioned to keep the sample in place. The entire base is lowered into another platform and secured to that platform. The horizontal and vertical positions of the platform, and therefore the aluminum base and sample, can be adjusted for precise positioning of the sample in the focal spot of the THz beam. However, there is no way to manipulate the angle of the base. Figure 4, below, shows the current fixture, along with labels of some of the components.

Figure 4: Current Sample Holder Design
2.4.2 Currently Existing Rotating Test Stages

Test fixtures that rotate samples do exist, but the many constraints applied by the laboratory setup and the samples would make it difficult to simply replace the current laboratory setup with minimal modifications, as it is desired to simply replace the current test fixture.

Figure 5: Rotating Sample Stage

Stages like the one in Figure 5, seen above, allow for the centering of an optical beam on a fixed portion of a sample. The issue in using this commercially available stage is that the optical hole is 1 inch wide, which is much too large for the THz beam, and the control dials interfere with the current laboratory setup. However, commercially available test fixture components will be investigated and may be incorporated into the design for the new rotating stage, such as a threaded dial with angle markings and spring clips to hold the sample in place during rotation.
3. Scope

3.1 Project Motivation

The result of this Major Qualifying Project was a test fixture that can repeatedly and consistently hold a sample at a desired angle for examination using terahertz spectroscopy. This MQP was necessary because it is desired to know the conductivity along the wires in the axial direction by tilting the sample. Previously, it was only possible to probe the nanowire samples in a way such that the THz pulse intercepts the substrate perpendicular to the bottom of the substrate surface, which only allows for characterization of the nanowire properties in the radial direction. Thus, some method was required to let the THz pulse intercept the substrate at an angle, allowing for more relevant electrical characterization along the length of the nanowires. Improved non-contact methods of measuring nanowire conductivity will aid in further research regarding nanowires and nanowire solar cells.

3.2 Project Goals

The ultimate goal for this project was to design, fabricate, and test a fixture that allows for the electrical characterization of nanowires along the length of the wires. Objectives in support of this goal include synthesizing aligned and unaligned nanowires as well as developing, testing, and analyzing preliminary designs for the test fixture, fabricating physical prototypes, and finally fabricating a final device that takes into account necessary improvements identified from earlier prototypes.
4. Methods

4.1 Design Considerations and Selection

In order to design a solution to the problem, it is important to consider the constraints involved. Some constraints and considerations encountered during the design process are discussed below. After identifying these several aspects that may affect our design, we selected which parameters were most important, and which ones to consider or incorporate in each design iteration.

Firstly, the fixture needs to be made of a resilient material, such as aluminum. Whichever material selected needs to be strong and rigid, so as to maintain its shape, as well as resistant to the visible laser beam. The beam is powerful enough to increase the temperature of the underside of the current fixture, which can cause damage if the wrong material is used.

There are several pieces of equipment in close proximity to the sample probing area; it is necessary that the rotating test fixture not interfere with or contact these devices. The largest is an angled parabolic mirror, used for redirecting and focusing the THz beam after the beam has exited the sample, which protrudes above and over one edge of the existing sample holder. Because portions of the fixture needs to rotate about its center, the location of nearby devices must need to be taken into consideration. Any mechanical interference could result in a smaller angular range than desired, as well as potentially damaging the fixture, nearby devices, or the sample. Installation of the test fixture should be accomplished with minimal modification to the existing laboratory setup, other than the removal of the current sample holder. Ideally, the new test fixture will occupy the same space as the current sample holder and utilize the same method.
for attachment to the xyz-stage platform.

In addition to the rotational functionality of the device, it was also necessary to devise a method of fixing in place the sample being studied. Because the samples are fragile, such a method needs to be sturdy without providing too much pressure, so as not to damage the sample. It also needs to be easily adjustable, and most importantly out of the way of the THz beam.

The manufacturability and ease of assembly of the device must also be considered. Components of the test fixture will need to be manufactured and assembled in-house, as opposed to purchased, so it is desirable to utilize components that can be manufactured and assembled using the available resources. Any parts that need to be sourced should be easily available and compatible with the proposed design. Additionally, it is necessary to avoid designs that theoretically function well as a computer model but would not be physically feasible to manufacture or assemble.

Although the fixture will not be subjected to significant mechanical forces, it does need to be sturdy enough that it will not break under these forces. Finally, the fixture needs to be safe, however the fixture is not likely to include components that are inherently dangerous, although care needs to be taken during the manufacturing process to ensure that there are no sharp edges.

**4.2 Fabrication of Nanowires**

In order to measure nanowire conductivity, various samples of Zinc Oxide nanowires on quartz had to be synthesized. In the Nanoenergy Lab, our group practiced and refined the process of growing ZnO nanowires. Samples were made sequentially over the course of the MQP in order to find the best method for growing unaligned and aligned nanowires. There are three main steps: selection and preparation of the substrate, seed layer deposition, and nanowire growth.
4.1.1 Selection and Preparation of Substrate

First, the substrate must be prepared. Quartz substrate was used instead of the standard glass or silicon because quartz is transparent to a THz beam. A transparent substrate is necessary so that the beam can pass through the substrate and measure the sample directly. Quartz is also transparent to visible light, which is required for use as a substrate in a solar cell. 1 mm thick quartz samples were cut into a reasonable sample size for the experiment (0.5” x 1.0”). The quartz is cut by carefully etching a marked line with the diamond cutter and then splitting with a bending tool. Samples are generally made in batches of three, which increases the chance of a successful growth, as well as being the maximum number of samples that should be used in one bath. Next, the substrates are cleaned by placing the samples in a beaker with enough alcohol mixture to cover them, and then suspending the beaker in a sonication machine on high power. The alcohol mixture is a rough 1:1:1 ratio of acetone, isopropyl alcohol (IPA), and deionized (DI) water. The sonicator is a machine that uses ultrasonic waves to mix substances or, in this case, remove fine particles from the surface of the quartz crystal. All marker lines and dust particles are gone after 10 minutes of sonication. After cleaning, the samples are removed from the alcohol solution and rinsed with water.

In an effort to grow more vertically aligned nanowires, as will be discussed later, we tried using silicon as a substrate rather than quartz. It has been shown that ZnO nanowires can grow with a high degree of alignment on a silicon wafer [Yang 2005]. The process for seed layer deposition and solution bath is nearly the same.

An additional cleaning process that helps nanowires stick to the substrate better was developed in C-term. This process involves a sequence of three 30-minute baths: The first is 4.4 mL H₂SO₄ with 0.8g K₂Cr₂O₇ in 15 mL DI water. The second is only nitric acid. The final bath
is 0.25 EDTA with 0.2g NaOH in 20 ml DI water. The samples are rinsed with DI water between each step. After the sequence of baths, the samples are ready for spin coating.

4.1.2 Seed Layer Deposition

Spin coating is used to prime one side of the sample for growth by depositing a thin layer of seed crystals. The samples are transparent and symmetric, so we etched one corner on one side of each sample to indicate the seeded side. One quarter of the to-be-seeded side is first covered with tape, so that a reference measurement for the THz spectroscopy using uncoated quartz can be made for each sample before measuring its respective nanowires later. Next, a pipette is used to coat the rest of the surface with the chosen solution. We chose two different solutions to compare the alignment and success of nanowire growth for each. We used ZnO nanopowder for half of the samples and zinc acetate dihydrate (ZAD) for the other half.

For ZnO powder, the process is as follows. 75 μL of ZnO powder in ethanol is deposited on the sample by using a precision pipette. The sample is affixed within the spin coating machine by a vacuum seal, and spun at 2000 rpm for 30 seconds. Finally, the sample is annealed on a 100°C hotplate for one minute. Once this is complete, the sample is re-covered with the solution and spin coated again to create a denser coat. Literature states that the process may be repeated 1-5 times, and some testing was done to determine an ideal number. We settled on two times. Since nanowires appear as a white film to the naked eye, only by using SEM imaging can we determine whether the number of initial coats, as well as the entire process, was successful.

The other half of the samples were coated using ZAD. 100 μL of 5 mmol ZAD in ethanol is deposited on the sample using a precision pipette. The sample is affixed within the spin coating machine by a vacuum seal, and the sample is spin coated with a machine at the same
RPM, 2000, but for 40 seconds. Afterwards, the sample is rinsed with ethanol by holding the sample at approximately 30°C and allowing ethanol to run down the coated surface of the substrate. It is then dried with the nitrogen air hose. The process of coating, rinsing, and drying is repeated for a total of 3-5 coats. Finally, the samples must be baked in the box furnace at 350°C for 20 minutes for annealing. Some papers recommend repeating the spin coating and annealing process once to ensure an even coating, but our experiments didn’t find this to be a consistent improvement. Once the seed layer deposition is complete, the samples are ready for the nanowire bath.

4.1.3 Nanowire Growth

The samples must be suspended in the growing bath at about a 30 degree angle with the spin coated side facing downwards. This is important as the solution contains particles that will settle due to gravity on the upward facing part of the sample. If the nanowires were being grown upwards, the settlement would ruin the forest of nanowires. The solution is a 50 mmol bath consisting of DI water, zinc hydride (ZNH), and hexamethylenetetramine (HMT). The sample is suspended by tying a metal wire around the non-coated (and tape covered) end of the glass, thereby hanging from the edge of the beaker. All samples that are in one beaker must rest at the same height so as to ensure equal heating. Heating rate during this growing solution is also important. The bath must be quickly heated from 0° to 90° C (accomplished by setting heat to 300°C at the start, then lowering to 150°C when the thermometer approaches 70°C). Although the hot plate reads 150°, the temperature within the beaker will only reach 90°C. Once at this temperature, the sample must bathe for four hours.

The last step is to remove the sample from the bath, wipe the side that is without seed
layer, and then immerse the samples in DI water for 5 minutes. Gently changing baths a couple of times was useful to thoroughly wash the samples. Finally, the nitrogen flow is used once more to completely dry the samples. Following this, the nanowires can be photographed with SEM imaging and, if growth was successful, THz spectroscopy.

The measure of a successful growth is whether the surface of the sample is evenly coated with the expected kind of wires. Visual inspection is used to select the best samples, and is followed up by SEM imaging at 1,000X and 25,000X resolution in four corners and the center of the sample to take a closer look at the nanowires. After Terahertz measurements are made, the samples can be split to take cross-sectional SEM images so that the exact height of the wires can be measured. For a four-hour growth using the predescribed methods, ZnO nanowires tend to grow about 3 micrometers tall and 50-100 nm in diameter. The aspect ratio, which is the ratio of length to diameter of these wires, generally comes out between 20 and 100.

4.1.4 Developing the Process

Since the goal of this research is to measure the charge carrier injection rate and axial conductivity of the nanowires, it was necessary to synthesize sufficiently aligned wires without the wires being too closely packed for future individual coating. We grew over half a dozen batches of nanowires throughout the course of the project, learning from our mistakes and improving the process each time.

The most important factor that we had control over was the choice of seed layer. It is possible to use different types of solutions, as well as varying concentrations of each solution, for just one material. We experimented separately with the ZnO ink as well as with ZAD nanoparticles. The ZAD tended to create a better seed layer, in that the nanoparticles stuck to the
surface and formed an even coating. It also resulted in a slightly more aligned array of nanowires, probably due to the coating density. Another factor affecting the quality of the seed layer is the number of times each sample is spin coated and annealed. For most samples, we spin coated between 2 and 4 times and then annealed. It is difficult, however, to tell what the appropriate number is. The seed layer is invisible to the naked eye, and varying the number of coatings, or even repeating the coating and annealing process once, had no measurable effect resulting nanowires. It is likely that this observation was due to the limited number of samples produced over the course of this MQP, and limited experience of our team.

The acid bath prior to seed layer deposition greatly increased the seed layer density on both quartz and silicon samples. Use of this method also increased alignment of nanowires. A common theme that arose through analysis of SEM images was that the consistency and thickness of nanowires on a sample strongly correlated with alignment of the wires. It seemed that the ZnO nanostructures would grow in random directions on the substrate if not confined by their neighbors. The unresolved question, therefore, is how to grow wires that are confined and vertical while also allowing for coatings for use in a nanowire solar cell.

4.1.5 Laboratory Procedures

All work done in the lab requires the use of latex gloves. These gloves protect samples and devices from the dirt and oil that our hands bring into the lab. They are not enough to protect against strong acids or high heat. Other personal protective equipment (PPE) may be used when operating the box furnace, handling specific hazardous chemicals, or in danger of inhaling fumes. All broken substrates or other glass containers should be placed into a bin specifically labeled “Broken Glass”. Pipettes use disposable heads that can be dropped into a regular trash
bin once the solution is deposited. Beakers containing solution such as the growth bath must be
dealt with in a manner that safely disposes of all chemicals. This usually involves pouring the
leftover solution into the hazardous materials container. Residual chemicals in the beaker are
washed out with a strong acid under a fume hood. The acid must be properly disposed of, and the
beaker is rinsed afterwards with DI water several times. Used ethanol and any other chemicals
used also have specific disposal containers.

5. Design Process

5.1 Preliminary Designs for Test Fixture

A few preliminary designs were made for the test fixture. These designs were initially
generated on paper, and then using computer-aided design software for better visualization and
ease of editing. The purpose of these preliminary designs was to help establish the parameters
that are most important in designing the fixture, as well as modeling and determining what may
or may not work. During the development and assessment of a few preliminary designs, several
deficiencies in designs were identified and improved in design evolutions.

The nanowire sample holder currently in use is a rounded rectangular, aluminum piece
that drops into an existing platform. Two main preliminary designs were developed in tandem.
These designs focus on the general movement of the fixture as a whole, and although preliminary
methods to hold the sample in place were considered, various mechanisms to hold samples in
place were developed in greater detail afterwards. After assessing the first designs, evolutions of
the two main preliminary designs were developed.
5.1.1 Preliminary Design 1

This first preliminary design utilizes a rounded rectangle with the same outer dimensions as the current sample holder so that it will drop into the existing platform without requiring any modification to the existing laboratory setup. Included in the rotating portion of the fixture is a slotted track with a cutout at the bottom, with steps to either side of the cutout. The sample would rest on the steps, and the beam would pass through the cutout on the bottom. Because the cutout is perpendicular to the axis of rotation, chopping the beam as a result of rotating the fixture should not be a problem, provided that the width of the cutout is greater than the beam width (approximately 1.5 millimeters).

Because this is a preliminary design, there are design issues that need to be addressed. The slot system requires that the sample be a particular width, but that could be remedied with a type of sliding/tightening mechanism. Also not included is any mechanism to hold the rotating stage at the desired angle, although this is addressed in a later design evolution. This design is fairly large relative to the preexisting holder, and there needs to be sufficient clearance so that other parts are not struck or interfered by the rotating stage. The length of the stage could be shortened without significantly affecting the effectiveness of the stage, which could eliminate the clearance problem. Additionally, a readout to set the angle of the stage would be needed. An idea for a readout could be some markings affixed to the rotating portion that displays the angle relative to the fixed portion.
5.1.2 Evolutions of Preliminary Design 1

Preliminary Design 1 was refined to better take into account the constraints and goals of the project and improve upon design issues that were identified. Although the general kinematic movement is similar, the axis of rotation was changed so that the axis of rotation is around the long axis of the rectangular holder and therefore the required clearance is reduced; this was changed after noticing that one of the parabolic mirrors may interfere with a mechanism for rotating the stage.

The mechanism for controlling the angle would be a knob that is concentric with the axis of rotation. The knob in this design evolution is contained within the rectangular holder and rotates with the inner portion; this is not likely to interfere with other parts of the lab setup and would not require a complex gear train, pulley system, or something similar. It could contain angle markings with reference to the stationary rectangular holder. Ideally, the angle of the stage would be maintained by the frictional forces between the component interfaces.
Another design aspect explored in evolutions of Preliminary Design 1 concerns in-plane versus out-of-plane rotation for the rotating portion of the device. A CAD model of a design with out-of-plane rotation can be seen below.

This design is similar to that of Preliminary Design 1 and the first evolution of that design, but the rotating portion is suspended above the non-rotating portion, so those two components are not in the same plane when the movable portion is not rotated. Additionally, the
rotating portion is not supported within the outer frame as in Main Body Design 1 and the first design evolution, but is supported by blocks that protrude above the outer frame. Therefore, the rotating portion is outside the plane of the non-rotating portion, and the axis of rotation is raised above the outer frame.

The out-of-plane design was created to address a difficulty in assembling the former in-plane design. With the in-plane design, the rotating portion included cylindrical protrusions on either side to serve as the axis of rotation. However, as the components must be manufactured separately, inserting the rotating portion into the non-rotating portion would be difficult or impossible. The protruding blocks of the out-of-plane design provides a possible solution for this problem, as a bolt or shaft can be inserted through the blocks and into the rotating portion. An advantage of this out-of-plane design is the angle adjustment knob may be easier to turn compared to the in-plane design. With this design, the top, left, and right extreme points of the knob are exposed, while in the in-plane design, only the topmost point is exposed.

A drawback of this design is that the clearance between the stage and other equipment, such as the parabolic mirrors, is reduced because the stage is now higher when compared to the in-plane design. This could limit the maximum angle of rotation that can be achieved. The vertical adjustment of the XYZ-stage that the design will be mounted on may not be able to sufficiently compensate for the increase in height.

5.1.3 Preliminary Body Design 2

Another initial concept was developed independently of Preliminary Design 1. A base that slots into the current stage was created as a setting for the rotational stage. Added to this base is a stand that can hold a shaft which supports the stage. The stage rotates about the center of the shaft, which was constructed to be the same point as the top of a testing sample. This was
necessary so that the same portion of nanowires are being analyzed when the sample is rotated.
Part of the design was inspired by a dial that was found in the optical equipment cabinets. The
dial is about two inches on each side, has markings that go a full 360 degrees, and is threaded in
the middle. This dial was incorporated into the initial SolidWorks design as a means of not only
rotating the stage – for the dial has a knob to do so – but also to measure the angle of rotation.
Previously, these were two major design constraints. With the rotating shaft threaded into the
mounted dial, they should both be solved.

Several problems with this design were realized. An elementary design flaw was that
there is no space for the dial to rest along the short end of the fixture. Deeper design flaws reside
in the fact that creating a 3mm hole for a THz beam to be focused creates a limit on material
thickness. In other words, when the stage is rotated, the thickness of the material will clip the
beam. Since the beam needs an opening of 1.5 mm to 3.0 mm, this limited the thickness of the
stage to a couple millimeters. This causes the stage to be very thin and therefore unstable.

Figure 9: Preliminary Design 2
5.1.4 Evolution of Preliminary Design 2

Preliminary Design 2 was evolved as a more reasonable and complete idea of the testing fixture to be produced. Ease of manufacturing was a priority during the creation of this model. Inside corners should be rounded and outside corners should be sharp, as it is easier and faster for the CNC End Mill to perform the operation in that manner. Also material thickness, size of parts, and stability of the shaft and stage were considered during creation of the SolidWorks model.

The slot cutout idea found in Preliminary Design 1 was accepted for THz spectroscopy and therefore eliminates the issue of material thickness and beam clipping. The angle control dial was moved to the long end of the stage as that is the only place where space is available and out of the way of the THz beam. A simple clamping mechanism using a spring clip embedded in the stage is included as one possible way of holding the sample in place during stage rotation.

Figure 10: Evolution of Preliminary Design 2
5.2 Mechanism for Holding Sample

Following the initial design effort that produced several ideas for the general structure of the test fixture, methods for holding the sample in place were developed in further detail.

5.2.1 Movable Ledge with Interference Fit

One method for holding a sample in place considered in the preliminary test fixtures designs is a movable ledge that the sample will rest against when the stage is angled. Because the sample would simply rest against the ledge, the risk of damage to the sample is reduced compared to a clamp. The ledge is inserted into the same groove where the sample rests, and moves along the groove to hold the sample in the desired location. One mechanism to hold the ledge in place is an interference fit. With an interference fit, a semi-flexible material, such as plastic strips, could line the groove that the ledge is inserted to. No moving parts would be necessary. The ledge would be moved along the groove by exerting a force; the weight of the sample itself would not be enough to move the ledge.

5.2.2 Movable Ledge with Rotating Arm

A second method for holding the sample in place involves a similar movable ledge, but this time the position of the ledge is maintained by contact with a rotating arm. The rotating arm would need some sort of mechanism to keep it stationary, such as a tightening mechanism or interference fit.
5.2.3 Slotted Arm

An evolution of the previously mentioned movable arm design is a slotted arm. In this design, one end of a slotted arm pivots around a fixed location, and ledge with a dowel above it is inserted into the arm’s slot. The ledge would slide along the rectangular cutout of the stage when the slotted arm is rotated. Similar to the previous design, the slotted arm would require a
mechanism to keep it in place. One possibility for this is incorporating a dowel with external threads and a wing nut, which would tighten the slotted arm to the ledge and keep the assembly in place.

Figure 13: Slotted Arm

5.2.4 Pressure Clamp

This clamp system is a simple pressure clamp. The team felt that a clamp that applied pressure to the top of the sample would most likely weaken over time, as such clamps generally use a spring. In this design, we attempt to solve that problem by having a clamp that applies pressure to the sides of the sample using a threaded screw mechanism. Rubber pads are attached to the ends of threaded rods, which are screwed through the threaded holes in the sides of the clamp frame. A sample is placed between the rubber ends, and the screws are tightened until the sample is firmly affixed between the rubber pads.

Unfortunately, this design has a few problems associated with it. Most significantly, the pressure applied could potentially cause damage to a sample if it is delicate. This is something to consider for all clamping mechanisms. In addition, the clamp is necessarily relatively bulky
compared to the stage, simply because the frame has to wrap around the sides in order to avoid interfering with space for the sample. The screws of the clamp may also interfere with the rotational capabilities of the stage.

Figure 14: Pressure Clamp
6. Physical Prototypes

6.1 Rudimentary Prototype

For physical prototypes, first a rudimentary prototype consisting of various clamps, rotation stages, and a small machined piece of aluminum with a cutout for the THz beam was constructed. The rotating portion of one rotation stage is screwed to a horizontal rod, and a second rotation stage is glued at perpendicular angle to the first rotation stage. The machined aluminum piece is taped to the second rotation stage. Having a physical model allowed for better understanding of how the area near the cutout on the aluminum piece for the THz beam could clip THz beam, namely that the area around the cutout should be as thin as possible. This led to further design refinements reflected in our first prototype.

The second rotation stage was intended to be simply a platform, but it could potentially rotate the machined aluminum piece that sits atop of it. Upon observing this, a potential user for the THz test fixture expressed a desire for the samples to rotate not only about the horizontal axis, but also about the vertical axis without having to contact the sample itself. The samples would need to rotate about the horizontal axis in ninety degree increments. A basic exploration of how two-axis rotation could be incorporated into our first prototype design is discussed later.
6.2 Design of First Physical Prototype

For the first physical prototype, it was decided that an in-plane design would be used, given the vertical adjustment limitations of the XYZ-stage. A tightening mechanism for holding the angle in place would be used instead of a friction-based approach found in the preliminary designs. Friction-based components used to hold the angle, such as a semi-pliable knob for adjusting the angle, could wear out over time or may not be sufficiently secure and cause undesirable movement of the stage. The first prototype utilizes several commercially-sourced parts; a prototype with custom parts manufactured in-house may be explored in the future. A mechanism for holding the sample in place would not be included in the first prototype; this feature will be included in later prototypes.
The first prototype contains design features from the preliminary designs as well as new features not found in previous designs. Further refinement of CAD models was therefore needed before the prototype could be manufactured. The rotating portion, the stage, is a rounded rectangle with an overall thickness of half an inch, and it contains a depression where the samples will rest, with a cutout on the bottom for the THz beam. The depression is necessary for making the area surrounding the cutout as thin as possible to avoid clipping the beam. The depression in the first prototype is much larger relative to previous preliminary designs to increase the range of sample sizes that the stage can accept. The thick, non-depressed part of the rotating stage contains a set of internal threads on each side for screws that serve as the axis of rotation. Unlike previous designs, the first prototype’s non-rotating portion, the base, is not simply a rounded rectangle; it contains cutouts on both ends, one larger than the other. The larger cutout is for the angle indicator which is outside of the base, and the smaller cutout is for the head of a screw that serves as the axis of rotation. The base contains two non-threaded holes concentric to the axis of rotation, one hole on each side.

An angle indicator was removed from a Newport Research Corporation RSX-1 rotation stage for use in the first prototype. The angle indicator contains internal ¼-20 threads, which dictated the use of bolts with ¼-20 threads. On the side of the fixture with the angle indicator, a fully threaded bolt slides through and connects the angle indicator, one side of the fixed base, and one side of the rotating stage. Flanged sleeve bearings are inserted into a hole in the base to reduce friction. On the other side of the fixture, a partially threaded bolt slides through the base and into the rotating stage. The threaded portion at the end of the bolt engages internal threads in the rotating portion. The non-threaded portion of the bolt turns freely inside the non-rotating portion, with a wing screw positioned above the bolt. The wing screw can be tightened to hold
the bolt and the rotating stage stationary. Flanged sleeve bearings are used to reduce friction, and the length of the sleeve bearings is short enough to allow for part of the non-threaded portion of the bolt to be exposed for contact with the wing screw.

Figure 16: CAD Model of First Physical Prototype; Isometric View
Figure 17: CAD Model of First Physical Prototype, Top View
6.3 Fabrication of First Physical Prototype

The first physical prototype was fabricated using a Haas CNC MiniMill. For both the frame and the stage, a face mill was used to smooth the aluminum stock and reduce its height to the desired height of half an inch. A $\frac{3}{8}$” end mill performed the cutting operations. A drill press was used to form the holes for the wing screw and sleeve bearings on the frame, and the holes on either side of the stage. Internal threads were formed using a tap.
When all of the pieces of the device were assembled, it was easy to move the dial in such a way that it did not turn along with fully threaded screw that connects the dial and the stage. When turning the dial clockwise, the dial would tighten against the frame and make rotation difficult. A solution was reached where a nut was inserted between the dial and the frame. The nut is tightened up to the dial and vice versa, so rotating the dial clockwise does not cause the dial to move relative to the bolt and push into the frame.

We encountered several challenges during the fabrication of the first physical prototype. When machining the frame, an error with the order of milling operations left the frame with a very thin and sharp lip; this lip was removed using pliers and a file. An error with setting the work offsets in the CNC machine resulted in the tool path contacting the frame in a place that was not intended, however the machining program was stopped shortly after this was realized and the overall effect on the function of the prototype was minimal. The cutout for the partially threaded bolt was too narrow; this was remedied by filing away material until the bolt could fit. The tolerance between the frame and the stage was too small, so the stage could not easily fit within the frame. The stage was filed and buffed until the length was slightly reduced and the stage could fit into the frame; a sander could not be used for this because it was inoperative.

Figure 19: Unassambled First Prototype
6.4 Design Changes to Physical Prototype

During and after the fabrication and testing process for the first physical prototype, we identified aspects of the device that can be improved upon in further prototype iterations and for the final device. In the first physical prototype, there was not enough clearance for the partially threaded bolt that acts as a shaft. There were also clearance problems with placing the stage within the frame; there was too little clearance which resulted in too much friction between the sleeve bearings and the stage. This, in turn, caused the dial and the bolts to become unscrewed.
from the stage. For the final device, we enlarged the cutout dimensions for both the partially threaded bolt and the stage to ensure that there is sufficient clearance.

The set screw design, which was intended to hold the bolts, stage, and dial together at an angle, was not effective when contacting the partially threaded bolt. The lack of effectiveness is likely because the frictional forces between the dial and the partially threaded bolt compete with each other. We attempted to remedy this problem by locating the set screw nearer to the dial, and above the fully threaded bolt. However, the problem with undesired turning of the stage persisted, because the bottom of the set screw was in contact with screw threads and had a very small contact area size. Additionally, when the stage was placed at an angle, tightening the set screw caused the stage to rotate by a few degrees. To solve this issue, we determined that in the design of our final device we will use a friction system to apply force against the dial. The bolts used as shafts and the dial will be tight enough that the components move together.
7. Fabrication of Final Device

For the final device, we incorporated design improvements that we identified while analyzing our first physical prototype. To apply friction to the dial, a rubber pad was inserted in between the dial and one of the frame protrusions surrounding the dial. The set screws and the associated threaded holes are no longer features of the final device. The frame protrusion on the opposite side of the dial was reduced in height so that the top of the protrusion is level with the center of the dial’s rotation axis. This can be used to read the angle marking off of the dial more easily than was possible with the previous prototype. In support of both of these design changes, the distance between the inside of the frame protrusions and the dial was reduced. Lastly, the size of the rotating stage was slightly enlarged.

As with the first physical prototype, the stage and frame of the final device was machined out of a piece of aluminum, and a drill press was used to form the necessary holes, and tap to form screw threads where needed. The design changes compared to the prototypes was successful, as there was no contact in between the stage and the frame, and the rubber pad successfully held the dial, shafts, and stage at any angle.
Figure 21: Final Device

Figure 22: Final Device in Laboratory Fixture
8. Results

8.1 Nanowire Synthesis

SEM images were taken of each generation of nanowires in order to analyze and understand the results of each process. Uniformity of coating, spacing and alignment of wires, and height of sample were important factors to understand for taking measurements. The following image is an example of samples that were fairly evenly coated and thus selected for SEM imaging.

Figure 23: This is an example of the quartz samples. Note how a portion of each sample is
uncoated to allow for substrate attunement.

Figure 24: Silicon samples loaded on SEM holder for imaging

Above, seen in Figure 24, is a picture of silicon samples loaded onto an SEM holder with the help of double sided copper tape. The SEM will only be able to take top-down photos of these samples.
Figure 25: One of the first attempts at synthesizing ZnO on quartz

Seen above in Figure 25 is an SEM image that was taken of the ZnO nanowires on quartz. This sample has a few notable traits. The wires are very aligned, as you can’t see their sides from a top-down view. They are also very close packed, which might cause inability to individually coat wires. The third is the large shards resting randomly on top of the sample. This might be dust or some other chemical that wasn’t properly removed during synthesis.
Figure 26: ZnO on quartz

Figure 26, above, is an image of a later synthesis of ZnO on quartz. With changes to the seed layer deposition methods, this sample shows better wire spacing and the absence of debris, but less alignment.
This image, Figure 27, shows ZnO grown on silicon substrate, in hopes that alignment would be increased. Here we can see that the nanowires are actually much thinner than previously, with spacing and consistency very good as well. However, alignment is not much improved.

### 8.2 Stage and Terahertz Measurements

Following the fabrication of the final device and reasonable nanowire samples, the device was used to measure conductivity in the nanowires with terahertz spectroscopy. The formerly used, non-rotating test fixture was removed and replaced with the device we fabricated. The
device fit into the xyz translation stage well and was easily operated. A ZnO nanowire sample was placed into the stage for conductivity measurements. In addition to measuring the sample while flat, additional measurements were taken at 10 degrees and 20 degrees. For each of these three trials, the terahertz pulse through the nanowire samples was measured and compared to a reference terahertz waveform, which was the terahertz waveform through only the quartz substrate. As expected, the waveform of the terahertz pulse through the nanowire samples was attenuated compared to the reference terahertz waveform.

Figure 28: Final Device in Use
For each of the three trials, a Fourier transform was performed to obtain the frequency domain of the terahertz pulses from the time domain. The following equation, where $n$ is the refractive index of the substrate, $Z_0$ is the impedance of free space, and $d$ is the sample thickness, was used to obtain the complex-valued conductivity of the nanowires. The output of this equation is multiple data point values for conductivity that tend to follow a trend.

\[
\frac{\tilde{E}_{\text{film+substrate}}(\omega)}{\tilde{E}_{\text{substrate}}(\omega)} = \frac{1 + n}{1 + n + Z_0 \tilde{\sigma}(\omega)d}
\]

The Drude model below is used to fit a line to the data points and allows for extraction to zero frequency, or DC conductivity.

\[
\tilde{\sigma}_{DS}(\omega) = \frac{n_{\text{film}} e^2 \tau_{DS}}{m^*} \left[ 1 + \frac{c}{1 - i \omega \tau_{DS}} \right]
\]
The graphs below show the conductivity data points and their associated best fit line for both real and imaginary conductivity values, for trials at 0, 10, and 20 degrees respectively.

Each trial resulted in a different $\tau_{DS}$ value, which is the average time between electron scattering events and is representative of the nanowires’ conductivity.
9. Discussion and Future Recommendations

Our device was successful in allowing nanowire samples to be probed by terahertz spectroscopy at various fixed angles. However, although the difference in conductivity at various angles is measurable, it is very small. This is a result of the nanowire material properties themselves. The mean free path of electrons in our samples, that is the average distance a charge carrier moves before interacting with a defect in the crystal, is on the order of a few nanometers. The width of nanowires in our samples was about 100 nanometers when grown on quartz, and 10 nanometers when grown on silicon. In either case, the mean free path distance was much shorter than the width of the nanowire, meaning that an electron is likely to interact with a defect before reaching a grain boundary. Therefore, vastly increasing the grain width by rotating an aligned nanowire sample would have little noticeable effect on the tau parameter. The tau parameter is the average time a charge carrier spends between collisions. Ideally, it should increase several times when the sample is rotated because there is a lot more horizontal distance that a charge

<table>
<thead>
<tr>
<th>Angle</th>
<th>$\tau_{DS}$ (fs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>30.03±0.035</td>
</tr>
<tr>
<td>10</td>
<td>30.66±0.055</td>
</tr>
<tr>
<td>20</td>
<td>32.97±0.057</td>
</tr>
</tbody>
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carrier could travel before hitting a grain boundary. The comparatively small mean free path here, however, prevented this.

One way to increase the mean free path and see more meaningful change in tau with rotation would be to reduce impurities in the nanowire samples. There are several possible ways of doing this, and this would be a focus for future work. Possible solutions include annealing more frequently or at higher temperatures. A change of material to something that grows with higher degree of crystallinity would help. One could also develop ways to grow thinner nanowires with higher aspect ratios.

Once a better material is developed, the seed coat depositions process and alignment of wires needs to be better understood. It seemed that the more spaced out the nanowires grew, the more likely they were to branch in random directions. This is because zinc oxide has several preferred crystal orientations, so if given the space it will grow from the substrate in a random direction. Since it is desired to have wires only grow along the z-axis, one needs to find a way to eliminate the possibility of the other options. It seems that increasing the seed coat density causes for a denser coating of wires, which in turn forces wires to grow more vertically. However, if the nanowires are too tightly packed, it can cause a short circuit for charge carriers. It is therefore important to strike some balance between nanowire density and alignment, or else find another material or growth method that forces the nanowires to grow along a single axis.
10. Summary & Conclusion

The ultimate goal of this project was to design, manufacture, and test a device that allows for the electrical characterization of nanowires along the length of the wires. The conductivity of nanowires can be obtained using terahertz spectroscopy, where a terahertz waveform probes nanowire samples. Because charge carriers present in the nanowires exhibit strong absorption in the terahertz range, the terahertz waveform that exits the sample is attenuated and delayed; this can be measured and compared to a reference terahertz waveform to determine conductive properties of the material. It is desirable to measure the axial conductivity of nanowire structural materials, as well as the carrier injection rates when coated with a semiconductor, in order to determine the suitability of nanowire solar cells as an alternative to traditional silicon wafer-based solar cells. Compared to traditional solar cells, nanowire solar cells allow for cheaper, more abundant, and less toxic materials to be used, in addition to improving the efficiency of the cell due to the shape of the nanostructures. Angled measurements of nanowire samples can be achieved by tilting the sample, something that was previously not possible with test fixtures available.

We designed several iterations of a device that allows for angled measurements of nanowire samples. Constraints that the device needed to meet include compatibility with other laboratory equipment, ease of use, manufacturability, and ease of assembly. After several rounds of concept generation using computer-aided design, a physical prototype was fabricated. Following testing and analysis of the physical prototype, a final device was manufactured that took into account necessary improvements identified with the prototype.

We grew our own nanowires with the intention of using them for conductivity measurements by terahertz spectroscopy. Outcomes of nanowire fabrication sessions varied
greatly in terms of nanowire alignment, spacing, and consistency. Variables such as seed layer type, seed layer deposition methods, annealing, and substrate material were modified in hope of achieving nanowire samples suitable for testing. Further work is required in this field.

Conductivity measurements using our device and nanowire samples were performed. Conductivity measurements were taken while the sample lay flat, at 10 degrees, and at 20 degrees. Although there was a measurable difference in conductivity between these three angles, the difference was very small. This is likely due to a material property of zinc oxide that causes electrons to interact with defects before they reach the sidewalls of a nanowire. Because of this, tilting the nanowires has a comparatively small effect.

Overall, the project was successful in meeting the objectives of developing, testing, and analyzing multiple prototype iterations, fabricating physical prototypes, fabricating a final device that fully takes into account improvements identified from earlier iterations, synthesizing nanowires, and measuring the conductivity of nanowires using terahertz spectroscopy. Future work that builds upon the outcomes of this project would be more materials focused rather than design focused, with a potential goal of improving nanowire synthesis methods so that changes in nanowire conductivity for varying angles would have a more drastic difference.
References


