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Reliability Analysis of Coating Material on Polyvinylidene Fluoride Layers Used in Piezoelectric Vibration Energy Harvesting Device

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Abstract: Piezoelectric energy harvesters operate by converting mechanical vibrations or strains into electrical energy. From recent research, it is understood that the choice of coating material for piezoelectric energy harvesters is a critical consideration that impacts the device's performance, durability, and compatibility with the intended application. Selecting the right coating material involves balancing the electrical, mechanical, and environmental requirements to optimize energy conversion and reliability. There are methods like the thermocycling process that can provide an accelerated ageing of the energy harvester in order to conduct a reliability assessment. The thermocycling process was carried out for 450 h on six samples of piezoelectric cantilever-type energy harvesters made of copper–nickel- and aluminum-coated PVDF (Polyvinylidene fluoride) piezoelectric material. The effect of aluminum and copper–nickel coating on PVDF piezoelectric material before and after the aging process was studied. The numerical results of the generated output voltage, surface resistance, and capacitance values measured before and after the accelerated aging process are presented in this study. This work also discusses the structure of the developed energy harvester, thermocycling experiment setup, and methodology of conducting the ageing process. It aims to provide a conclusion on the suitability of the PVDF metal coating material, the type of conductive adhesive to be used in order to seal the PVDF material to the harvester core, improvements in the structural design and selection of materials to reduce mechanical fatigue and ensure even stress distribution, and minimizing points of stress concentration, to help mitigate piezoelectric material delamination risks.

Keywords: piezoelectricity; polyvinylidene fluoride; metal coating; reliability; copper–nickel coating; aluminum coating; generated output voltage electrical capacitance; surface resistance

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1. Introduction

As climate change accelerates, efforts are being made to shift from the extraction and use of fossil energy sources. These conventional sources are endangered and have a negative impact on nature and human health. There has been growth in sustainable, environmentally friendly, and continuously renewable energy [1–3]. With the help of advances in high technology, efficient ways are being sought to obtain renewable energy; in addition to sources of renewable energy such as the sun or water, mechanical vibrations are also excellent sources [4–6]. The low-cost production, mechanical strength, and piezoelectric properties of PVDF films allow their application in mechanical-to-electrical power generation. One of the factors contributing to the higher efficiency of piezoelectric power harvesters is the durability of the device, which is affected by the different operating conditions, type of piezoelectric material, coating material of the piezoelectric film, electrode material of the harvester, and so on [7]. The choice of coating material directly affects the performance of the piezoelectric element. To maximize energy conversion, the piezoelectric material and the coating material must be compatible in terms of their mechanical properties, such as stiffness and thermal expansion coefficients, as highlighted in references [8,9]. In many practical applications, piezoelectric energy harvesters are exposed to various environmental conditions. The choice of a protective coating material is crucial to shield the sensitive piezoelectric element from moisture, dust, and other contaminants. Coating materials must also serve as electrical insulators to prevent short circuits between the piezoelectric element and any conductive components or the external environment. This is particularly important in applications where the harvester operates in challenging conditions, such as high humidity [10], or corrosive environments [10,11]. The link between electrical power and durability can be explained through the following theoretical principles:

- Piezoelectric properties: PVDF is a piezoelectric material, which means it generates an electric charge when subjected to mechanical stress. Repeated mechanical deformation can cause material fatigue and lead to structural defects, such as the crumpling and peeling of the film [12,13].
- Thermal expansion and stress: When materials experience cyclic thermal stresses, their coefficient of thermal expansion comes into play. This can create mechanical stress at the interfaces and within the material itself. Cyclic thermal stresses can induce mechanical strain, leading to the delamination or peeling of the film due to the differential expansion and contraction between layers, as seen in references [14,15].
- Electrical connection loss: When cracks, corrosion, or chipping occur on the surface of the PVDF film, the electrical connection to the surface electrodes can be compromised [16,17]. In a piezoelectric energy harvester, a continuous and reliable electrical connection is crucial for efficient energy conversion.

For energy harvesting systems where reliability is a critical factor, such as in healthcare or in aerospace applications, variations can impact safety and risk management. If reliability assessments vary significantly, it may be challenging to determine the level of risk associated with system failure, potentially leading to the overestimation or underestimation of risks. Variations in reliability assessments can also have legal and financial implications. The durability of piezoelectric energy harvesters can be investigated by subjecting the samples to different types of external stresses [18–20]:

- Aging of the energy harvester by placing it in a cold and heat chamber at a constant temperature for a certain period of time;
- Thermocycling energy harvesters according to a certain temperature profile for a certain No. of cycles;
- Cyclical external mechanical impact or impulse;
- Combination of temperature change and external cyclic mechanical action.

In this study, the process of thermocycling was used to accelerate the aging process of the developed energy harvester. The article is presented in the following structure. Section 2 presents the theory behind piezoelectricity, the structure of the energy harvester, and the design of the experiment. The initial parameter measurements and the thermocycling profile are also described in Section 2. The results of variation in generated output voltage, capacitance, and resistance that determine the quality of the metal coating on PVDF are presented in Section 3. Section 4 discusses the possible reasons for the obtained results and suggests ways of improvement to overcome the degradation of energy harvesters in the long run. Section 5 summarizes the conclusions.

2. Methodology

2.1. Structure of the Energy Harvester and Experimental Design

In the durability study, a piezoelectric generator was investigated which consists of several structural parts: a steel or plastic polyester (PET) core with a metalized PVDF film sealed with a conductive adhesive. A 3D model of the piezoelectric generator structure is shown in Figure 1. The film is glued on both sides of the core and is bonded in parallel so that the polarization direction is the same. In the case of a plastic core, copper electrodes are bonded. The core is mounted between two printed circuit boards with surface electrodes. The printed circuit boards are secured to each other with 3 mm diameter screws. Normally, a weight is attached at the free end of the piezoelectric power generators, depending on the operating conditions of the generator, i.e., the resonant oscillation frequency of the electric generator. The experiment involved 6 piezoelectric generators. The cores of three of the piezoelectric generators are made of 560 µm thick PET and the other three of 80 µm steel sheets. The generators were manufactured using PVDF films of two different metallicities. The piezoelectric generators with a PET core use copper–nickel-coated films, and those with a steel core use aluminum-coated film. The films are bonded to the core with a uniform, elastic, conductive, two-component adhesive. All piezoelectric energy harvesters with a steel core and aluminum-coated PVDF film have the same length and width (74 mm × 26 mm). The detailed parameters of the test objects are given in Table 1.

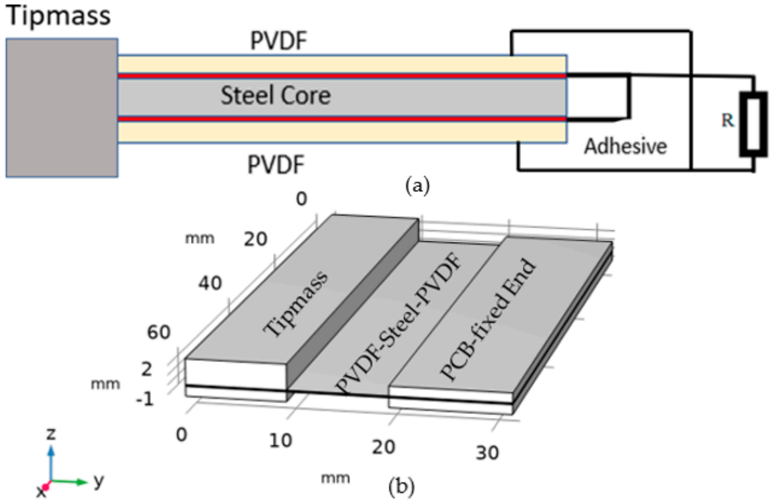

Figure 1. (**a**) The constituent layers of the proposed piezoelectric energy harvester. (**b**) 3D geometric structure modeled in COMSOL multiphysics software.

Table 1. Parameters of the energy harvesters tested in the experiment.

Energy Harvester No.	PVDF Electrode Material	Core Material	Core Thickness, µm	Piezoelectric Material Thickness, nm
1	Copper–nickel	PET	560	50
2	Copper–nickel	PET	560	50
3	Copper–nickel	PET	560	50
4	Aluminum	Steel	80	30
5	Aluminum	Steel	80	30
6	Aluminum	Steel	80	30

2.2. Thermocycle Test Profile

Temperature test experiments were carried out on six energy harvester prototypes. The temperature test simulated working conditions for the specimen; this means that an electromagnetic exciter should be used inside the temperature chamber to excite the specimen as in normal working conditions. Hence, along with a temperature test, a long-duration mechanical vibration test was also carried out. The test temperatures were in the range [−5 °C; 45 °C], and the study evaluated the variation in electrical quantities, output voltage and electrical capacitance, of piezoelectric energy harvesters by vibrating the free ends of the generators at a constant amplitude of 1 mm at a frequency of 50 Hz.

The signal input/output measuring circuit was connected from the outside of the chamber where there is room temperature. A constant acceleration input was maintained at 1 mm in all experiments. An optimal load resistance of 310 KΩ was applied. An appropriate temperature test chamber from Guangdong Yuanyao Test Equipment Co., Ltd. (Dongguan, China) was used, shown in Figure 2b. The entire thermocycling process was conducted for a duration of 450 h, where in one cycle, the temperature rose from −5.50 °C to a peak of 450 °C in 8 min then dropped to −5.50 °C in 16 min. The exciter mounted with the harvester was placed in a temperature chamber; the connections are shown in the flowchart in Figure 2a.

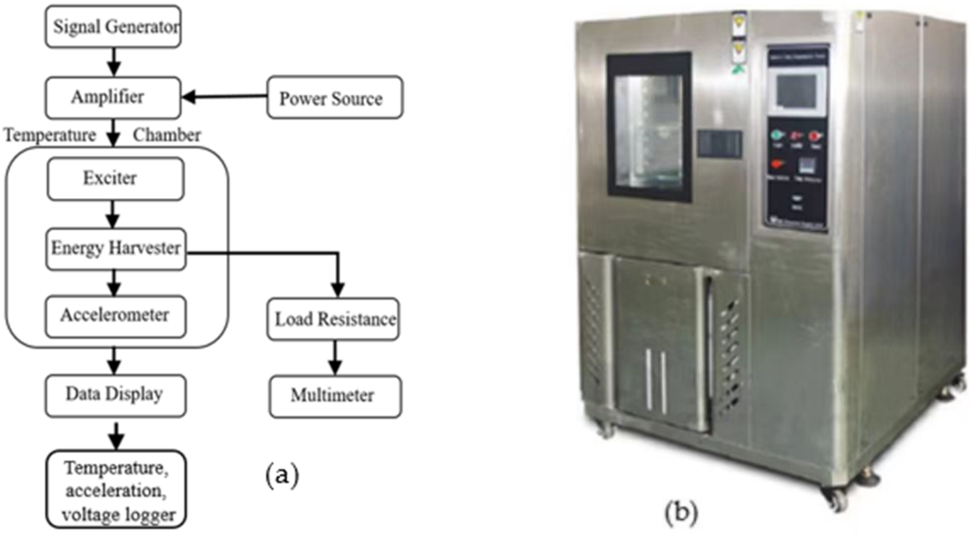

Figure 2. (**a**) Flow chart for the thermocycling process; (**b**) Guangdong Yuanyao test equipment used for the thermocycling process.

2.3. Initial Parameters of the Test Objects

This subsection presents the conditions for testing the durability of piezoelectric power generators. A temperature profile plot, mechanical parameters of the piezoelectric generators investigated, and measured values of the initial electrical capacitance are presented.

2.3.1. Conductive Adhesive

Epoxy resin serves as the adhesive intermediary bonding the PVDF layer to the metal substrate. Compatibility assessments and aging experiments were conducted to evaluate the appropriateness of this adhesive agent. Assessing the adhesive's efficacy is crucial as it must securely affix the PVDF film to the metal base, to seem like a unified material layer. Consequently, in order to assess the adhesive's performance, a thin layer of epoxy resin was applied to attach aluminum foil to the metal substrate. After the adhesive had fully cured, the foil was subsequently partitioned into discrete blocks, as illustrated in Figure 3. The standard deviation readings show that parts 1 to 8 have a higher deviation, suggesting that these parts have less connectively compared to parts 9 to 16. This could be because of the wear and tear near parts 1 to 8 where the tipmass is fixed. Aluminum foil glued to the metal core samples is shown in Figure 3.

The resistance of each block was recorded both before and after subjecting it to prolonged excitation. The resistance measurements remained relatively consistent across all samples, indicating that the adhesive maintained its integrity and effectively secured the foil to the metal substrate. Various ratios of epoxy and hardener were applied and evaluated to determine the optimal mixture proportion. It was determined that an equal mixture of epoxy and hardener yielded the desired consistency for the adhesive, allowing for convenient application within a 20 min window upon exposure to open air. This proportion also resulted in a robust, long-lasting bond between the PVDF layer and the metal substrate. Achieving an equal mixture of epoxy and hardener ensures a balance in the chemical reaction. In other words, it provides the right number of reactive sites in the epoxy resin and hardener to form a well-defined, interconnected network. Deviating from this balanced ratio can result in incomplete curing or an excess of unreacted components, leading to compromised mechanical properties and durability. The standard deviation readings show that parts 1 to 8 have a higher deviation, suggesting that these parts have less connectively compared to parts 9 to 16. This could be because of the wear and tear near parts 1 to 8 where the tipmass is fixed. Four samples were tested, and the average resistance and standard deviation readings are shown in Table 2.

Figure 3. The aluminum foil was glued to the steel core to measure the quality of the glue.

Table 2. Resistance and standard deviation of the aluminum (Al) foil glued on the metal base over a time period.

Part. No.	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**	**16**
Week 1 (Ω)	1.7	3.2	25.3	2.3	2.7	1	7	1	0.9	2.1	2	1.6	1.1	1.8	7.4	9.5
Std Deviation Week 1 (σ)		1.6	1.7	10.3	18.1	1.4	1.5	2.4	0.6	0.7	0.5	0.8	0.5	0.9	0.4	2.3
Week 4 (Ω)	1.4	0.9	62.4	1	18.6	1.6	0.9	1.4	1.1	2.2	1.5	2.7	3.5	2	25.4	1
Std Deviation Week 4 (σ)		1.4	1.5	26.3	15.7	12.5	1.0	0.6	0.4	0.3	1.1	0.2	0.6	0.9	0.7	3.0

2.3.2. Electrical Capacitance

Prior to the test, the electrical capacitance values of both the PVDF films of the piezoelectric generators were measured between the core electrode and the lower and upper film electrodes. The measurement of the electrical capacitance was carried out in order to assess the quality of the bonding between the metallized piezoelectric film and the core electrode connecting the two film layers. Ideally, the capacitance after bonding should not differ from the capacitance value between the top and bottom layers of the PVDF film. After bonding the layers, the capacitance should be between 3.2 nF and 3.9 nF. Measurements were made using a laboratory LCR meter. The capacitances of the piezoelectric oscillators between the core and film electrodes are given in Table 3.

The measured capacitance values indicate that the connection between the electrodes of the piezoelectric generators and the electrodes of the core is of good quality at the initial moment. The differences in capacitance values are due to the manual manufacturing of the piezoelectric generators, and the difference in capacitance values may be affected by inaccuracies in the manufacturing process, such as errors in cutting the PVDF strips, preparation of the core for bonding, and compression of the core during bonding.

Table 3. Capacitances of energy harvester between the core electrode and PVDF film electrodes.

Energy Harvester No.	The Capacitance between the Core and the Bottom Layer, nF	The Capacitance between the Core and the Top Layer, nF
1	3.61	3.46
2	3.65	3.54
3	3.45	3.48
4	3.71	3.61
5	3.57	3.52
6	3.14	3.3

3. Results

This subsection provides an overview of the changes in electrical quantities after the experimental test. The effect of thermocycling on the polyester (PET) and steel core generators and on aluminum- and copper–nickel-coated PVDF film is discussed.

3.1. Variation in Generated Output Voltage of the Energy Harvester after the Thermocycling Process

The dependence of the generated output voltage of the energy harvesters on temperature and operating time is presented in Table 4. The percentage decrease in the generated voltage, comparing the value of the output voltage at the beginning of the test with that at the end of the test, is 61% for energy harvester No. 1, 36% for energy harvester No. 2, 46% for energy harvester No. 3, and 74% for energy harvester No. 4. The dependence of the output voltage of all the energy harvesters on the duration of the experiment is shown in Figure 4. The energy harvesters Nos. 1, 2, 3, and 4 remained in operation until the end of the test. Energy harvesters No. 5 and No. 6 did not generate any voltage after the thermocycling process. The results of the investigation of the three PET core energy harvesters (Nos. 1, 2, and 3) show that the output voltage of the plastic-core piezoelectric generators is temperature-dependent. The dependence of the generated output voltage of the energy harvesters on temperature and operating time is presented in Table 4. The voltage drop after the thermocycling process is shown in Figure 4.

Table 4. Generated output voltage of energy harvesters before and after the thermocycling process.

Energy Harvester No.	Voltage Generated before Thermocycling (V)	Voltage Generated after Thermocycling (V)	Percentage Reduction (V %)	Running Time in Operation (H)
1	1.64	0.68	61	450
2	2.26	1.45	36	450
3	2.31	1.24	46	450
4	0.93	0.34	74	450
5	0.32	0	100	142
6	0.51	0	100	217

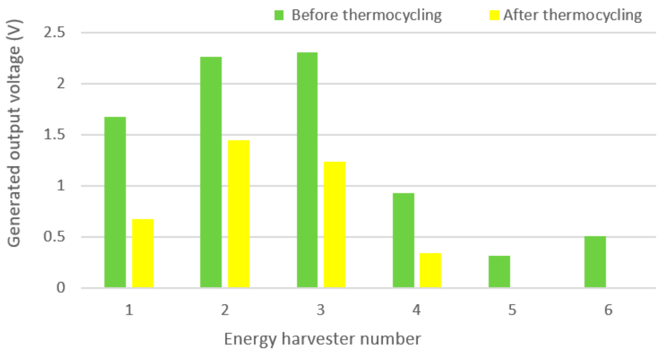

Figure 4. Histogram of change in generated voltage of the energy harvesters before and after the thermocycling process.

3.2. Variation in the Electrical Capacitance of the Energy Harvester between the Core and the Surface Resistance of the PVDF Film Electrode

After the thermocycling process, the results show that all the generators tested have a reduced electrical capacity due to cyclical temperature and mechanical stress. The values measured after the test show that all energy harvesters have reduced electrical capacities. The capacitance between the core electrode and the bottom layer decreased from 41.7% to 65.4%, and between the core electrode and the top layer, the capacitance decreased from 47.1% to 79.2% for energy harvesters Nos. 1 to 3. The smallest reduction in capacitance was measured in harvester No. 3. The highest capacitance changes in all the energy harvesters were measured in energy harvesters No. 5 and No. 6. At the end of the study, the following was observed: a 97.4% and 98.0% reduction in electrical capacitance between the core electrode and the lower and upper layers of harvester No. 5. In harvester No. 6, the reduction in electrical capacitance was 100% in both layers as the capacitance was not measurable at the end of the test. These harvesters ceased to operate during the course of the test (at a test time duration of 142 h and 217 h, respectively). A graphical representation of the variation in the lower and upper PVDF film layers of the energy harvester is shown in Figure 5. The estimates of the electrical capacitances of the piezoelectric generators are given in Table 5.

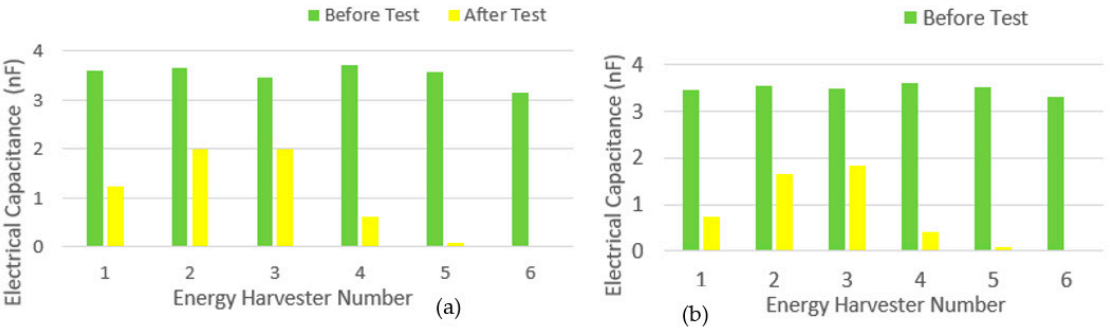

Figure 5. Change in electrical capacitance of the lower layer (**a**) and the upper layer (**b**) after thermocycling in a temperature chamber.

Table 5. Capacitances of energy harvesters between the core and PVDF film electrodes after thermocycling process.

Energy Harvester No.	Capacitance between the Core and the Bottom Layer, nF	Capacitance between Core and Top Layer, nF	Change in Capacitance between the Core and the Lower Layer, %	Change in Capacitance between the Core and Top Layer, %
1	1.25	0.72	65.4	79.2
2	2.01	1.64	44.9	53.7
3	2.01	1.84	41.7	47.1
4	0.61	0.41	83.6	88.6
5	0.07	0.09	98	97.4
6	-	-	100	100

Visual assessment of the harvesters clearly shows that cyclic thermal and mechanical loading has caused significant mechanical damage to the steel core and aluminum-coated PVDF films of the energy harvesters. A post-test image of an energy harvester with aluminum-coated PVDF film and a metallic core is shown in Figure 6.

PVDF film damage is also visible in energy harvesters with a plastic core and a copper–nickel coating. The evaluation of energy harvesters with a plastic PET core and a copper–nickel coating film showed that the surface was also affected by thermal cycling, and several areas of complete wear of the coating are visible. The piezoelectric PVDF film coating was beginning to crumble and peel due to external cyclic mechanical and thermal stresses. A visual assessment of all the harvesters shows a general trend of metallization chipping, corrosion, and metallization cracks. The PVDF electrodes connect various components and are responsible for the flow of electrical current. Longitudinal electrode abrasion can result in increased electrical resistance at the contact points, potentially leading to voltage drops, reduced electrical conductivity, and overheating. This can affect the overall functionality and lifespan of electronic devices.

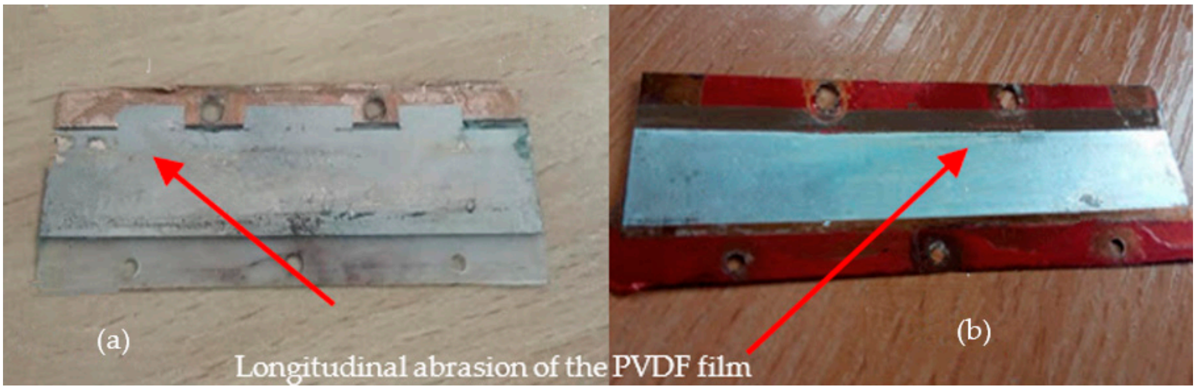

Figure 6. (**a**) Energy harvester No. 3 with a plastic PET core and copper–nickel-coated PVDF film. (**b**) Energy harvester No. 4 with a steel core and an aluminum-coated PVDF film.

3.3. Variation in the Resistivity Measurements of the Surface Electrode of a Piezoelectric Oscillator with PVDF Film

In order to further assess the damage to the piezoelectric PVDF film in the durability study, impedance measurements of the surface PVDF film at the end of the study were performed. Measurements were taken between the initial point 0 and four other points spaced further apart in the metallization plots along the piezoelectric PVDF film. The resistance of the surface electrode of a 10 mm wide by 74 mm long piezoelectric PVDF film, irrespective of the type of coating, should not exceed 50 Ω between the 0 and 4 points prior to the reliability tests of the energy harvesters. The detailed results of the measurement of the surface electrode resistance of the PVDF film with respect to point 0 are given in Table 6.

Table 6. Results of surface resistivity measurements (relative to point 0).

No.	**Type of Piezoelectric PVDF Film Coating**	**PVDF Film Layer**	**Surface Resistance between Point 0 and Other Points, kΩ**			
			1	**2**	**3**	**4**
1	Copper–nickel	Top	1.1	2	14.9	16.4
		Bottom	4.3	5.6	12.6	18.2
2	Copper–nickel	Top	2.4	3.5	7.2	14.6
		Bottom	2	3	4	10
3	Copper–nickel	Top	2.2	10.2	12.3	15.2
		Bottom	3.4	9.8	12.6	16.7
4	Aluminum	Top	1.2	2.3	3.3	4.1
		Bottom	0.7	2	2.1	2.7
5	Aluminum	Top	-	-	-	-
		Bottom	1.2	2.1	2.5	3.6
6	Aluminum	Top	2.5	5.1	7	8.1
		Bottom	-	-	-	-

The structure of the measurements of the surface resistivity of the PVDF film of the energy harvesters is shown in Figure 7.

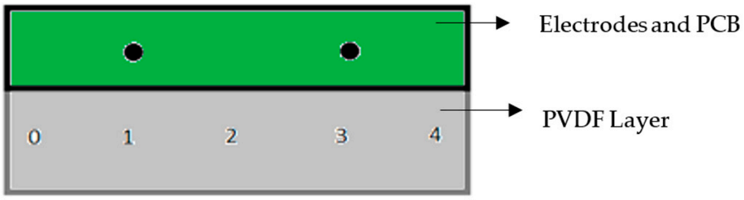

Figure 7. Points at which the surface resistances of the harvester were measured.

4. Discussion

Apart from the tests used for quality control functions such as thickness, porosity, adhesion, strength, hardness, ductility, chemical composition, stress, and wear resistance, there are other practical ways of testing surface coatings [21]. Machine efficiency, reduced power requirements, and longer running life are some of such practical methods [8,22]. In this study, we measured the surface resistance, surface capacitance, and the voltage generated by harvesters before and after the thermocycling test to evaluate the coating adhesion to the PVDF material. From the results of the surface resistivity measurements, it can be seen that the surface resistivity of the piezoelectric PVDF films of all the piezoelectric power generators increased when measured along the PVDF film. The highest increase in electrode resistance was measured in energy harvester No. 1 with a copper–nickel coating on the PVDF film. The resistance of the PVDF film of the generator between point 0 and point 4 increased to 16.4 kΩ in the top layer and 18.2 kΩ in the bottom layer. The surface resistivity of energy harvester No. 2 increased to 10 kΩ in the lower layer and 14.6 kΩ in the upper layers, and that of energy harvester No. 3 increased to 16.7 kΩ in the lower and 15.2 kΩ in the upper layers. In energy harvester No. 4 using aluminum-coated PVDF film, an increase in impedance was seen between point 0 and the other points. Between point 0 and point 4, a resistance of 4.1 kΩ in the lower layer and 2.7 kΩ in the upper layer was measured. In harvester No. 5, the resistance in the lower layer was measured at 3.6 kΩ, while in harvester No. 6, no resistance value was seen in the lower layer, but a value of 8.1 k Ω was measured in the upper layer. A comparison of the changes in the resistivity of the piezoelectric PVDF film electrodes shows that the largest increase in surface resistivity was in copper–nickel-coated films. Copper and nickel alloy coating is known to have low friction and good corrosion resistance; additionally, the natural characteristics of the alloy protect the films from high-temperature wear damage. However, aluminum coating is cheaper compared to nickel and copper alloy; it works better for corrosion resistance but is suitable only for low to moderate temperatures, making it unsuitable for high temperatures. The effect of thermocycling in newly manufactured energy harvesters can be reduced by improving the generator design: laminating the core with additional protective layers and coating the PVDF film with a protective spray coating. A protective coating would increase the resistance of energy harvesters to temperature and humidity and protect the PVDF film against corrosion. In addition, it is advisable to adopt a new bonding technique in subsequent products, i.e., vacuuming the core of the piezoelectric generator during the bonding process, in order to ensure that the PVDF film is pressed evenly against the bonding surface and to avoid air voids between the PVDF film and the core surface. Air voids not only increase the delamination risks but also lead to inconsistent electrical performance by introducing variability in the electrical connections between the PVDF film and the core. This can result in fluctuations in the generated electrical output.

5. Conclusions

The following conclusions were made:

1. Metallized PVDF films are used to generate electricity from mechanical oscillations using piezoelectric energy harvesters. The aging of the devices is fastest when temperature cycles and mechanical vibrations are applied simultaneously.
2. The thermocycling process has shown that energy harvesters with a PET core and a PVDF film coated with a copper–nickel coating generate a ~3.5 times higher output voltage than those with a steel core and an aluminum-coated PVDF film.
3. The output voltage of the PET-core energy harvesters decreased by 51% during the experiment (450 h), while the output voltage of the steel-core piezoelectric oscillator decreased by 65% (two energy harvesters stopped functioning before the end of the experiment).
4. Delamination can lead to very undesirable conditions, such as lower durability and reliability, and most likely to electrical short circuits. All harvesters showed a reduction in the electrical capacity between the core and PVDF film electrodes due to degradation of the PVDF films and film delamination. Also, an increase in the metallization resistance of the PVDF surface was observed due to various PVDF damages (corrosion, chipping, longitudinal electrode fractures).
5. The analysis of temperature and mechanical effects on energy harvesters led to the following solutions to improve the durability of piezoelectric oscillators: spraying a protective coating on the PVDF film and using an intermediate plastic sheet between the core and the PCB to prevent the metallization of the PVDF film from wearing away the edge of the PCB during the flexing of the core of the energy harvester can allow the minimization of the effect of fatigue on the bonding condition and the degradation of the PVDF film under different thermocyclic and mechanical stresses.

Author Contributions: C.R. and V.M. jointly conceived the idea. C.R. and V.M. designed and fabricated the device, built the experimental setup, and performed the experiments. C.R. wrote the manuscript with contributions from all co-authors. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

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