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**Verification of Piezoresistive and Piezoelectric Properties in 2D
Transition-Metal Dichalcogenide PtSe₂ and PtTe₂ Materials**

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**Verification of Piezoresistive and Piezoelectric Properties in 2D
Transition-Metal Dichalcogenide PtSe₂ and PtTe₂ Materials**

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Abstract

Verification of Piezoresistive and Piezoelectric Properties in 2D Transition-Metal Dichalcogenide PtSe₂ and PtTe₂ Materials

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The University of Texas at Austin, 2022

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Transition-metal dichalcogenide (TMD) materials have promising properties that make them suitable for wearable bioelectronics and biosensors. Specifically, the Platinum based TMD materials, PtSe₂ and PtTe₂, can be fabricated at low temperatures, allowing for growth on flexible substrates, such as polyimide film. Exploration of piezoresistivity and piezoelectricity in PtSe₂ and PtTe₂ can enable more applications for the novel 2D material. This report focuses on the experimental setup and results for the verification of the piezoresistive and piezoelectric property in PtSe₂ and PtTe₂. The piezoresistivity experiment focuses on the change in resistance in the material due to strain induced by bending using a two-point bending fixture. In the piezoelectricity experiment, samples are put in a periodic strain at 2 Hz with the voltage response of the material being measured using a digital oscilloscope. An analysis of the experimental results is discussed along with a proposal for further applications for using piezoelectric materials as energy-harvesting elements of the next-generation bioelectronics.

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Chapter 1: Introduction

With the success of graphene, there has been a growing interest in novel 2D materials and their unique and useful properties [1]. A group of materials that has been growing in interest is the transition-metal dichalcogenide (TMD) materials, such as MoS₂, for its plethora of unique properties [2]. TMD materials are made up of a M, transition metal atom, and a X, chalcogen atom, in the form of MX₂ [2]. The unique layering structure of TMD materials gives them interesting thickness dependent electronic properties [3]. Bulk TMD material has semimetallic or metallic properties while multi- and single-layered TMD material has semiconducting properties [3].

Some promising TMD materials are Platinum Diselenide (PtSe₂) and Platinum Ditelluride (PtTe₂). These Platinum based TMD materials require a lower fabrication/growth temperature when compared to other TMD materials such as MoS₂, making them great candidates for use with standard semiconductor processes and a multitude of substrates [4]. One of these potential substrates that PtSe₂ and PtTe₂ can be grown on is polyimide film, making these Platinum based TMD materials great for applications in wearable bioelectronics and biosensors [3]–[5]. The atomically thin thickness of these materials makes them great candidates for human skin contact with their uniformity and flexibility to the skin.

On top of these properties of PtSe₂ and PtTe₂, there are more properties to be explored and verified that can expand the applications of the Platinum based TMD materials. In this report, the piezoresistivity and piezoelectricity in PtSe₂ and PtTe₂ is explored. The fabrication of the samples and the experiments for verification of the piezo properties are provided and analyzed.

Chapter 2: Literature Review

For the piezoresistivity and piezoelectricity experimental setup for this report, multiple techniques were explored and adapted to fit constraints of the lab equipment available. To measure the change in resistance due to varying strain in the material, a measurement technique for strain needed to be established for the experiments.

PIEZORESISTIVE AND PIEZOELECTRIC PROPERTIES

A material with the piezoresistive property has its resistance changed under applied mechanical changes, such as strain. In piezoresistive materials when a strain, ε , is induced in the material, the resistance changes such that

$$\Delta R/R_0 = G \times \varepsilon$$

where G is the gauge factor of the material [4].

A material with the piezoelectric property generates a voltage under applied mechanical changes, such as strain. There are two types of piezoelectric 2D materials: in-plane and out-of-plane. MoS₂, a TMD material, has been shown experimentally to be an in-plane piezoelectric material with other TMD materials predicted to be in-plane piezoelectric materials as well [6]. Strain induced in MoS₂ displaces Molybdenum (Mo) cation atoms and Sulfur (S) anion atoms, creating electric dipoles that result in piezoelectric polarization charges on the material's surface [6].

MEASUREMENT TECHNIQUES FOR PIEZORESISTIVITY AND PIEZOELECTRICITY

In a previous study, piezoresistivity in a material was verified by measuring the resistance of the material while bending the thin film material [4]. Depending on the mechanism used to manipulate the material, the bending technique varied. The thin film material was bent using a custom three-point flexure rig and a cantilever bending setup

[4]. In order to verify the piezoresistive property, the strain of the material due to the bending structure has to be recorded alongside the resistance of the material in order to see a correlation between material strain and the resistance of the material. These measurements are enough to see a correlation between strain in the material and resistance of the material, verifying piezoresistivity.

For piezoelectricity, there are multiple measurement and verification techniques as well as multiple sample preparation methods. There are experiments with samples as simple as having the thin film material be cut into rectangles with a length of 25 mm and a width of 10 mm with electrical contacts made using colloidal silver paint [7]. There are other experiments that require samples to be prepared more rigorously with etchants to make metal-insulator-metal (MIM) structures or interdigitated electrodes out of the sample material that are then placed on plastic substrates [8], [9].

Similar to piezoresistivity experiments, there are multiple ways of bending the material such as using a two-point, three-point, or four-point flexure rig. In one of the piezoelectricity experiments, the samples are mounted on an aluminum plate which is then fixed on one end of the plate with a vice. The other end of the plate is then attached to a modal shaker, which bends the aluminum plate with the samples mounted at frequencies of 40 Hz to 110 Hz [7]. Overall, most experimental set ups require a periodic excitation of the sample material.

The actual measurements for the verification of piezoelectricity that are needed are done using an electrometer or an oscilloscope. These measurement tools are used to measure and detect a voltage response that is generated from fast bending of the material. Piezoelectric materials under excitation from bending will generate a noticeable voltage response, allowing for verification of the piezoelectric property in a material.

MEASUREMENT OF STRAIN ON A MATERIAL

With growing interest in 2D materials over the years, there has been a lot of studies exploring and observing a material's properties with the application of strain on the material. This report uses strain calculations based on bending of thin films to adhere to the bending experiments for the verification of piezoresistivity and piezoelectricity in PtSe₂ and PtTe₂.

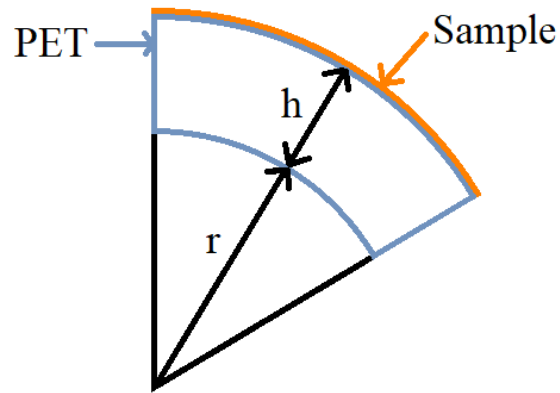


Figure 1: Bending Schematic of Sample on PET [10]

Figure 1 above shows the bending schematic for a sample of PtSe₂/PtTe₂ placed on PET material. The h denotes the thickness of the PET material, and the r denotes the radius of the arc of the PET material, which the PtSe₂/PtTe₂ sample is fixed on. From these two measurements while the sample is bending, the bending-induced surface strain, ϵ , on the thin film PtSe₂/PtTe₂ material can be calculated by

$$\epsilon = (0.5 \times h)/(r + 0.5 \times h)$$

where h and r are as seen in Figure 1 above [10].

Chapter 3: Device Fabrication

The actual materials that are to be tested for piezoresistivity and piezoelectricity were fabricated by the Jung Research Group of the University of Central Florida. These novel 2D TMD thin film materials were fabricated using a CVD process of selenization and tellurization of Platinum. These samples were then affixed on PET and were modified to be suitable with the bending tests that the experimental setup for the verification of piezoresistivity and piezoelectricity required.

FABRICATION OF PtSe_2 AND PtTe_2

The PtSe_2 and PtTe_2 samples used were provided and fabricated by the Jung Research Group of the University of Central Florida [11]–[13]. The 2D TMD materials are grown on Kapton using a CVD selenization and tellurization of Platinum (Pt), creating PtSe_2 and PtTe_2 , respectively. A thin film of Pt is deposited using electron beam evaporation. The PtSe_2 and PtTe_2 samples used in the experiments have varying thicknesses of 0.7 nm, 1 nm, 2 nm, 3 nm, and 6 nm. These thicknesses are the Pt thickness before selenization and tellurization, which are controlled by electron beam evaporation. The thin film of Pt is then moved into a quartz tube CVD furnace that has an alumina boat filled with either selenium powder for selenization or tellurium powder for tellurization [13]. The pressure in the quartz tube is then pumped down to 1 mTorr, using a vacuum pump. The inside of the quartz tube is then purged with Argon (Ar) gas to remove impurities before selenization/tellurization. Next, the CVD furnace is set to dwell for 50 minutes at 400 °C while Ar gas flows at 100 sccm [13]. The alumina boat filled with the selenium/tellurium powder is heated to 200 °C as well during the Ar gas flow, bringing the selenium/tellurium to the thin film Pt to grow $\text{PtSe}_2/\text{PtTe}_2$ thin films. After 50 minutes, the furnace is cooled down naturally to room temperature, and the samples

are taken out of the quartz tube CVD furnace to be examined for PtSe₂/PtTe₂ thin film growth.

FABRICATION OF SAMPLES

The PtSe₂ and PtTe₂ samples that were fabricated by Dr. Jung's group are cut into rectangles with a length of 30 mm and a width of 3.5 mm. These cut PtSe₂ and PtTe₂ samples are then placed on PET using double sided tape. The PET that the samples are placed on has a length of 7.25 cm and a width of 1.5 cm. The PET with double-sided tape has a combined height of 0.33 mm. After affixing the PtSe₂/PtTe₂ sample on the PET with double-sided tape, wires are placed on the PtSe₂/PtTe₂ material and a contact between the wire and the material is made using silver conductive epoxy adhesive. After curing the epoxy, a strong electrical connection is made between the PtSe₂/PtTe₂ with the wire. This process is repeated for all the PtSe₂ and PtTe₂ samples, preparing the materials to be tested for piezoresistivity and piezoelectricity. An example of a completed sample can be seen in the figure below.

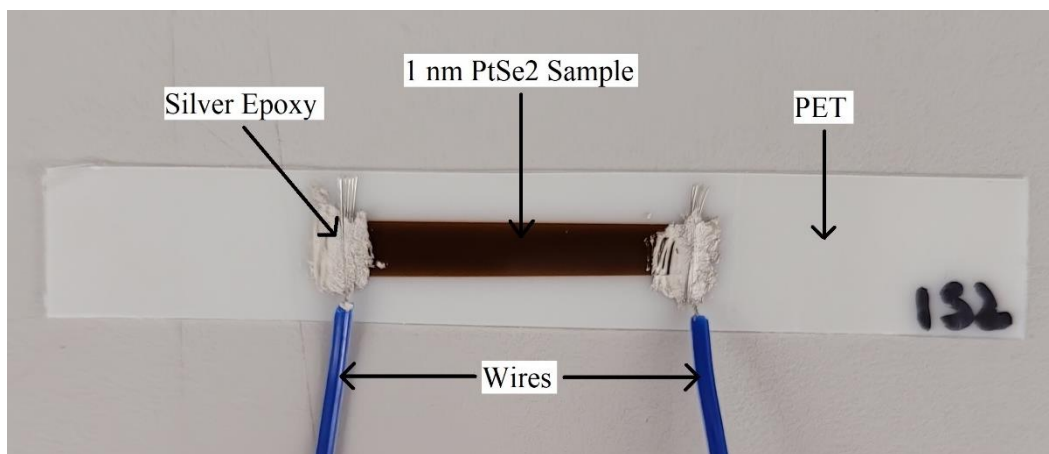


Figure 2: A Completed Sample of 1 nm PtSe₂

Chapter 4: Piezoresistivity

For the experiment setup for piezoresistivity verification in PtSe₂ and PtTe₂, a two-point bending fixture is used to bend the samples, and a Keysight B2902A source measure unit is used to measure the resistance across the sample.

METHOD OF PIEZORESISTIVITY EXPERIMENT

A two-point bending fixture is used to apply strain to the samples as shown in the figure below. The samples are placed in the two-point bending fixture such that there is 0% strain in the sample when the two points of the fixture are 5 cm apart. For all piezoresistivity experiments, the two-point bending fixture is moved such that the two points are 5 cm apart at first then in 0.1 cm increments the two points become 4.5 cm apart and then back to 5 cm in 0.1 cm increments.

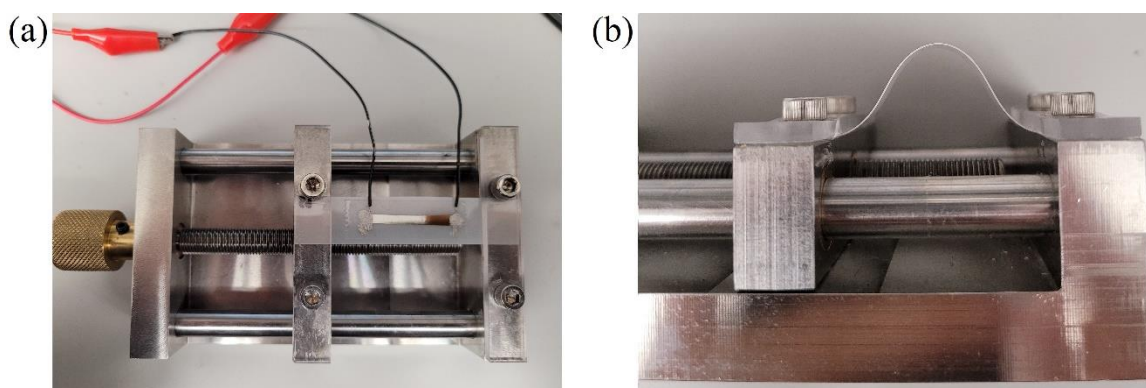


Figure 3: (a) Sample with 0% Strain connected to Source Measure Unit (b) Bending of Sample using Two-Point Bending Fixture

Strain calculations are done for each state of the two-point bending fixture by using a dummy sample with the same fabrication methods and dimensions as other samples. By measuring the thickness of the PET with double-sided tape and the radius of the arc while the sample is bent, the correlation between the length between the two

points in the fixture and the induced strain in the material are calculated. The calculations for the strain in the material are organized in the following table.

Table 1: Strain Calculations for Two-Point Bending Fixture

Length Between Two Points (cm)	Strain (%)
5 cm	0.00%
4.9 cm	0.40%
4.8 cm	0.60%
4.7 cm	0.80%
4.6 cm	0.94%
4.5 cm	1.08%

Each sample of PtSe₂ and PtTe₂ of different thicknesses is placed on the two-point bending fixture and connected to the Keysight B2902A source measure unit. The resistance across the sample is measured while the two-point bending fixture is manipulated in 0.1 cm increments from 5 cm to 4.5 cm and then back to 5 cm. The resistance measurements are then correlated with the corresponding strain due to the two-point bending fixture, allowing for examination of the relationship between strain in the material and the resistance of the material.

PIEZORESISTIVITY EXPERIMENT RESULTS

The first piezoresistivity experiment used 6 nm bare Pt films as a control to compare the PtSe₂ and PtTe₂ samples. 6 nm PtSe₂ and 6 nm PtTe₂ samples were created for the resistance measurements of the first piezoresistivity experiment. These three

different sample types were strained using the two-point bending fixture with the samples facing up as well as facing down, as seen in the figure below. The second piezoresistivity experiment focused on using PtSe₂ and PtTe₂ samples with varying thicknesses of 0.7 nm, 1 nm, 2 nm, and 3 nm. These samples with varying thicknesses were measured while bending them facing up.

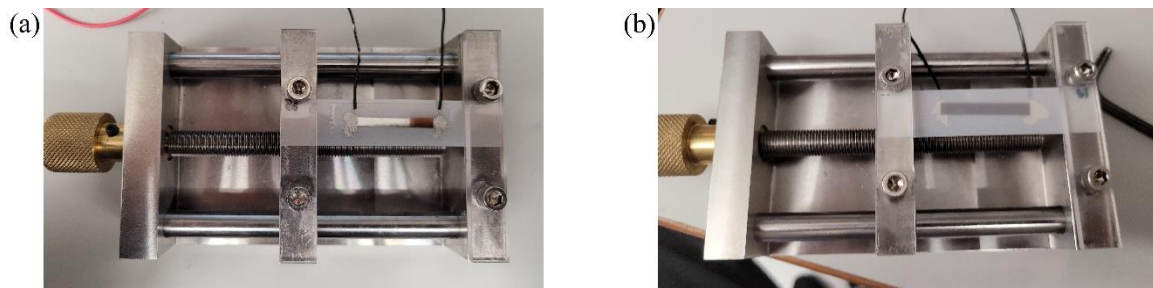


Figure 4: (a) Sample facing up in two-point bending fixture (b) Sample facing down in two-point bending fixture

6 nm Sample Piezoresistivity Experiment Results

Samples of 6 nm Pt, 6 nm PtSe₂, and 6 nm PtTe₂ are bent using the two-point bending fixture. Resistivity measurements are done while bending and recorded. The change in resistance when compared to the resistivity of the material under 0% strain is calculated. The following figures show the plots for the samples, denoting the change in resistance vs. strain in the sample. The black lines are for the samples that were bent while face up, and the red lines are for the samples that were bent while face down. The plots show the average resistance response to strain for multiple measurements for each of the samples.

The following graph shows the average change in resistance for 6 nm Pt samples depending on the strain in the material. For 6 nm Pt, there is a clear linear relationship with the change in resistance and the strain in the sample. When 6 nm Pt is bent face up with a strain of 1.08%, there is a 0.91% change in resistance. When 6 nm Pt is bent face down with a strain of 1.08%, there is a -0.81% change in resistance.

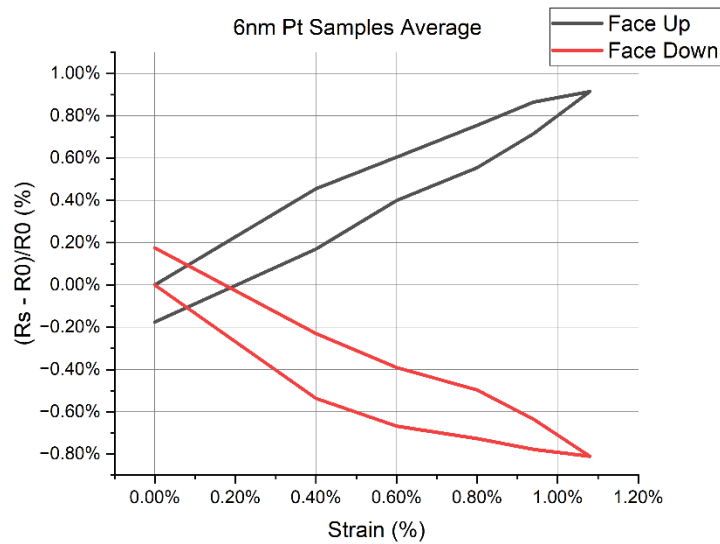


Figure 5: 6 nm Bare Pt Samples, Change in Resistance vs. Strain Measurements

The graph below shows the average change in resistance for 6 nm PtSe₂ samples as the strain in the samples changes. Like the previous 6 nm Pt sample, there is also a clear linear relationship between strain and resistance in the 6 nm PtSe₂ material. For bending of 6 nm PtSe₂ samples while facing up, there is a 3.49% change in resistance when the material is under 1.08% strain. For bending while facing down, there is a negative 3.13% change in resistance when the material is under 1.08% strain.

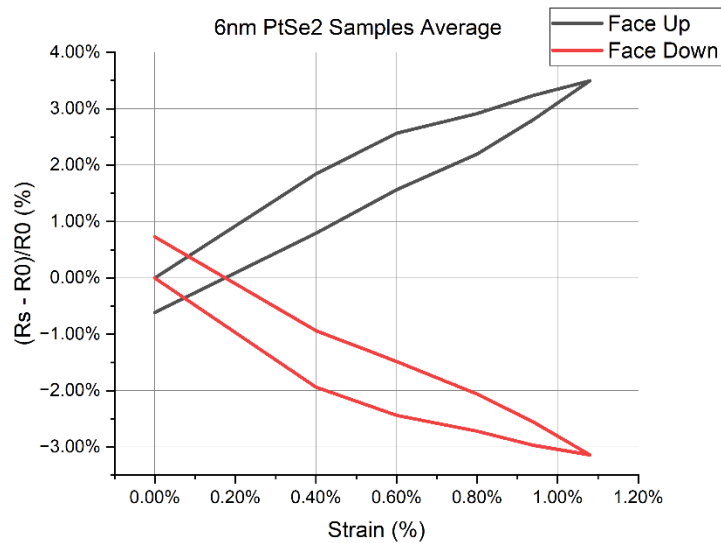


Figure 6: 6 nm PtSe₂ Samples, Change in Resistance vs. Strain Measurements

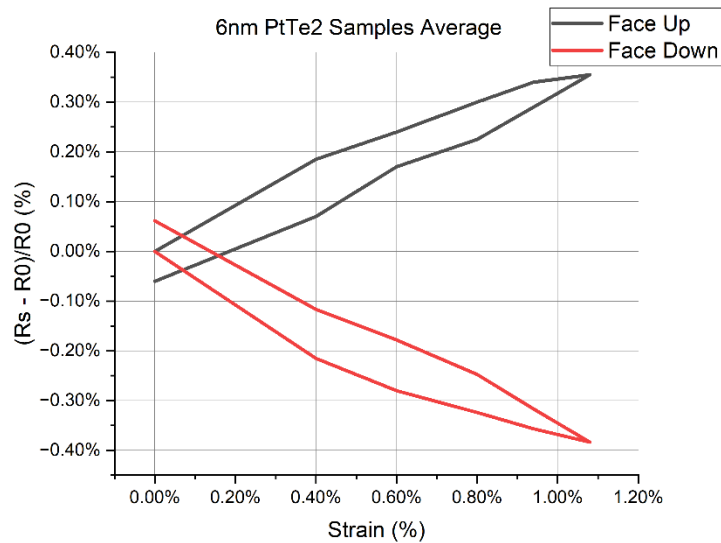


Figure 7: 6 nm PtTe₂ Samples, Change in Resistance vs. Strain Measurements

The graph above shows the change in resistance versus strain in the material for PtTe₂ materials. The average of all measurements for 6 nm PtTe₂ face up and face down bending are shown above. The resistance in 6 nm PtTe₂ samples is shown to have a linear relationship with the strain in the material. For face up PtTe₂ samples, the average change in resistance due to 1.08% of strain is 0.35%, and for face down PtTe₂ samples the average change in resistance due to 1.08% of strain is -0.38%.

For the three graphs previously shown in Figures 5-7, the lines have a “V” shape due to the resistance of the samples not changing exactly at the same rate when the strain in the samples rises from 0% to 1% and when the strain in the samples lowers from 1% to 0%. For all the 6 nm samples, when they are under tensile strain, there is a positive relationship with strain in the material and resistance of the material while compressive strain on the samples results in a negative relationship with strain and resistance of the material. The magnitude of the change in resistance due to equal magnitudes of either tensile or compressive strain is the same, thus the samples do not have to be strained in a certain orientation for a stronger piezoresistive response.

In a final analysis, all the resistances of the samples have a linear relationship with the strain induced in the samples. The 6 nm PtSe₂ samples have the strongest piezoresistivity response with 3.5% change in resistance with around a 1% strain in the samples. 6 nm PtSe₂ seems to be piezoresistive with a significant change in resistance due to straining the thin film material. Surprisingly, the 6 nm Pt samples have a higher change in resistance at around 0.85% than the change in resistance in 6 nm PtTe₂ samples at approximately 0.35% when both types of samples are under 1% strain. A less than 1% change in resistance due to 1% strain in the material is negligible, signifying the lack of a piezoresistive property in the material, which Pt is known to not be piezoresistive.

Therefore, with a change in resistance of only 0.35% due to 1% strain, 6 nm PtTe₂ is concluded to not have the piezoresistive property.

Varying Thickness Piezoresistivity Experiment Results

With the findings of the previous experiment, the samples of PtSe₂ and PtTe₂ of varying thicknesses were bent only facing up. The samples were measured using the exact same methods used in the 6 nm sample experiments, where the applied strain on the samples ramped up to 1% from 0% and back down to 0% from 1%. The following graphs show the average change in resistance for PtSe₂ and PtTe₂ samples with 6 nm, 3 nm, and 2 nm thicknesses.

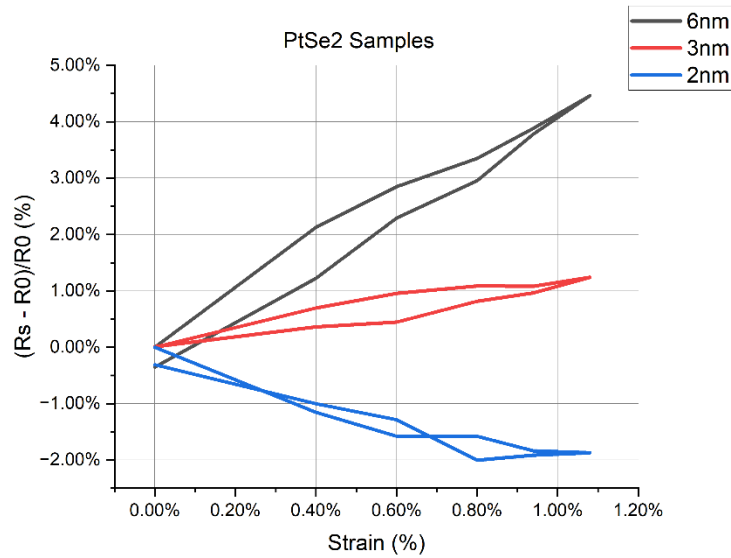


Figure 8: Piezoresistivity of PtSe₂ Samples with Varying Thicknesses

The graph above shows the average change in resistance due to strain for PtSe₂ samples with thicknesses of 6 nm, 3 nm, and 2 nm. As the thickness of PtSe₂ gets smaller, the linear relationship between the change in resistance and the strain in the

sample becomes smaller and eventually negative. When the PtSe₂ sample is of the thickness 2 nm, there is a clear negative linear relationship between the change in resistance in the material and the strain in the material. With the magnitudes of the change in resistance considered, the 6 nm PtSe₂ samples have the greatest value in change in resistance at 4.46% with a 1% strain in the material. The 2 nm PtSe₂ samples have a 1.87% change in resistance due to a 1% strain, and the 3 nm PtSe₂ samples have a 1.24% change in resistance.

The following graph in Figure 9, shows the average change in resistance for 6 nm, 3 nm, and 2 nm PtTe₂ samples. Unlike PtSe₂ as the PtTe₂ thickness lowers, the change in resistance due to straining samples increases. The 6 nm PtTe₂ experiments showed that PtTe₂ had a very low piezoresistivity response, but the following data shows that 2 nm PtTe₂ samples under 1% strain have a change in resistance of 3.4%, which is comparable to the piezoresistivity response of 6 nm PtSe₂.

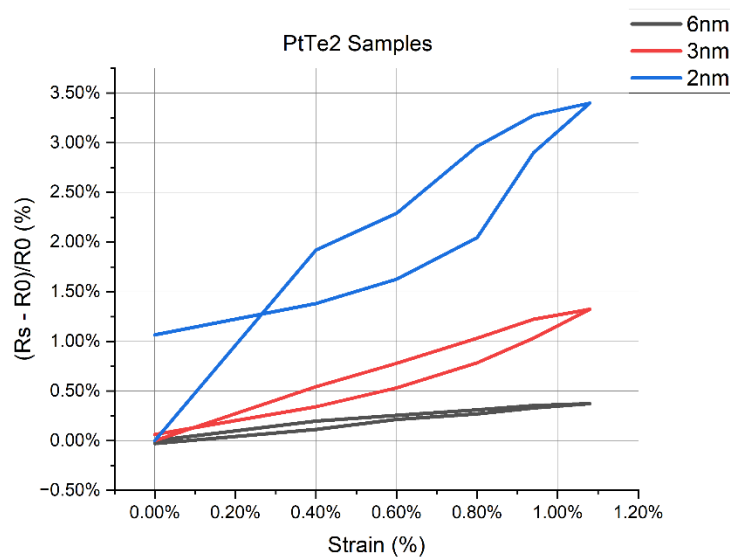


Figure 9: Piezoresistivity of PtTe₂ Samples with Varying Thicknesses

The same measurements were done with 1 nm and 0.7 nm samples of PtSe₂ and PtTe₂, but they were not included in the previous graphs due to their volatility and measurement inconsistencies. 1 nm and 0.7 nm samples were too volatile in resistance measurements for both PtSe₂ and PtTe₂ samples.

Ultimately as the thickness lowers from 6 nm for PtSe₂ samples, the piezoresistivity response lowers and is not as high as that of 6 nm PtSe₂ samples. Lower thickness PtSe₂ samples also shift to a negative response in change in resistance due to strain in the material. For PtTe₂ samples as the thickness lowers, the change in resistance due to strain increases and suggests that 2 nm PtTe₂ samples are piezoresistive.

Chapter 5: Piezoelectricity

For the experimental setup for piezoelectricity verification in PtSe₂ and PtTe₂, a Keysight MSO-X 2024A digital oscilloscope is used to measure the voltage response of the sample under periodic tensile strain.

METHOD OF PIEZOELECTRICITY EXPERIMENT

Due to equipment limitations, a manual method is used to achieve a periodic tensile strain on the samples while measuring their voltage response with a digital oscilloscope. Periodic tensile strain is applied to the samples by hand at 2 Hz using a metronome. The voltage response is then recorded using the oscilloscope in order to observe a potential piezoelectric response in the sample tested.

6 nm Pt samples and 28 μm polyvinylidene fluoride (PVDF) samples were used as a control in the experiment. 6 nm bare Pt is known to not be piezoelectric while the 28 μm PVDF film is known to be piezoelectric. These samples' voltage responses were used as a reference to verify the piezoelectric property in the tested samples. The samples that were tested are PtSe₂ and PtTe₂ with thicknesses of 6 nm, 3 nm, 2 nm, 1 nm, and 0.7 nm.

PIEZOELECTRICITY EXPERIMENT RESULTS

The voltage response of 28 μm PVDF under 2 Hz excitation is shown in Figure 10. The graph shows a voltage pulse being generated at around ~2Hz with a peak-to-peak voltage of 32.1 mV. This is the voltage response of a material with the piezoelectric property that will be referred to. In Figure 11 below, the voltage response of 6 nm bare Pt is shown. Platinum is known to be not piezoelectric and the graph in Figure 11 shows noise with no clear voltage pulse present.

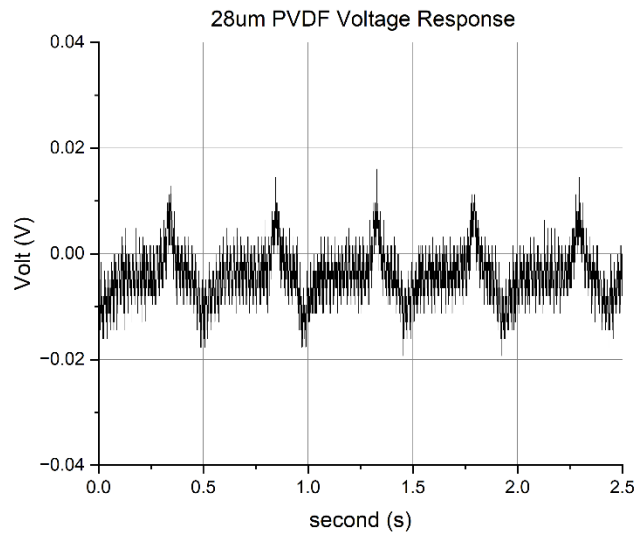


Figure 10: 28 μm PVDF Voltage Response

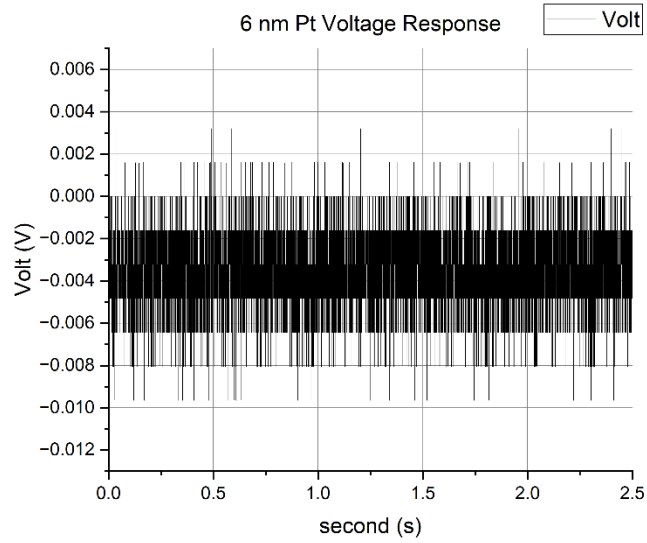


Figure 11: 6 nm Pt Voltage Response

The described methods for piezoelectricity measurements are done on PtSe₂ samples with thicknesses of 6 nm, 3 nm, 2 nm, 1 nm, and 0.7 nm. The findings from this experiment are shown in the graphs in Figure 12 below. Not shown above are the voltage responses of the 6 nm, 3 nm, and 2 nm PtSe₂ samples, which consists of a noisy signal with no clear voltage pulse similar to the voltage response of the non-piezoelectric 6 nm Pt samples. The 1 nm and 0.7 nm PtSe₂ samples have a profound voltage pulse present at around 2 Hz. The 1 nm PtSe₂ samples have a voltage pulse response with a peak-to-peak voltage of 52.9 mV, and the 0.7 nm PtSe₂ samples' voltage response has a 36.9 mV peak-to-peak voltage.

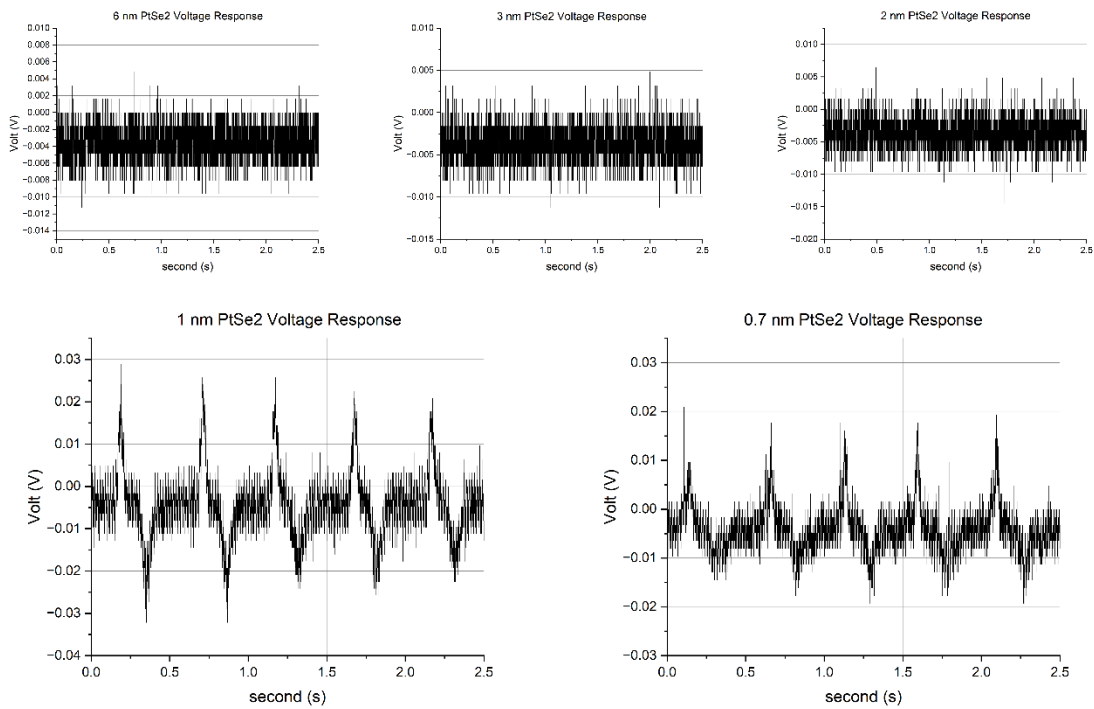


Figure 12: Varying Thickness PtSe₂ Voltage Response

The graphs in Figure 13 below, show the different voltage responses for PtTe₂ samples with thicknesses of 6 nm, 3 nm, 2 nm, 1 nm, and 0.7 nm. These samples' voltage response is measured while they are under periodic tensile strain at 2 Hz. All the voltage responses for all thicknesses of PtTe₂ samples show no clear voltage pulse like that of the known piezoelectric material PVDF in Figure 10. The voltage response of the PtTe₂ samples more resembles the noise signal of the 6 nm Pt sample that is not piezoelectric.

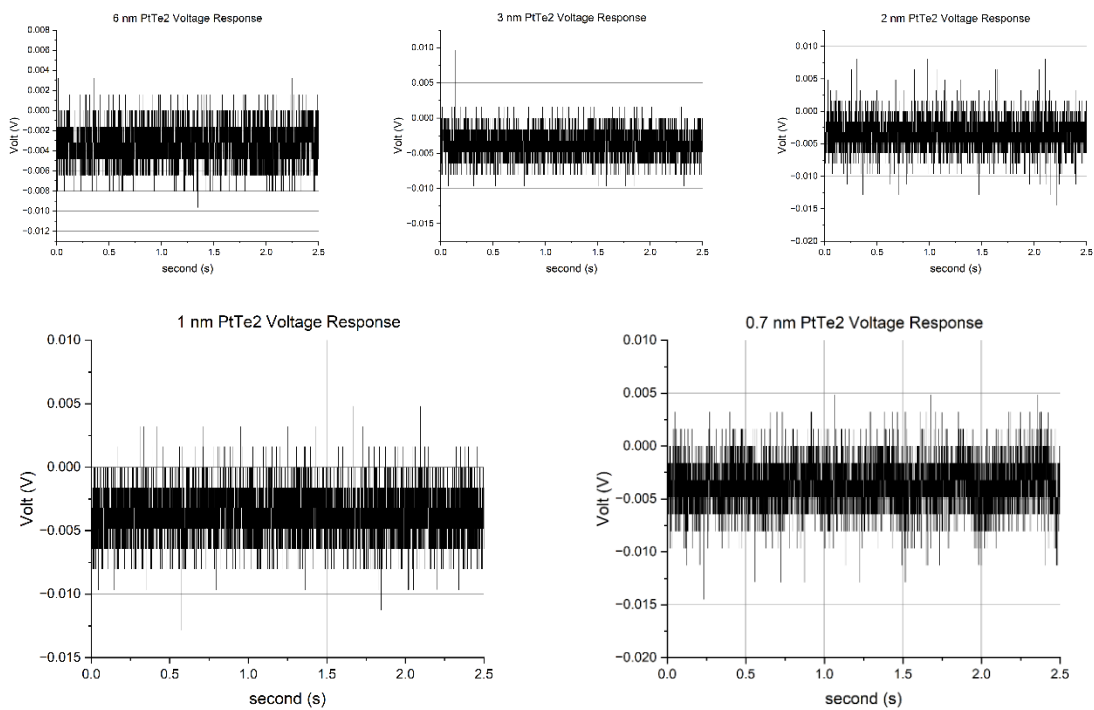


Figure 13: Varying Thickness PtTe₂ Voltage Response

Chapter 7: Conclusions and Future Work

CONCLUSIONS

This report presents experiments and results on the verification of piezoresistivity and piezoelectricity in PtSe₂ and PtTe₂ thin film materials. For the piezoresistivity experiments, tensile and compressive strain was applied to the samples while measuring the resistance. From these experiments, 6 nm PtSe₂ was found to be strongly piezoresistive with a 3.5-4.5% change in resistance while under 1% strain, and thinner PtSe₂ samples were found to be not as piezoresistive as the 6 nm PtSe₂ samples. 6 nm PtTe₂ was found to be not piezoresistive with a 0.35-0.4% change in resistance while under 1% strain, showing close to no relationship between strain and resistance in the material. The thinner PtTe₂ samples of 3 nm and 2 nm increased the change in resistance due to strain with 2 nm PtTe₂ samples being comparable to 6 nm PtSe₂ samples with a 3.4% change in resistance while under 1% strain. In the final analysis, 6 nm PtSe₂ and 2 nm PtTe₂ show the strongest piezoresistive response for PtSe₂ and PtTe₂, respectively.

For the piezoelectricity experiments, periodic tensile strain was applied to samples at a frequency of 2 Hz. 28 μm PVDF film and 6 nm bare Pt film were used as a control, where PVDF film is known to be piezoelectric, and Platinum is known to be not piezoelectric. The samples that were tested were PtSe₂ and PtTe₂ of different thicknesses of 6 nm, 3 nm, 2 nm, 1 nm, and 0.7 nm. For the PtSe₂ piezoelectricity experiments, the samples with the thickness of 1 nm and 0.7 nm had significant piezoelectric voltage responses like that of the PVDF film voltage responses. 1 nm and 0.7 nm PtSe₂ can be concluded to be piezoelectric while the thicker PtSe₂ samples are concluded to be not piezoelectric. For the PtTe₂ piezoelectricity experiments, all thicknesses of PtTe₂ showed

no piezoelectric response like that of the 6 nm bare Pt samples. It is concluded that PtTe₂ is not piezoelectric.

FUTURE WORK

Further work can be done with the piezoelectricity verification experiment by acquiring and setting up a bending structure to apply a periodic and dynamic tensile strain on the PtSe₂ and PtTe₂ samples. Such an experimental set up can be done with the samples fixed on an aluminum plate. The aluminum plate is then attached to a modal shaker that excites the aluminum plate sinusoidally at different frequencies. This setup will allow for testing of voltage responses at different frequency excitations of the samples.

After further experimentation on the verification of piezoresistivity and piezoelectricity in PtSe₂ and PtTe₂, the materials can be used in a multitude of applications. By utilizing their piezoresistive properties, PtSe₂ and PtTe₂ can be used in applications for bioelectronic pressure sensors and MEMS devices. The piezoelectric property of PtSe₂ can be utilized to make PtSe₂ energy harvesters in bioelectronics, improving device power supply solutions, such as batteries.

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