Poling of PVDF matrix composites for integrated structural load sensing

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ABSTRACT

The purpose of this study is to create and evaluate a smart composite structure that can be used for integrated load sensing and structural health monitoring. In this structure, PVDF films are used as the matrix material instead of epoxy resin or other thermoplastics. The reinforcements are two layers of carbon fiber with one layer of Kevlar separating them. Due to the electrical conductivity properties of carbon fiber and the dielectric effect of Kevlar, the structure acts as a capacitor. Furthermore, the piezoelectric properties of the PVDF matrix can be used to monitor the response of the structure under applied loads. In order to exploit the piezoelectric properties of PVDF, the PVDF material must be polarized to align the dipole moments of its crystalline structure. The optimal condition for poling the structure was found by performing a 2^3 factorial design of experiment (DoE). The factors that were studied in DoE were temperature, voltage, and duration of poling. Finally, the response of the poled structure was monitored by exposing the samples to an applied load.

Keywords: Smart composite structures, carbon fiber reinforced composites, structural health monitoring, PVDF films, piezoelectricity

1. INTRODUCTION

Carbon fiber reinforced composite materials are being widely used in different industries where high strength, high stiffness, low weight, and high conductivity are required. Some application areas include aerospace and nuclear engineering, wind turbine blades, and automobile bodies[1]. For the safety and reliability of these designs, the integrity of the structure needs to be monitored through structural health monitoring[2]. Structural health monitoring is the process by which a structure is inspected for damage during its service life. This monitoring can consist of periodic inspections that are manually conducted while the equipment is taken offline or it can be continuous through the use of sensors. The limitation of periodic inspection or sensors is that only the locations that are checked or equipped with sensors are evaluated for damage. The technology that will be explored by this study will enable integrated structural stress and strain sensing through the creation of smart composite structures. These smart composite structures will be simultaneously functional and will also behave as an integrated sensor.

The proposed smart composite structure is enabled by taking advantage of the electrical conductivity inherent in carbon fiber fabrics that are used for carbon fiber reinforced polymers. This conductivity property will be used to form the electrodes of the smart composite structure. Typically, the polymers used for carbon fiber reinforced polymer composites are either an epoxy resin or a thermal plastic[3]. For thermal plastic based fiber reinforced composites, polyether ether ketone (PEEK) is typically used. If polyvinylidene difluoride (PVDF) thermal plastic is used as the polymer for the fiber reinforced composite, the piezoelectric properties of PVDF can be harnessed to sense strains and stresses applied to the structure. This integrated sensing capability enables structural health monitoring without the need for taking the structure out of service for manual inspection or the requirement of embedding sensors at a limited number of locations within the structure.

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2. METHODOLOGY

This study consists of three main steps: 1. sample preparation, 2. poling experiment, and 3. tensile test. The required procedures for each step are described in the following sections.

2.1 Sample Preparation

For making the samples, two layers of carbon fiber were used to form the electrodes of the piezoelectric sensors. In order to prevent the two carbon fiber layers from shorting to each other, a Kevlar layer is placed between them as a dielectric barrier. Kevlar fibers have mechanical properties near those of carbon fiber materials[4], so there is little sacrifice to the mechanical properties of the overall structure. In order to achieve high electric field between the two carbon fiber layers when the structure is under stress, it is desirable to make the Kevlar layer as thin as possible, which also decreases the Kevlar layer's overall impact on the mechanical properties of the composite structure. Two layers of 80 um thick PVDF films were used in between each of the fabric layers. Figure 1 and Figure 2 illustrate the structure of the samples.

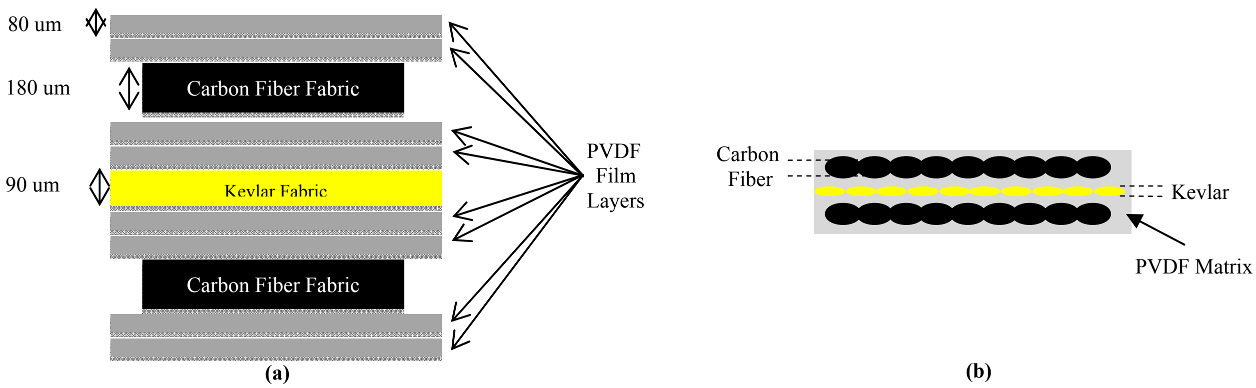

Figure 1. Schematic of the smart composite structure and material thicknesses (a) the stack-up of reinforcement fabrics and PVDF film layers before melting, (b) after melting.

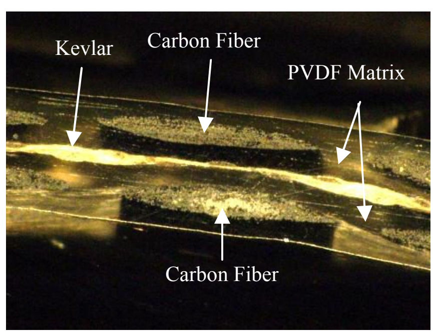

Figure 2. Cross section of the prepared samples.

The samples were made by applying pressure and heating the PVDF film to its melting temperature using an oven (see Figure 3). The duration of heating was 4 hours and the temperature of oven was set to 200°C. The capacitance of the 100 mm × 80 mm samples was measured to be between 800 and 900 pF.

For performing the tensile test, the samples must be placed between two grips. Using the samples shown in Figure 3(b) is not appropriate for this purpose because the grips will cause a compression on the sample that will affect the results of the tensile test. In order to prevent this error, the above sample structure was further extended by adding two layers of Kevlar on each side of the sample along with two layers of PVDF on top and bottom as shown in Figure 3. With this new structure, the attached layers of Kevlar will be placed between the grips so no pressure is applied to the carbon fiber electrodes during the tensile test.

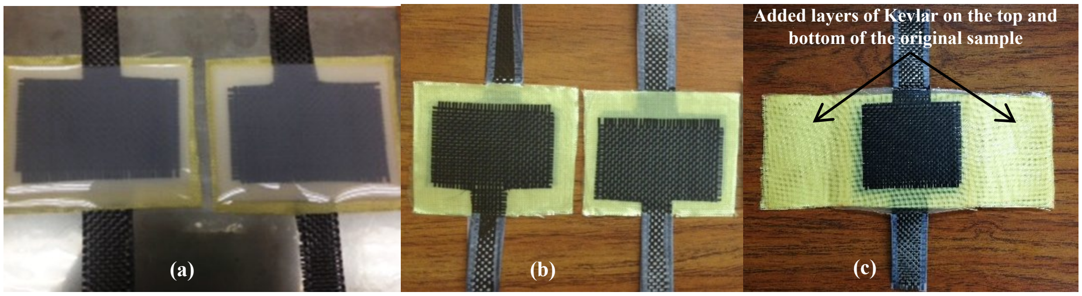

Figure 3. Prepared samples (a) original samples before melting the PVDF films, (b) original samples after melting the PVDF films, (c) extended sample for tensile testing.

2.2 Poling

In order for the smart composite structure to generate responses due to an applied load, the PVDF material must be polarized to align the dipole moments of its crystalline structure. The crystalline structure of the PVDF film will determine the polarization properties of the film. There are four possible crystalline forms of the PVDF polymer: the alpha, beta, gamma, and delta phases[5]. Only the beta, gamma, and delta phases can be polarized. However, the alpha phase can also be polarized by applying a large electric field, which will convert the alpha phase to the delta phase[5]. The processing conditions of the PVDF film determine the dominant crystalline phase. When PVDF film layers in the samples are melted and solidified, the dominate phase is the alpha crystalline phase[6]. Since the alpha phase is the dominate phase upon solidification, an electric field must be applied to convert the alpha phase to the delta phase and to align the dipoles. Existing research shows that an electric field of around 125 MV/m is required to fully polarize a predominately alpha phase PVDF film[7]. However, a higher piezoelectric coefficient can be attained by polarizing a beta phase film since the beta crystal phase has a greater dipole moment than the delta phase[8]. One of the most commonly means mentioned in the literature to achieve a predominately beta phase PVDF film is to stretch the film mechanically to many times its original length[9]. A second method for transition from an alpha phase film is to apply a very high electric field on the order of 500 MV/m[7]. A third method mentioned in the literature to achieve a predominately beta phase PVDF film is to cure the film under high pressure[10]. Finally, the copolymers of PVDF containing trifluoroethylene (TrFE) or tetrafluoroethylene (TFE) can be added to the film to encourage the formation of the beta phase[11].

For the purpose of this study, the most common polarization method of stretching is not applicable because the carbon fiber and Kevlar reinforcements are very stiff and prevent strain greater than a few percent. Moreover, previous works have required very high fields to polarize a predominately alpha phase film. However, in this study due to the inevitable imperfections of the composite structure, high fields cannot be applied because it will cause dielectric breakdown. Consequently, this study will focus on polarizing the PVDF films by applying relatively lower electric fields for a specific duration of time while heating the samples. In order to find the optimal poling condition, a factorial design of experiment was performed that will be discussed in details in Section 3.

In addition, a cyclical electric field was applied to the samples in order to characterize their ferroelectric properties. For this purpose, a modified Sawyer-Tower circuit[12] (Figure 4) was used for quantifying the polarization of samples. This circuit includes Zener diodes to protect ADC equipment from sample shorting. A 0.2 Hz triangle wave was applied as the voltage excitation. The hysteresis plot (P-E loop) was obtained using LabVIEW software. This plot represents the polarization or the charge developed in the samples due to the applied electrical field at a specific frequency[13].

It is worth pointing out that the charge developed on the sense capacitor in Figure 4 can be calculated using equation (1):

$$Q(t) = C_{sense}V = \int_0^t i\,dt \tag{1}$$

where i is the current generated by the response of the samples to the cyclical applied voltage and it can be formulated as follows[14]:

$$i = \frac{C_{sample} dV}{dt} + \frac{AdP}{dt} + \frac{V}{R_{sample}} \tag{2}$$

In equation (2), C is the sample's capacitance, A is the area, P is the polarization, t is time, V is the applied voltage, and R is the sample's resistance. In other words, the three terms in equation (2) represent the response of the sample due to its capacitance, ferroelectricity, and resistance, respectively.

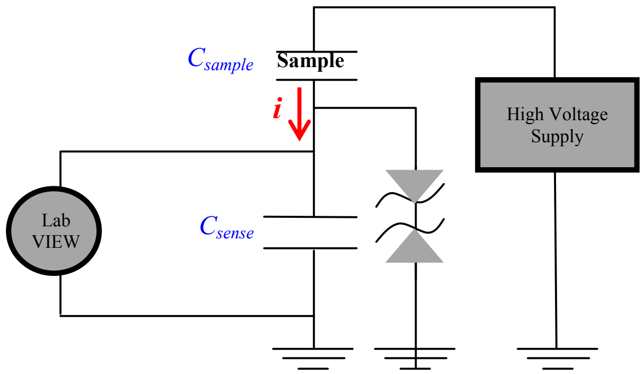

Figure 4. Schematic of the circuit used for generating hysteresis plots (P-E loops).

In order to generate the hysteresis loop, the second term is desired. Consequently, in order to determine the polarization, the capacitive and resistive responses should be compensated for. However, the capacitive response is in phase with the polarization and it does not affect the measurement of the remnant polarization. On the other hand, the resistive response is not in the same phase with polarization and has a 90 degrees phase lag. Consequently, in order to compensate for this, the resistance of the samples was measured and the resistance compensation was performed in the LabVIEW program by subtracting the integral of the third term from the sample's response.

2.3 Tensile test

After polarization, the response of the samples to applied tensile loads was observed using an MTS material testing system. A cyclic force with the amplitude of 130 N was applied to the samples at the frequency of 1 Hz. The charge generated in the sample due to the applied tension will be extracted as the output. The setting used for the tensile test is shown in Figure 5 and Figure 6.

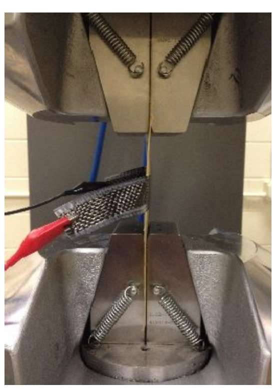

Figure 5. Experiment setting for tensile test.

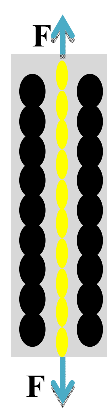

Figure 6. Schematic of the applied load to the samples at the tensile test.

3. DESIGN OF EXPERIMENT

3.1 Experimental procedure

In order to find the optimal condition for poling the smart composite structure and to also investigate the effects of different factors on sample's polarization, a 2^3 full factorial DoE method was used. The three factors that were studied in the DoE were temperature, voltage, and duration of poling. These factors and their levels are shown in Table 1.

Table 1. Design of Experiment factors and their levels.

Factor	*Low Level*	*High Level*
Temperature	50 °C	150 °C
Voltage	1000 V	2000 V
Duration of Poling	5 min.	20 min.

In each of the runs, a constant voltage was applied to the sample for a specific period of time while the sample was placed on a hot plate at a specified temperature. After the poling step, the response of the samples was monitored in the tensile test and the ratio of the charge developed in the sample to the applied force was calculated (Charge/Force or C/F). For the reliability of the results, each of the experiments was replicated two times resulting in 16 runs. The results obtained from the DoE were analyzed using Minitab 16 software.

3.2 Main effects

The main effect plots depict the effect of the change in each of the factors on the response. The main effect plots of temperature, voltage, and duration of poling are shown in Figure 7.

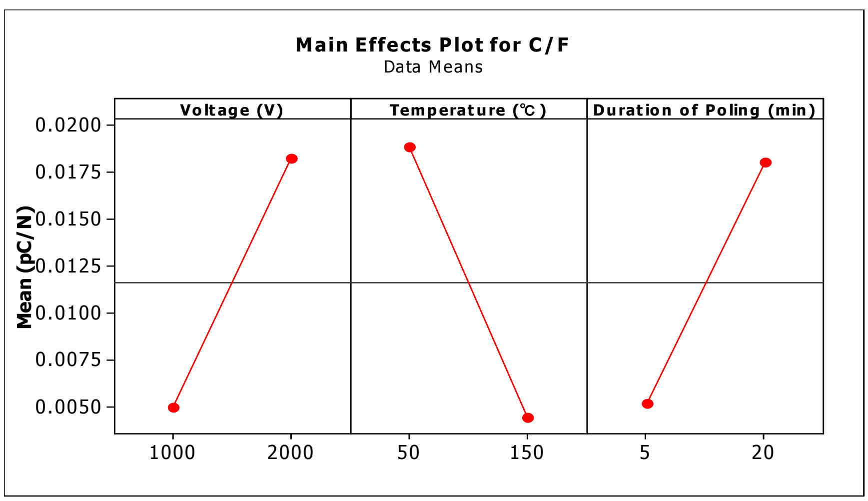

Figure 7. Main effect plots for C/F.

As it can be seen in Figure 7, changing voltage and duration of poling from the low level to their high level will cause an increase on the response of the sample. On the other hand, changing the temperature from low level to high will decrease the response.

3.3 Interaction plot

Figure 8 illustrates the interactions between each pair of the factors. Also, for each pair, it shows the sensitivity of the response to the changes of one factor at a specified level of the other. The fact that each pair of lines has different slopes signifies that all the factors have significant interactions. According to the voltage and temperature interaction plot, the response has a higher value at 50°C than 150°C for both levels of voltage. Also, the response is more sensitive to the changes of temperature at the high level of voltage. The voltage and duration of poling interaction plot suggests that the response for poling at the duration of 20 minutes is higher than 5 minutes for both levels of voltage and it is more sensitive to the changes of duration of poling at the high level of voltage. Finally, the temperature and duration of poling interaction plot shows that poling the samples for a longer duration (20 minutes) causes a higher response than shorter duration (5 minutes) for both levels of temperature. In addition, the response is more sensitive to the changes in the duration of poling at the low level of temperature.

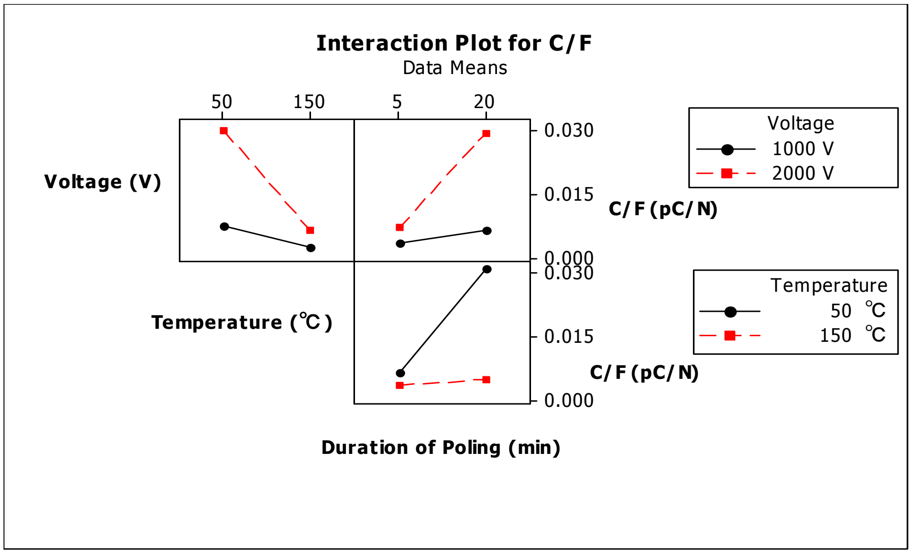

Figure 8. Interaction plot for C/F.

3.4 Finding the optimal temperature

According to the above results, samples generate higher response when they have been poled at the higher levels of voltage and duration of poling and at the lower level of temperature. However, since temperature had a negative impact on the response, its lower level (T=50°C) could not be accepted as the optimal condition and it was postulated that the samples have a higher response at a temperature between 50°C and 150°C. In order to find that optimal temperature, a single factor experiment was performed for different temperatures between 50°C and 150°C (T=75°C, 100°C, and 125°C) at the high levels of voltage and duration of poling. For the integrity of the experiment, each of the runs was replicated two times. The results, as shown in Figure 9, suggest that the samples generate the highest response at 75°C. Moreover, samples that are poled at 150°C have the lowest response.

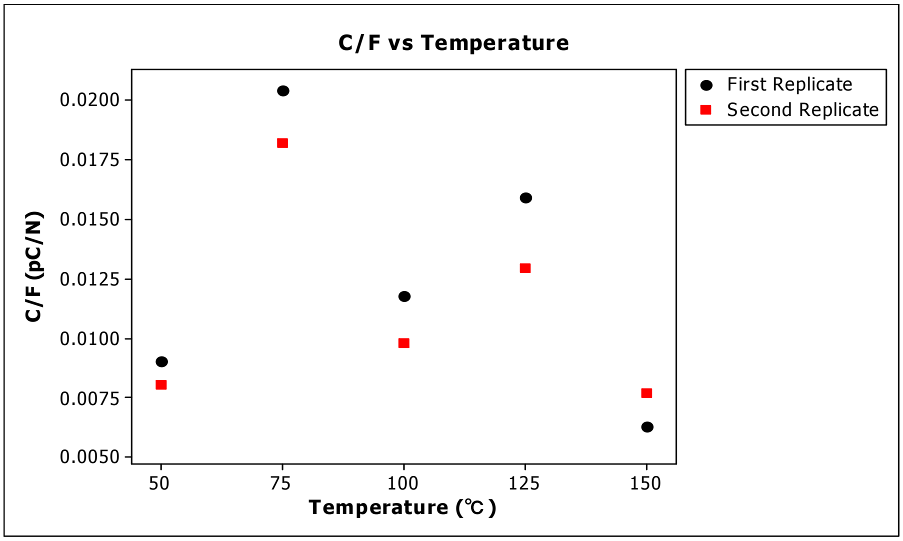

Figure 9. Sample's response at different temperatures.

4. FERROELECTRIC CHARACTERIZATION

In order to compare the ferroelectric properties of samples at different temperatures, the modified Sawyer-Tower circuit described in section 2.2 was used. As it can be seen in Figure 10, at 75℃, the coercive field is 0.097382 MV/m and the polarization is 0.122901 µC. Although samples that are poled at 75℃ generate the best response to the applied load, Figure 11b depicts that samples that are poled at higher temperature have higher polarizations, even after compensating for the resistive response. A justification for this observation could be that according to the interaction plots resulting from the DoE, samples are more sensitive to the changes in temperature for longer poling durations. For obtaining the hysteresis plots, a frequency of 0.2 Hz is used that results in a polarization period of 2.5 seconds for the positive portion of the triangle signal. Our DoE results show that a longer polarization time is required for the polarization to be developed at lower temperatures.

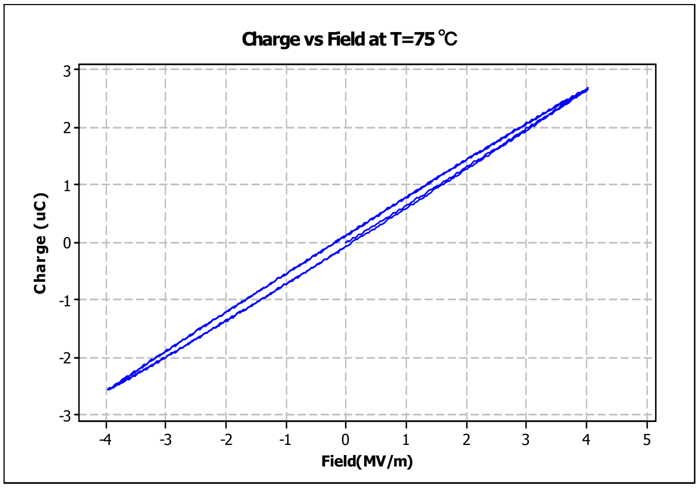

Figure 10. Hysteresis loop at T=75℃.

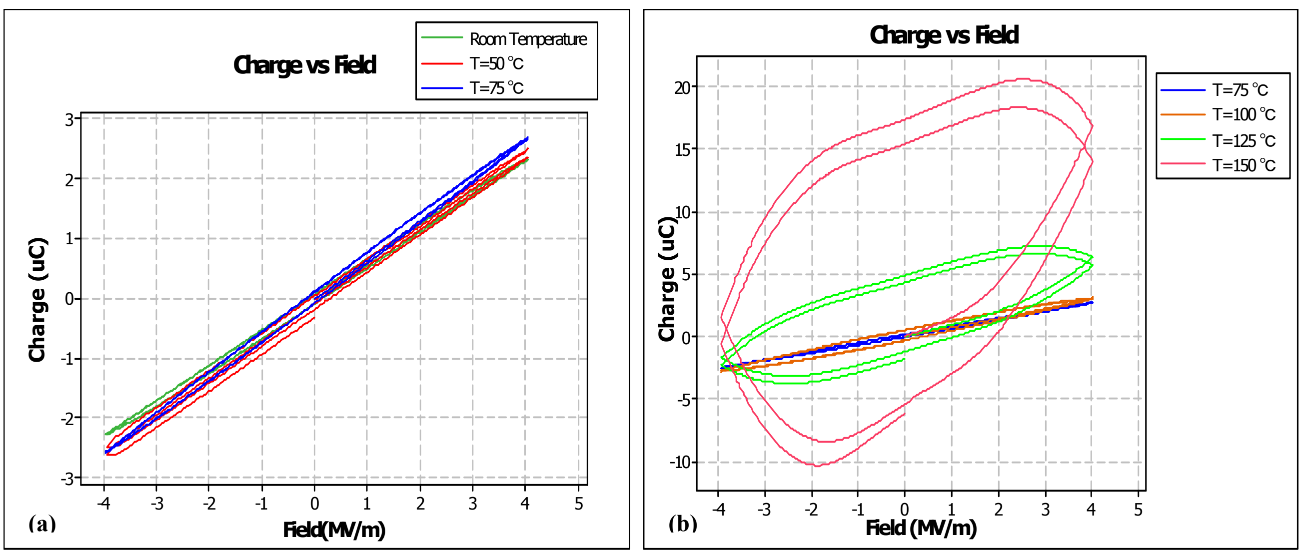

Figure 11.Comparison of the hysteresis loop at T=75 ℃ with (a) hysteresis loops obtained at lower temperatures, (b) hysteresis loops obtained at higher temperatures.

5. RESULTS

As discussed in the previous sections, the optimal conditions for poling the samples found to be at the high levels of applied voltage and duration of poling, and at the temperature of 75°C. Despite the fact that at 75°C, samples do not show high polarization according to their hysteresis plots, when poled for a long duration, they will generate the highest response when compared to other temperatures. Figure 12 depicts the charge generated by the sample that is poled by applying a 2000 V static voltage for 20 minutes at 75°C. As it can be seen, the sample generated a charge magnitude of 2.5 pC for that applied load.

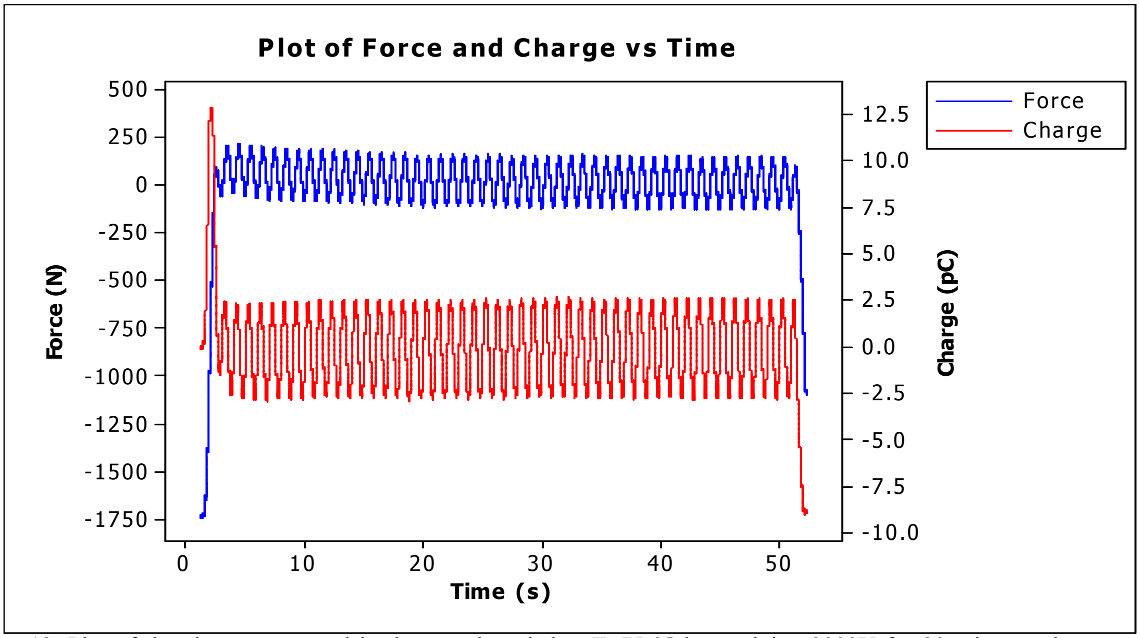

Figure 12. Plot of the charge generated in the sample poled at T=75 °C by applying 2000V for 20 minutes, due to an applied force in tensile test.

6. CONCLUSIONS

This study investigates a new smart composite structure that is mainly based on the conductivity of carbon fiber and the piezoelectric properties of PVDF. For this purpose, the samples were prepared and poled, and finally their response to an applied load was observed. Different factors can affect the poling of samples. The impacts of three of these factors, temperature, voltage, and duration of poling, were studied using a DoE method. It was observed that high levels of voltage and duration of poling has a positive impact on the charge generated by samples when exposed to an applied load. However, the effect of temperature was not as clear as the two other factors. Given the temperatures that were tested, poling at 75 °C showed the highest response.

The application of the samples was studied in a tensile test and it was observed that the samples are sensitive to an applied load and generate certain amount of charge. Among the samples that were poled at different conditions, samples that have been poled at 75 °C for 20 minutes by applying 2000V generate the highest response.

Overall, this study showed that the proposed composite structure is viable and it has the potential of being used as an integrated sensor for the purpose of structural health monitoring. Further development of this composite structure includes improvements in polarization and the application of the structure to failure detection.

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