Experimental Investigation of Continuous Usage Stability of Organic Polymer Electret in Micro Energy Harvesting

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Abstract— An electret is a stable dielectric material with a quasi-permanently embedded static electric charge or dipole polarisation. Theoretically, electrets should retain deposited charges for a long period of time. However, their charges are known to deteriorate with time due to polarising charge relaxation. There are organic and inorganic electrets. Cyclic Transparent Optical Polymer (CYTOP) is a good organic electret material with high charge density. Most studies on CYTOP charge stability has been concentrated mainly on storage, as it is believed that the same stability is exhibited in usage. We present the experimental investigation carried out, on storage stability, and continuous usage stability of CYTOP (grade A) electret. In this work, thin-film fabrication of four samples of CYTOP (CTL-809A) electret of same thickness was carried out; and corona charged with different voltages. One of them was stored and the others were used in vertical vibration based cantilever-electret micro-power generation for 52 days. Their surface charge decay (by surface potential measurement) with number of days was investigated. It was found out that the surface charges of the fabricated CYTOP electrets deteriorated faster with continuous usage than in storage.

Index Terms— Energy Harvesting, Electret, CYTOP, Charge Stability, Corona Charging, Surface Potential, Micro Power Generation.

1 INTRODUCTION

IN recent times, the power requirements of portable electronic devices have steadily increased, and are expected to exceed the capacity of conventional secondary batteries in future. With the emergence of MEMS, NEMS devices and other handheld devices, the issue that calls for concern is the battery life of these devices; and as a result, micro power generation systems that could replace these batteries have begun to receive significant attention [1], [2], [3], [4]. Electromagnetic induction is not suitable for very small electric power generation in energy harvesting, but "electrostatic induction offers numerous advantages, such as a simple structure and high output voltage at relatively slow speeds" [5]. Use of electret has proved promising in this area.

An electret is a stable dielectric material with a quasi-permanently embedded static electric charge or dipole polarisation. It is an electrical counterpart of a permanent magnet. The embedded static electric charges in electrets, due to the high resistance of the material, will not decay for a long period of time. Malecki (1999), reported that resistivity of electret material has little effect on the time scale of its charge decay [6]. Inorganic electrets are quartz, and other forms of silicon dioxide. Organic electrets are made from synthetic polymers like fluoropolymers, polypropylenes, polyethyleneterephthalate, etc.; waxes, and resins [7]. For conventional applications of electrets, fluorinated polymer materials, such as PTFE and FEP, are often used.

Cyclic Transparent Optical Polymer (CYTOP) is a perfluoronated amorphous (non-crystalline) polymer, which was developed by Asahi Glass Company Limited, Japan [8]. It has a peculiar polymer structure shown in Fig. 1, which is different from any other existing fluoropolymers, but it is similar to Polytetraflurorethylene (PTFE) in chemical and physical characteristics.

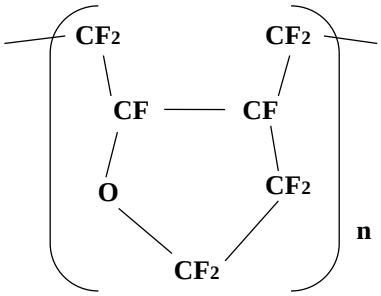

Fig. 1: The Molecular Structure of CYTOP [8].

Attractive properties of the polymer include: high thermal stability (decomposition temperature> 400°C), low dielectric constant (2.0 to 2.1) and high dielectric breakdown strength

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(10kV/0.1mm); low water absorption (<0.01%) and high impermeability; low surface tension and excellent chemical resistance to acids, alkaline and organic solvents. Since it is a thermoplastic, it lends itself well to extrusion coating. CYTOP also maintains a low refractive index of 1.34, low coefficient of optical dispersion, and good lamination properties. CYTOP can be easily patterned with O_2 plasma etching and is compatible with MEMS fabrication process because it is soluble in perfluoronated solvents; thus thick films can be obtained by multiple spin coating [8]. CYTOP has been demonstrated to be a good electret material with high charge density [3], [9], [10]; and its use as electret material is promising as some are reported in [1], [3], [10], [11], [12], [13], [14], [15], [16], [17], [18], [19], [20], [21], [22], [23], [24], [25], [26], [27], [28].

CYTOP is available in three different commercial grades (grades A, M and S) for different applications. Each has different end group which are, CTL-S (trifluoromethyl: -CF_3); CTL-A (carboxyl: -COOH); and CTL-M (amidosilyl: -CONH-C_3H_6-$Si(OC_2H_5)_3$. The modification of the end group is performed to increase the physical and chemical properties of CYTOP. CTL-S has excellent transparency and non-adhesion properties because of the absence of a functional end group. Carboxyl end groups of CTL-A form hydrogen bonding with the hydroxyl groups of organic and inorganic substrate, and amidosilyl end groups of CTL-M can form strong chemical bonding with an inorganic substrate to promote the adhesion property dramatically [8], [22].

Electrets, when charged, are in non-equilibrium state that disappears when in operation due to polarising charge relaxation. Depending on the storage temperature, relatively rapid charge decay is first observed, then stabilises for a long period of time. Tsutsumino *et al.* (2005), examined the temporal evolution of the surface charge density of a 3μm-thick CYTOP, corona charged between -6kV and -10kV for 3 minutes and then stored for about 400 hours. Their findings revealed that the surface charge density depends on the corona voltage; and remained stable for the period of the 400 hours though the one charged with corona voltage of -8kV showed little initial rapid decay for about the first 50 hours. Also, trapped charges in the CYTOP were found to be stable if charged at least up to its glass transition temperature [9].

Bartsch *et al.* (2008), investigated long term storage stability performance of CYTOP electret layers, charged by corona charging, and stored under 23°C. Their findings revealed that the material retains, at least, 92% of its initial charge 143 days after charging [29].

Sakane *et al.* (2008), examined the temporal change of surface charge density of the three grades of CYTOP of 15μm thickness, charged with corona voltage of -8kV for 3 minutes, and stored under 23°C and 60% humidity for up to about 4000 hours. Their results showed that the surface charges of both the CTL-M and the CTL-A are stable though the surface charge density of CTL-A is lower to that of CTL-M. However, CTL-S is not stable as it quickly loses its charge. Therefore, they concluded that a small number of functional end groups such as carboxyl or amidosilyl significantly enhance the electret performance. The surface charge density becomes higher, and the charge decay is suppressed. Also, thermal stability is improved [22].

Mescheder *et al.* (2008), worked on 500nm silicon dioxide (SiO_2) films, charged by ion implantation; and observed the surface charge drift for a storage period of two months. Their work showed an initial exponential short term decay, followed by an almost linear long term decay in the surface potential decay pattern. Also, they observed that the decay time shows a strong dependence on the initial surface potential especially the first 10% decay time [30].

Saad *et al.* (2010), used ionic hairdryer instead of corona charging to charge two CYTOP electrets of different film thickness, and stored for about 140 days. Their results, among others, showed that the maximum initial surface potential attained by the electrets partly depends on the CYTOP thickness. Also, the de-charging behaviour of the one with higher initial surface potential is more pronounced than that of the other [5].

Wang and Hansen (2013), carried out humid test on storage stability of silicon dioxide (SiO_2) and CYTOP electrets. After 440 minutes exposure to R.H. 97% humid environment, the surface charges for CYTOP suffers a continuous surface potential decrease of about 5%; while the inorganic electret, SiO_2, drop by about 2% [26].

From the foregoing, it appears that CYTOP electret stability depends on various conditions including film thickness, terminal end groups, technique of charging, corona voltage, charging temperature, initial surface potential, storage conditions etc. Most studies on CYTOP charge stability has been concentrated mainly on storage. However, functionality of applications mainly based on electrets is known to deteriorate with use over time, and this has always been attributed to the characteristics of the storage stability behaviour of the electrets.

Therefore, the objective of this work is to examine, through experimental investigation, storage stability, and continuous usage stability of CYTOP electret surface charges. In this work, thin-film fabrication of four samples of CYTOP (end group CTL-A (CTL-809A)) electret of same thickness was carried out; and corona charged with different voltages. One of them was stored and the others were used in vertical vibration based cantilever-electret micro-power generation for 52 days. Their surface charge decay (by surface potential measurement) with number of days was investigated.

2 MATERIALS AND METHODS

2.1 Thin Film Electret Formation

Four samples of 20 mm by 20 mm substrate were fabricated from a copper plate with a thickness of 1.5 mm. Rough edges of the substrate were smoothened and cleaned using emery cloth. The sample substrates were washed with ethanol (CH_3COOH) and distilled water using a vibrator, after which the samples were dried using nitrogen (N_2) gas. Three drops of aminopropyltriethoxy silane were deposited on the copper substrates and spin-coated for 10 seconds using a spin coater (Mikasa 1H-D7). Then, Cyclic Transparent Optical Polymer (CYTOP (CTL-809A)) from Asahi Glass Company, Japan [8], was applied to the substrates, spin-coated and soft-baked at 100°C for 10 minutes. This process was repeated four times to obtain an 8μm -thick film. After this, the CYTOP film was then cured fully at 180°C for 90 minutes. Electrical connection was made to three of the sample substrates to complete them as lower electrodes in micro power generation setup.

2.2 Corona Charging Process

The electrets were charged using a corona discharge setup that is illustrated in Fig. 2. The discharge voltage was set between -2.11kV and -2.29kV. The grid voltage was set between -400V and -600V. The charging current was 0.02mA. Charging was performed for 3 minutes after which the average surface voltage of the samples was measured at a tip-to-wafer distance of 3.8mm using Electrostatic Voltmeter (TREK 344 ESVM) with a stage controller. The parameters of corona charging and usage/storage conditions of the four electret samples are shown in Table 1.

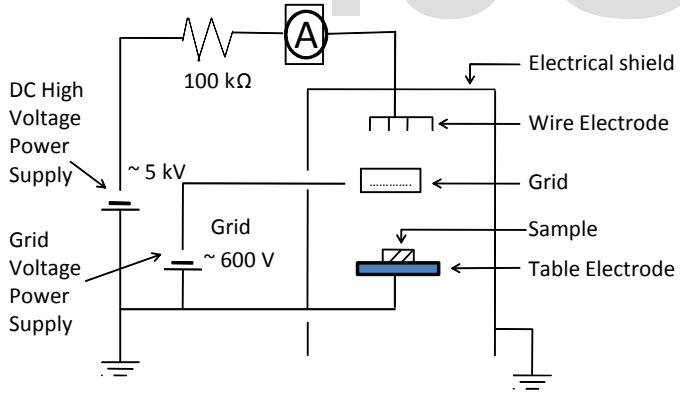

Fig. 2: The corona discharge setup.

Samples 1, 2 and 3 were used in micro power generation set up for 52 days during which the surface voltages of the electrets were being measured. Sample 4 was stored in an environment under a controlled temperature and humidity for the same period, and its surface voltage was also measured with time.

Table 1: Parameters of corona charging and conditions of storage/usage of the four electret samples

Sample No	Discharge Voltage (kV)	Grid Voltage (V)	Initial Surface Potential *(V)	Charging Temperature (°C)	Relative Humidity (%)	
1	-2.22	-600	-453	26	59	Usage
2	-2.29	-600	-525	26	57	Usage
3	-2.11	-400	-380	26	68	Usage
4	-2.19	-400	-400	26	72	Storage

* *The initial surface voltage on the electrets after charging.*

2.3 Micro Power Generation Setup

Samples 1, 2 and 3 were used in a cantilever-electret micro power generation setup in the laboratory with vibration in the vertical plane (out-of-plane vibration), for a period of 52 days during which the surface voltage of the electrets were being measured. The concept of the vertical vibration based cantilever-electret micro power generator is illustrated in Fig. 3(a). An electret dielectric with air is placed between two plate electrodes as shown in the figure. The electret is attached to the lower electrode (base electrode) while the upper electrode is allowed to vibrate freely in the vertical direction. The upper electrode was made in form of an A-shaped cantilever which was fabricated from a material of double-sided copper with embedded glass epoxy. Electrical connection was made to the cantilever. Figs. 3(b) and (c) show a completed sample electret with a conductor connected, and the dimensions of the fabricated A-shaped cantilever respectively.

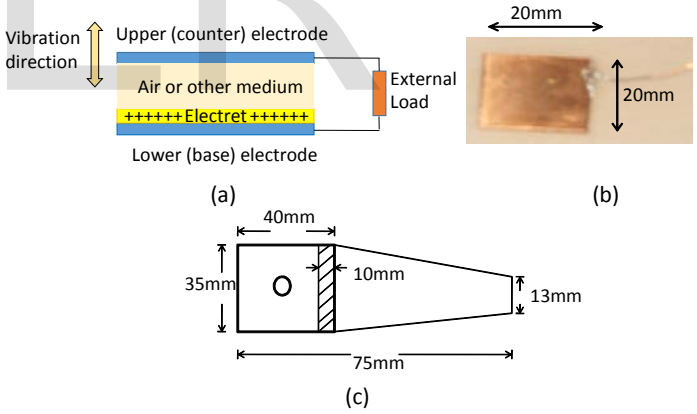

Fig. 3: (a) Basic concept of the vertical vibration based cantilever-electret micro power generator. (b) A completed sample electret with a conductor connected. (c) Dimensions of the fabricated A-shaped cantilever.

The upper electrode and the lower electrode (with electret film on it) facing each other were connected, through a coaxial cable, to an external load resistor of 1MΩ across which the output of the generator was obtained. The schematic of the cantilever-electret micro power generation set up is shown in Fig. 4. The micro power generator was mounted on a shaker (Vibropet-05) and also attached to a precision stage. The shaker is controlled by a Vibration Exciter (APD-200FCG) that sets the vibration waveform, amplitude, acceleration and the frequency. The precision stage was connected to a stage control-

ler which was used to set the mean distance between the cantilever electrode and the electret.

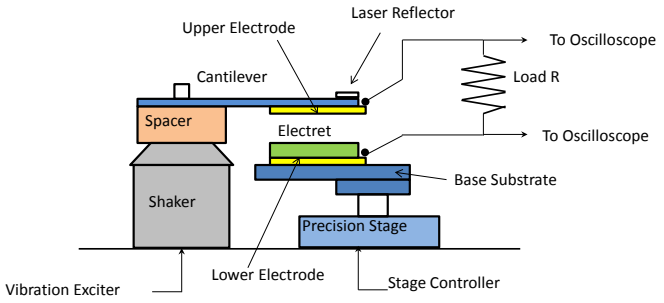

Fig. 4: Schematic of the setup for the micro-power generation.

The upper electrode of the generator was vibrated in the vertical direction to represent mechanical vibrations from the environment. The external vibration waveform was set to be sinusoidal at a fixed frequency of vibration of 100Hz, acceleration of 30.2m/s² and cantilever vibration amplitude of 0.15mm (pk-pk). The movement of the cantilever with respect to the lower electrode was observed with a high frame rate digital camera and a laser vibrometer (LV-1710). The electrical output of the micro power generator was connected to one of the input channels of a digital oscilloscope (Tektronix TDS2014B), which was used for observation. The micro power generator was operated using the electret samples 1, 2 and 3 for 52 days under the conditions shown in Table 1.

3 RESULTS AND DISCUSSION

3.1 Initial Surface Potential

Table 1 in Section 2.2 shows the charging and storage/usage conditions of the four electret samples. Fig. 5 shows the magnitude of the attained initial surface potentials of the four electret samples with their respective corona charging voltage.

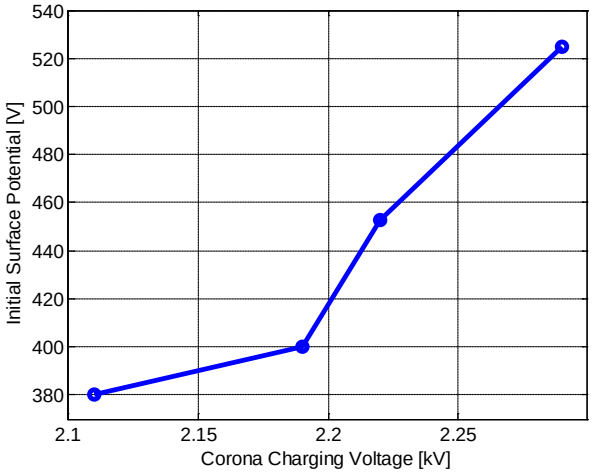

Fig. 5: Corona charging voltage with initial surface potentials of the electrets (actual values are negative).

From Fig. 5, it can be observed that given the same length of charging time, the four samples attained different initial surface potentials in a way that is proportional to the corona discharge voltage. In corona charging, the grid controls or limits the electret's surface potential that results from charges that were injected. Charging will stop when the electret's surface potential equals the grid voltage. In Table 1 and Fig. 5, sample 2 that was charged with the highest corona charging voltage attained the highest surface potential, while sample 4 attained a limiting potential of -400V which is equal to the grid voltage used in its charging. This agrees with Tsutsumino *et al.* (2005), whose work showed that the initial surface charge density of CYTOP electret also depends on the corona charging voltage [9].

3.2 Stability

Long time retention of electret surface charges is of utmost importance, especially for its use as outdoor energy harvesters. Electret surface charge density is related to its surface potential. From the measured surface potential V, and for a given electret thickness d, the surface charge density ρ_s can be calculated as

$$\rho_s = \frac{\varepsilon_o \varepsilon_r V}{d} \quad (1)$$

where ε_o and ε_r are the permittivity of free space and the electret dielectric constant respectively. Equation (1) shows that the surface charge density is directly proportional to the surface potential. Therefore, an observation of the decay of the surface potential with time gives an indication of the stability of its surface charge density. Samples 1, 2 and 3 were used in micro power generation setup, while sample 4 was kept in storage. Fig. 6 shows the electrical output obtained from the micro power generation setup using one of the electret samples. The frequency spectrum shows a main component of 100Hz, which is the same as the set frequency of vibration.

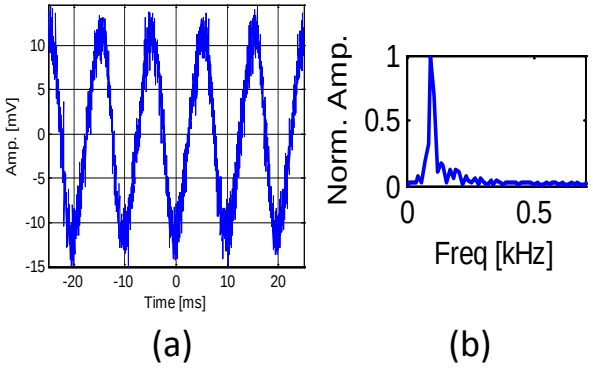

Fig. 6: Electrical output obtained from the micro power generation set up using one of the electret samples (a) Waveform (b) Its frequency spectrum.

Fig. 7 shows the decay of surface potentials of the four electret samples with the number of days of usage and storage. From the figure, it can be observed that the surface potential of all the four sample electrets decay with number of days.

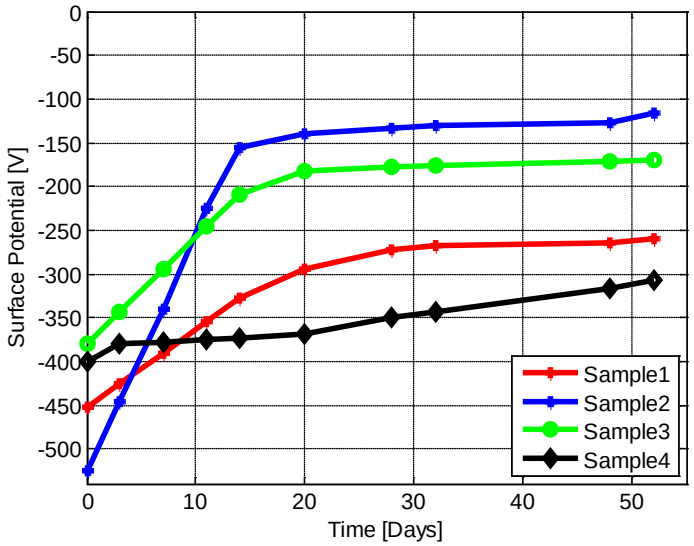

Fig. 7: Surface potentials of the four electret samples with number of days.

For ease of observation and comparison of the decay of the surface potentials of the four electret samples, the surface potential of each sample is normalized with its respective initial value and express in percentage. This is shown in Fig. 8.

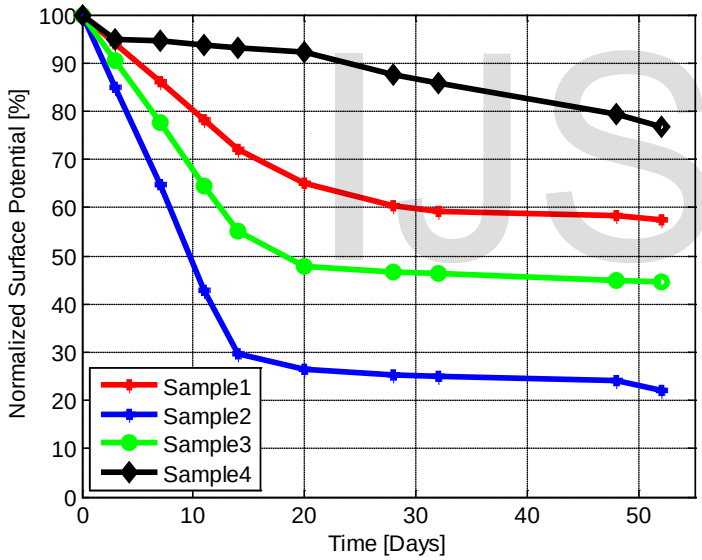

Fig. 8: Normalized surface potentials of the four electret samples with number of days.

From Fig. 8, it can be observed that the surface potential of all the four sample electrets decay with number of days, exhibiting an exponential short term decay, followed by an almost linear long term decay in their decay patterns; thus agreeing with Mescheder *et al.* (2008) [30]. The three electret samples that were used in micro power generation exhibit a relatively initial rapid exponential short term decay of the surface potential for the first twenty days after which they remain fairly stable for the remaining number of days. Of the three samples, relatively, sample 1 shows the least initial rapid decay while sample 2 shows the most initial rapid decay with time. At the end of 52 days of use, sample 1's surface potential has reduced to 58% of its initial value, sample 2's surface potential has reduced to 22% of its initial value, and sample 3's surface potential has reduced to 45% of its initial value.

Sample 2, which attained the highest initial surface potential, surprisingly exhibits the worst usage stability; which therefore seems the higher the initial surface potential is, the lower the stability becomes. This agrees with the observation of Mescheder *et al.* (2008), that the 10% decay time shows a strong dependence on the initial surface potential [30]. It is also in consonance with observation of Saad *et al.* (2010), in which the de-charging behaviour of a CYTOP electret with higher initial surface potential is more pronounced than the one of lower value under the same storage conditions [5]. In this light, conversely, sample 3 which attained the least initial surface potential exhibits worse stability behaviour compared to sample 1 probably because of its usage humidity (68% R.H.) which is much higher than that of sample 1 (59% R.H.). High humidity tends to damage CYTOP electret stability as also shown in the investigation of Wang and Hansen (2013) [26]. Charge relaxation in polymer films is influenced by their surface conductance, which increases by several orders in humid medium.

Fig. 8 also shows the surface potential decay pattern of sample 4 that was in storage for the same number of 52 days. It exhibits better stability pattern than all the samples that were used in micro power generation, with an initial rapid short term decay of about 3 days, after which it reduces almost linearly with time. At the end of the 52 days, its surface potential has reduced to 77% of its initial value. Comparing with the investigation of others on CYTOP storage charge stability as in [5], [9], [22], [26], [29], [30]), a better storage stability pattern is expected than what sample 4 exhibits, given the initial surface potential. This may be due to its high storage humidity (72% R.H.). As reported by Wang and Hansen (2013), inorganic electret materials are able to keep surface charges more stable than CYTOP electret with increasing humidity [26]. Another reason could be the charging process. As reported by Chiu and Lee (2013) that, improved charge stability of electrets can be obtained if additional charging cycles are included in the charging process [31]; therefore, it is believed that, improved stability pattern can be obtained for sample 4 (and the other three samples) relatively if longer charging time is used.

Another significant observation in Fig. 8 is the marked difference between the surface potential decay pattern of the electret samples that were used in micro power generation, and sample 4 that was in storage. For example, considering sample 1 which shows the best performance of the three samples put to use, and sample 4; the difference in the percentage of the potentials with respect to their initial values at the end of the 52 days is (77-58) %, that is 19%; which is not insignificant. This shows that CYTOP electret charge stability deteriorates faster when in continuous use for power generation compares to when in storage; and probably by extension, other types of

electrets. Though this behaviour is not theoretically expected as electrets are the equivalent of permanent magnets in electromagnetic applications, yet this phenomenon needs to be further investigated and better understood. Part of the cause may have to do with leakages from parasitic and stray capacitance in the micro power generation set up which might as well be generator design – dependent. There are applications in which electrets have been in use as a form of micro electrical power generator, but most of the time, the electrets in them are in storage condition; and the periods of generation of electricity are intermittent. An example is electret microphones which can remain operational for a couple of years. These days, electrets are gaining more interest in outdoor ubiquitous devices that can harvest energy/produce power from environmental vibrations continuously for MEMS/NEMS devices. Therefore, this aspect of its continuous long term usage stability in micro energy harvesting, when compared to its electromagnetic counterpart requires further investigations.

4 CONCLUSION

Thin film fabrication of CYTOP (CTL-809A) electret; its charging using corona charging method; and its long term surface charge stability under different conditions of humidity when in continuous use in micro power generation for energy harvesting, and in storage have been presented. This work revealed that within the limits imposed by the grid voltage, the initial surface potential depends on the corona discharge voltage; and the higher the initial surface potential, relatively the lower the charge stability. Also, this work showed that CYTOP electret stability depends to a great extent on environment humidity. CYTOP electrets perform poorly when used or stored in high humid medium.

In literature, CYTOP electrets have been known to display good storage charge stability with time. In this work, the surface charges of the fabricated CYTOP electrets deteriorated with continuous use in micro power generation setup more than in storage, which could be due to effect of parasitic and stray capacitance in the setup. Further investigations are necessary in order to understand this behaviour better. Also, further research on improvement in CYTOP electret fabrication and charging towards longevity of the surface charges is recommended.

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