Developing A Low-cost Electric Hysteresis Measurement Device and Studying Hysteresis Effects of Thickness in PVDF

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Abstract

Poly-vinylidene fluoride or PVDF is a unique polymer due to its rare room-temperature piezoelectric, ferroelectric, and pyroelectric properties [1]. PVDF is a semi-crystalline polymer with a structure consisting of monomer units, (CH_2CF_2). With this machine, we used the β-crystal phases, due to the fact that all the dipoles are aligned inward giving the crystal a zero-net charge; which gives the β-phase a stronger ferroelectric and piezoelectric properties over the other phases. [1] The Raspberry Pi and LabQuest Machine or RLM, is a machine that we developed to be able to send an electrical pulse, in the form of a voltage, to a sample material, which in turn creates an electric field through the sample. Along with producing the electrical pulse, the RLM is designed to measure the voltage produced and charge across the sample material, using the voltage meter and the charge sensor. With the knowledge of the area and thickness of the test sample, the RLM is able to determine the electric polarization of the material and electrical field applied to the sample and graph them as a polarization vs. electric field curve (P/E). To test the RLM, ferroelectric PVDF was used, meaning, if a current is applied through the PVDF, alterations in the polarization and electrical field of the material is detectable. Once the RLM was found to create and measure the signal, the coercivity of the PVDF could be determined. The RLM had a hysteresis curve that was more of a box shape, but coercivity values could still be obtained. With coercivity measured, samples were created to measure the effect of the thickness of the PVDF layers had on the coercivity. These measurements have been performed before, and this experiment was used to show that the pattern of coercivity versus thickness as obtained by the RLM is in agreement with the pattern obtained by more expensive, commercially purchased devices.

Sample Preparation

- PVDF was deposited using Langmuir-Schaefer film fabrication method, resulting in atomic layer deposition of 1.75 nm thick layers of PVDF [2,3]
- The atomic layers of PVDF crystal were in b phase so the crystal structure had zero net polarization, but maintained ferroelectric properties
- Conductive silver paste and gold wires were connected to the sample in order to apply an electric field to the sample

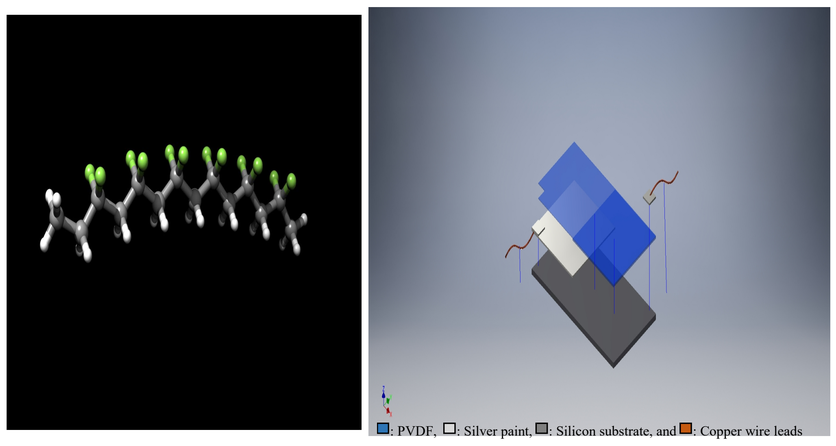


Figure 1: (a) The β form of the molecular chain of PVDF and (b) test sample preparation

Raspberry Pi and Labquest Machine Composition

- The Raspberry Pi and Labquest Machine (RLM) is made up of four major parts
  1. Core of the machine is a raspberry pi microcomputer.
  2. Raspberry pi hat, a DC stepper motor and driver. It is connected to the raspberry pi using GPIO, and uses pulse width modulation to send a voltage signal
  3. Vernier Voltage sensor
  4. Vernier Charge sensor

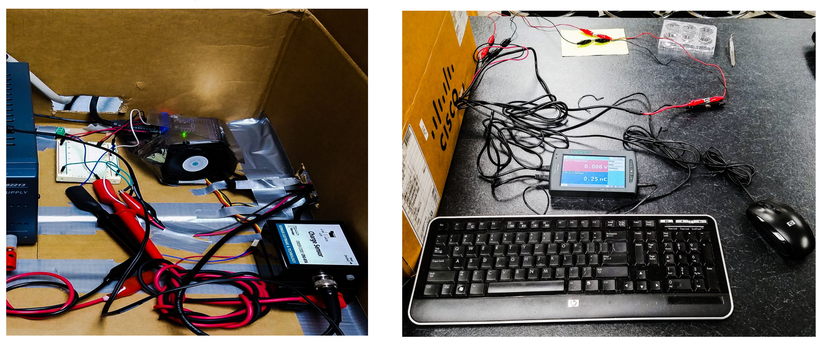


Figure 2: (a) Raspberry Pi and Vernier Sensors and (b) Labquest and sample connection

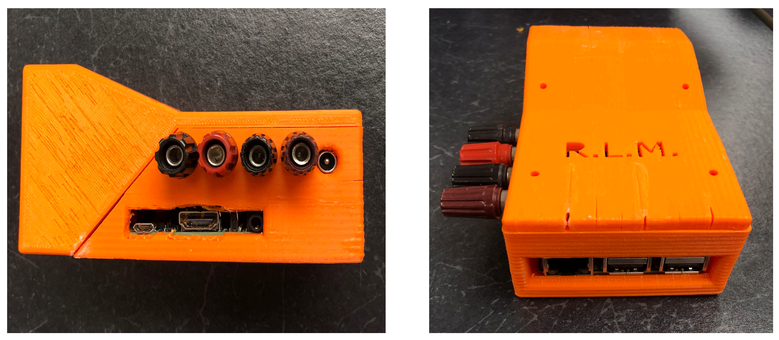


Figure 3: (a) Raspberry Pi machine with case

Theory

- By knowing the charge (Q) across the PVDF, and the area (A) of the substrate. The polarization (P) of the material can be found by using the formula $P = \frac{Q}{A}$ [4]. When the voltage (V) across the PVDF is known as well as the distances (d) that the voltage will travel, which is the thickness of the layer of the PVDF on the substrate, electric field (E) can be found by the formula $E = \frac{E}{d}$ [4].

Results

- The RLM produced a voltage signal that varied with time, in order to produce an applied electric field through the PVDF sample

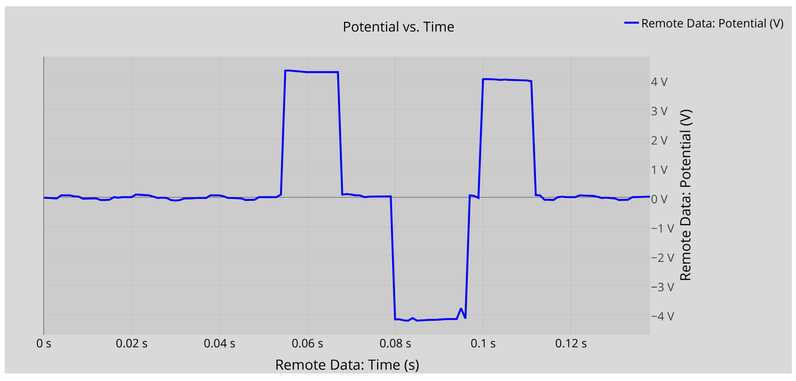


Figure 4: Electric Signal Generated by RLM

- The results from the RLM when testing the sample of PVDF. Shows that the dipoles of the PVDF in β–crystal form orientated to match the current flow.

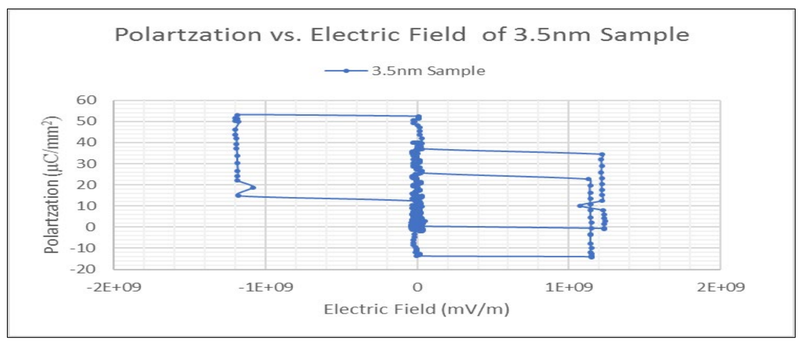
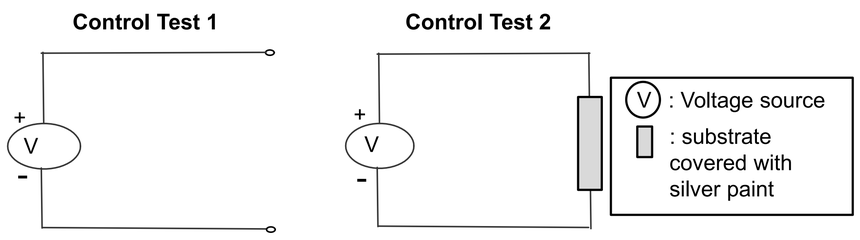
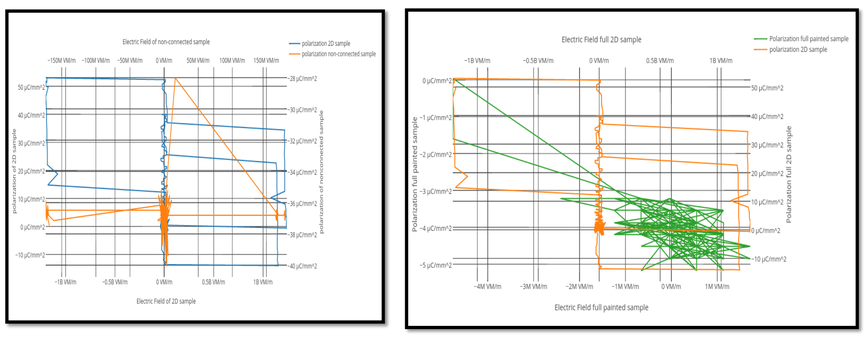


Figure 5: Polarization vs. Electric field curve from the RLM when testing a sample of 3.5nm *thick* β*- phase crystal structure of PVDF.*

Control Testing

- Two control test were performed to ensure that the RLM was detecting the changes in the polarization in the PVDF crystal. The first control test was performed with the with the RLM not connected to a sample of PVDF. As a result the RLM can be modeled an open circuit. The second control test that was conducted, the RLM was connected to a sample of the silicon substrate that was fully coated with the silver conductive paint. In this control test the RLM can be modeled as a completed circuit.





Sample Testing

- In testing this hypothesis, five test sample were created ranging from 6ML to 100ML, with each monolayer being 1.75 nm thick. Each test sample was tested by the RLM five times to ensure the most accurate sampling of each sample. Out of the five tests per sample, similar PE graphs where averaged to obtain a single PE graph that represented that particular test sample as shown in figure 6.

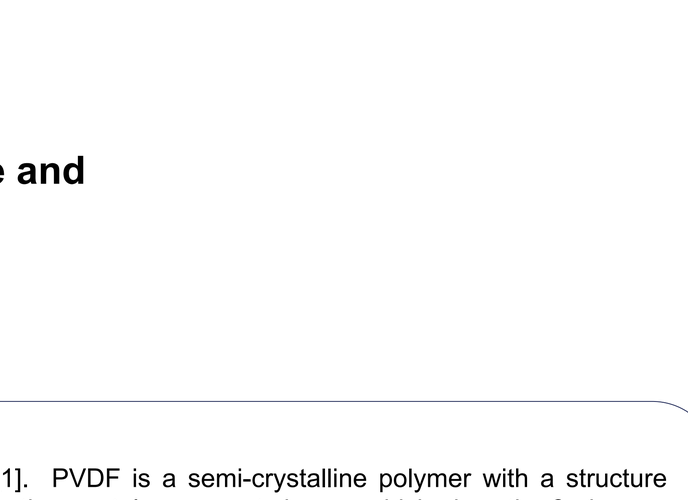
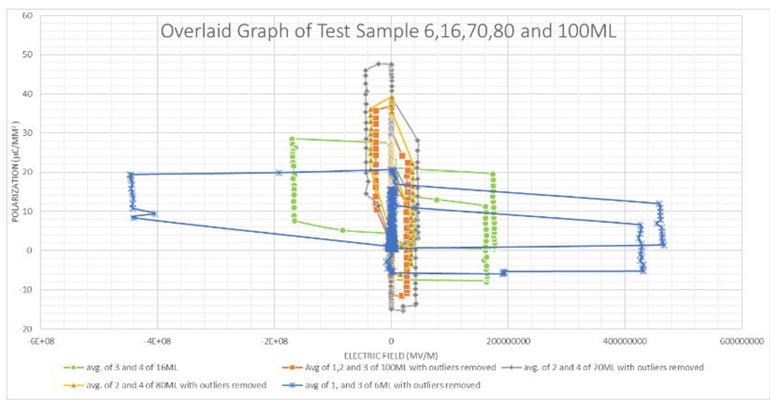


Figure 6: The PE graph of all five test runs of the 28 nm thick sample.

- When an average PE graph was obtained for each of the test samples. They were overlaid on the same PE graph for comparison, this can be seen in figure 5. By overlaying the five PE graphs a trend of smaller coercivity becomes noticeable.

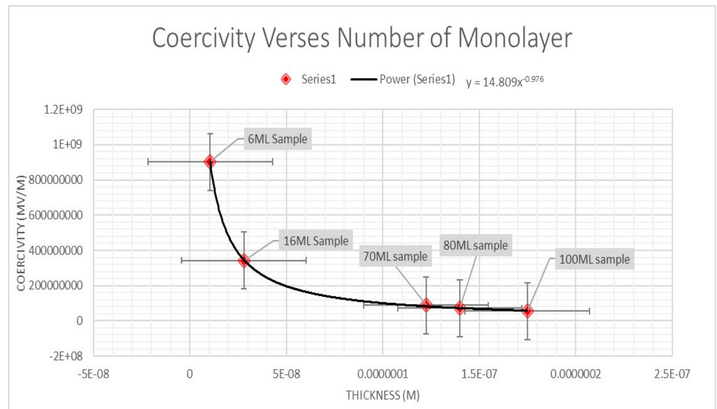


- Noting that there is a definite variation in width or coercivity of the different PE graphs of the test samples, equation 1 was used to calculate the width of each test samples' average PE graph.

$$Coercvitiy = \left[\frac{\sum nE_{max}}{n} - \frac{\sum nE_{min}}{n}\right]$$

Equation 1: The formula for calculating the coercivity of the PE graph, nE: number of data points for a given value of electric field, n: number of points summed.

- When all of the coercivity of the test samples were calculated, they where plotted by the number of monolayers verses coercivity of that sample



- This graph matches previous work for thickness dependence of coercivity in PVDF films [5]
- This proves that the RLM is able to be used to test samples for ferroelectricity in future work, such as combining ferroelectric nanomaterials

Conclusions

- The RLM has the ability to detect the change in the polarization and electric fields within the PVDF test sample. This is accomplished by producing an electrical pulse through the PVDF that is in the β-crystalline phase and then measuring the electrical response of the crystals. By testing the RLM with two kinds of control test with samples that was fully coated with conductive silver paint as well as not coated with conductive paint. The RLM was able to distinguished between the control tests and that of the test sample of the PVDF. Thus making the RLM an effective low cost polarization and electric field detector. By changing the thickness of the PVDF film, the RLM was able to detect changes in the coercivity due the film thickness.

References

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