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Charging and characterization of non-patterned organic micro electret arrays

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Abstract. This paper presents the charging and characterization of organic micro electret array for vibration energy harvesting. Micro sized electrets with stable and high surface potential are essential for long-term effectiveness of micro electrostatic power generators which harvest low-level vibration energy. In this study, a localized corona charging method to obtain micro sized electret areas on non-patterned macro sized dielectric thin film has been proposed to solve the problem of low charging efficiency and fast charge decay of micro sized electrets. This method involves using a shadow mask to transfer charge patterns and a triode corona charging configuration to facilitate charge penetration. Charging efficiency of 93.6% is achieved on an electret area of 100µm × 100µm, and 87% of its initial surface potential remains on the shrunken area of 50µm × 50µm after 240 days of storage. A technique combining SEM surface topography and non-contact measurement of the average surface potential has been developed to map the charge distribution on locally charged dielectric thin film and measure the surface potential on the micro sized charged area by incorporating the layout characteristic of an micro electret array.

1. Introduction

Vibration energy harvested from the ambient environment offers a promising sustainable alternative or complementary source of power for electronic devices and systems, such as wireless integrated sensor (WINS) nodes that require low power consumption [1]. Among the power generators based on one of the three major energy conversion principles, i.e. electrostatic, electromagnetic, piezoelectric, the electrostatic ones have the advantage of easy implementation with silicon micromachining technology for batch fabrication [2]. In addition, by integrating electrets, which are dielectric material implanted with charges, into the device, electrostatic power generators are fully self-sustaining without the need of charge pumps [3].

In order to harvest energy from low-level vibration sources with small acceleration (<1g) that are ubiquitous in the environment [4], the size of capacitive electret cells in the power generator devices is designed from dozens to hundreds of micro meters to produce capacitance change at small travel length [5-7]. One commonly adopted method for charging micro sized electrets is to pattern dielectric thin film into micro sized arrays and then to implant charges by corona charging [8-10]. However, low charging efficiency and fast charge decay have been reported[11]. Leonov et al [12] have reported that a charging efficiency of less than 10% can be achieved on 500 µm wide SiO₂ strips. For strips that are narrower than 400µm, it is impossible to effectively implant charges into the material. It has also been
reported that even if charges could be successfully implanted in strips narrower than 1mm, they would completely drain after two days. Another way to charge micro sized electrets is to charge selected electret area on dielectric thin film without patterning the material. The emphasis of the reported work on selective charging has been placed on achieving high surface potential after charging. In the work reported by Naruse et al [13], charging is aided by aluminium electrodes patterned on top of certain areas of SiO₂ thin film. During charge implantation, it is observed that the charges in areas with aluminium electrodes tend to leak, and the charges in areas without aluminium electrodes remain. This selective charging method results in higher surface potential than the conventional charging-after-patterning method. Another way to guide charges into the selected area can be found in [14], in which the silicon mass of the inertial power generator is used as the charging grid. Local charging on a whole piece of CYTOP polymer thin film is conducted after the device is assembled. This charging process sacrifices the mass which is a crucial factor for the power generation of inertial power generators, as the mechanical energy available to be converted into the electrical energy is proportional to the mass. Etching away a portion of the mass material to create slits not only decreases the weight of the mass, but also increases air damping that prevents the mass from vibrating. Wang et al [15] conducted selective charging by selectively treating the whole charged SiO₂ electret thin film surface with an FDTS layer, turning the electret surface hydrophobic and protecting the charges from later exposure to humidity. In these various methods of selective charging, the charging efficiency still needs further improvement. In addition, the characterization of the stability of micro sized electrets in long-term observation is of concern and in need of more investigation and study, because the instability of micro sized electrets will inevitably impair the harvesting effectiveness of electret power generators deployed for long-term use.

The objective of this work is to achieve high charging efficiency and obtain stable micro sized electret arrays by a proposed triode localized corona charging system. The charge distribution and charge migration of micro sized electret arrays over the long term will be discussed. Our work has manifested the application of this localized charging method in the fabrication of micro electret power generators and prototype testing [16].

2. Methodology

2.1 The modelling of charging micro sized electrets

Electrets can be obtained either from inorganic material such as SiO₂/Si₃N₄ multilayers, or organic dielectric material, such as polymer material CYTOP, PTFE, and Teflon AF [17]. Inorganic thin films can be either thermally grown [18, 19] or deposited via plasma-enhanced chemical vapour (PECVD) [20]. However, the low deposition speed and high residual stress of these techniques lead to difficulties to prepare thick inorganic layers (>2µm). Organic electrets can be easily manufactured with a thickness more than 10µm, which is beneficial for maintaining low parasitic capacitance in power generator devices [21]. In order to analyse the cause of low charging efficiency and fast charge decay occurring in the charging process of micro sized electrets, we conducted the modelling of the electric field inside and outside of micro sized dielectric material during charging, as the movement of charges is directed by the electric field.

Figure 1 shows a corona charging system on which an ANSYS model is based. The charging voltage \( V \), applied on the metal grid for uniform charging is 600V. A two dimensional 8-node element of Plane 121 which depicts the cross section of dielectric material is meshed by a mapping scheme. The thickness \( d \) of the dielectric material is set at 50µm. The length \( L₀ \) of the material is allowed to vary while the gap between the grid and the surface material is set at 200µm. Since the dielectric constant of organic material commonly used for power generators is around 2 (2.2 for parylene HT\(^{\text{®}}\), 2.1 for CYTOP, 1.9 for Teflon AF and 2.1 for PTFE) [22], the dielectric constant \( \varepsilon \) is set as 2 in the model. In the model, \( E₀ \) denotes the central surface electric field inside the material, while \( E_f \) refers to the fringing field near the edge of the material as shown in figure 1(a). The electric field gradient across the material, \( \Delta E \),
is determined by $\Delta E = E_f - E_c$, a difference between the central surface electric field and the fringing field. The electric fields are investigated in two conditions, uncharged and slightly charged. The uncharged condition is the initial stage of charging when the surface potential $V_s$ on dielectric material is equal to 0. In the uncharged condition, the central surface electric field is $E_{c,0}$, and the fringing field is $E_{f,0}$. In the slightly charged condition, a small amount of charges are implanted into the shallow surface of the material and the surface potential is assumed as 30V. The central surface electric field and the fringing field change to $E_{c,30}$, and $E_{f,30}$, respectively. Figure 1(b) shows the electric field distribution in and out of electret material when $L_0$ is 100μm.

Figure 1. (a) Corona charging model; (b) Modelled electric field distribution for dielectric material with $L_0$ of 100μm

Percentage change of central surface electric field, $\rho$, is represented by $(E_{c,30} - E_{c,0})/E_{c,0}$. Negative $\rho$ indicates a drop of central surface electric field, whereas positive $\rho$ indicates an increase of central surface electric field. Figure 2 plots $\rho$ with respect to $L_0$. If the value of $L_0$ is greater than 400μm, $\rho$ has a positive value. This indicates an increase in the central surface electric field owing to the accumulated charges. If $L_0$ is reduced to a size less than 400μm, $\rho$ registers a negative value, and the central surface electric field inside the material records a drop owing to charge accumulation on the surface.
Figure 2. Percentage change of the central surface electric field $E_c$ as a function of the length, $L_0$, of dielectric material during charging

Figure 3 shows the electric field gradient, $\Delta E$, as a function of $L_0$. The electric field gradient is observed to decrease with $L_0$ in both charged conditions. A smaller electric field gradient is desired during charging, as a large electric field gradient generates a fringing field that is stronger than the central electric field. This limits the charge implantation, as the stronger fringing field would either divert the majority of the incoming charges to the material edges or to the surrounding air. This restricts the charges from penetrating deeply into the bulk of the material. This therefore accounts for the low charging efficiency in the process of charging micro sized dielectric material. In addition, the close proximity of the charges at the edges to the surrounding air in micro sized dielectric material would cause neutralization with the atmospheric ions, leading to charge leakage from the material during storage.

From the above analysis, the inference is that $\rho$ and $\Delta E$ are size-dependent. Macro sized (>1000µm) dielectric material is more effective than micro sized material (<100µm) in capturing and holding charges in the charging process, and selectively charging micro sized areas on macro sized dielectric material will reduce the size effect. Hence our proposed charging method will be based on this mechanism.
2.2 The triode localized charging system

In order to achieve high charging efficiency, two properties of organic dielectric material are considered, namely density of trap level and dielectric strength. A high density of trap level in dielectric material denotes that there are a significant number of traps per unit area. This material would have a high capacity to trap more charges thereby providing a higher charge density or surface potential. Dielectric strength of the material is another important property as it relates to the amount of charges a material can hold before electric breakdown occurs. According to Table 1, LDPE (low density polyethylene) is found to have the highest density traps and dielectric strength amongst commonly used polymer materials for electrets. LDPE is also readily available and more environmentally friendly, compared with soluble fluoropolymers (CYTOP, PTFE and Teflon AF) which emit toxic substances when heated up during the production cycle [23]. Hence LDPE, in thin film form, purchased from Goodfellow Cambridge Limited, is chosen as the electret material for this work.

<table>
<thead>
<tr>
<th>Polymer material</th>
<th>Electron trap density $N_{et}(\times10^{18} m^{-3})$</th>
<th>Hole trap density $N_{ht}(\times10^{18} m^{-3})$</th>
<th>Dielectric strength $E_{m}(MV/m)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>PTFE (Polytetrafluoroethylene)</td>
<td>0.6797 [24]</td>
<td>0.5768 [24]</td>
<td>18 [26]</td>
</tr>
<tr>
<td>CYTOP</td>
<td>/</td>
<td>/</td>
<td>110 [26]</td>
</tr>
<tr>
<td>Teflon AF</td>
<td>/</td>
<td>/</td>
<td>21 [26]</td>
</tr>
</tbody>
</table>

Before charging, macro sized LDPE thin films are first manually cut from the LDPE sheet. Figure 4 shows the schematic configuration of the proposed triode localized positive corona charging system. During the charging process, the LDPE thin film is sandwiched between a shadow mask (top) and a silicon substrate (bottom) with patterned gold electrodes. Each gold electrode on the bottom silicon substrate is mapped to the shadow mask opening. To ensure that charges are uniformly implanted into micro sized areas, the LDPE thin film should be fully flat on the substrate plate. A 5 µm thick spun-on epoxy layer is used as a bonding adhesive layer between the LDPE thin film and the silicon substrate plate. After charging, the shadow mask is removed and can be repeatedly used for charging. DC voltage supply is set at 10 kV and connected to a Beryllium copper needle of high electrical conductivity and low resistivity of 6µΩ/cm to ionize the air. The distance between the needle tip and the shadow mask is set at 5mm. The shadow mask is biased by a positive voltage $V_c$ which is equal to the targeted surface potential on electrets. The holes under shadow mask openings have vertical sidewalls and are conductive. This is to make sure ions arriving at the shadow mask can move through the holes and implanted into the thin film below the shadow mask. The back side of the thin film is biased by a negative voltage $V_e$, for the purpose of facilitating the charge movement inside the thin film. It should be noted that the charging system can be changed to a negative triode corona charging system by switching the polarity of voltages.
In this localised charging method, the shadow mask will be made of silicon. This is because the shadow mask fabricated from silicon wafers are rigid and flat and can be micro machined to a good degree of accuracy by MEMS technology. The thickness of the silicon shadow mask is kept thin (200µm) and yet has adequate strength to deal with handling and fragility problems. After a DRIE process of creating through-holes, a sputtering process is then applied to deposit Cr(20nm)/Au(300nm) layer on the surface of the mask facing the needle and the sidewalls of through-holes. Compared with the evaporation process, sputtering is preferred as it has a better step coverage, faster deposition rate, better uniformity, and better adhesion to the substrate [27]. Figure 5 shows SEM images of a sputtered shadow mask designed with openings of 100µm × 100µm. The metallic layer, which is the bright area in the images, covers the surface of the shadow mask and the side wall of through-holes.

![SEM images](image)

**Figure 5.** (a) SEM image of the top view of a shadow mask designed with an array of 100µm × 100µm openings; (b) SEM image of the cross section of through-holes in the shadow mask

### 3. Experimental

#### 3.1 Evaluation of the triode corona charging system

In the triode system for localized charging, \( V_e = -V_c \) will be applied. By biasing the dielectric material, the electric field crossing the material is larger than the value when the dielectric material is merely grounded. To examine the effect of the larger electric field on the trap level occupation of charges, experiments are carried out to globally charge samples of 10 mm × 10 mm × 15 µm. Globally charged samples will have the whole surface areas exposed for charge implantation by using a metal grid which is biased by the charging voltage. Two charging voltages are applied: 1kV and 2kV. Samples are charged for 30 minutes under...
different conditions (S1-S4). The initial surface potential $V_0$ of samples are then measured immediately after charging and presented in Table 2. The samples are stored in an environment with a constant temperature of 25 °C ±2°C and a humidity of 48%-51% Rh. The surface potential is measured using a noncontact electrostatic voltmeter (Model 542, Trek).

**Table 2. Initial surface potential $V_0$ on samples charged under varied conditions**

<table>
<thead>
<tr>
<th>Charging conditions</th>
<th>S1: $V_c=1$ kV, $V_e=0$ kV</th>
<th>S2: $V_c=1$ kV, $V_e=-1$ kV</th>
<th>S3: $V_c=2$ kV, $V_e=0$ kV</th>
<th>S4: $V_c=2$ kV, $V_e=-2$ kV</th>
</tr>
</thead>
<tbody>
<tr>
<td>$V_0$</td>
<td>630V</td>
<td>800V</td>
<td>1300V</td>
<td>1700V</td>
</tr>
<tr>
<td>$V_0/V_c$</td>
<td>0.63</td>
<td>0.8</td>
<td>0.65</td>
<td>0.85</td>
</tr>
</tbody>
</table>

Table 2 shows that charging conditions S3 and S4 result in higher surface potentials on samples than S1 and S2, due to the higher charging voltage $V_c$. Sample (S2) and sample (S4) charged with a larger electric field have higher charging efficiencies $V_0/V_c$ than the other samples (S1 and S3). The initial charge stability of the various samples is plotted in figure 6. The surface potential has been normalized against the initial value of each sample and is expressed as a ratio of $r$.

![Normalized Surface Potential Decay](image)

**Figure 6. Normalized surface potential decay in the first 600s**

A model based on the isothermal relaxation current theory in reference [28] is applied numerically in the initial charge decay stage to determine the distribution of trapped charges at different energy levels after charging. At constant temperature, the decreasing current $I_c(t)$ inside the electret caused by charge detrapping relates to the decay of surface potential: $I_c(t)=CdV_s(t)$, where $C$ is the capacitance of electret. The current density $J$ therefore is written as follows:
\[
J = \frac{\varepsilon_0 \varepsilon}{d} \frac{\partial V_x(t)}{\partial t}
\]

(1)

Where \( \varepsilon_0 \) is the dielectric constant of vacuum and \( \varepsilon \) is the dielectric constant 2.2 of LDPE[25]. The energy level \( E_t \), and the density of the trapping energy levels \( N(E_t) \) and \( J \) can be derived from the following equations [24]:

\[ E_t = kT \ln(\gamma t) \]  

(2)

\[ J = \frac{qd kT}{2t} f_0(E_t) N(E_t) \]  

(3)

Where \( f_0(E_t) \) is the initial occupancy of trapping levels, assuming as 1/2, \( q \) the electron charge, \( k \) the Boltzmann’s constant, \( \gamma \) the attempt-to-escape frequency, usually of the order \( 10^{10} \) to \( 10^{12} \) sec\(^{-1} \) [28], assumed as \( 10^{11} \), \( T \) the temperature, and \( t \) is the time.

Based on the measurement of surface potential in figure 6, the density of trapping levels are plotted against the energy level of trap based on the above three Equations (1), (2) and (3). Graphs in figure 7(a) and (b) show the distribution of trap density in samples over a range from 0.8eV to 0.9eV, where energy trap levels of LDPE are located [29]. By performing the integral of the density of the trapping energy level, the number of traps occupied by charges are determined and listed in the column graph figure 7(c), in which S3 and S4 charged with 2kV have more trap levels occupied than S2 and S1 charged with 1kV. S2 and S4, charged with a larger charging electric field, are also found to trap more charges than S1 and sample S3, resulting in higher surface potential.
Figure 7. (a) Density of the trapping energy level as a function of the energy level of trap of samples charged under S1 and S2 conditions; (b) Density of the trapping energy level as a function of the energy level of trap of samples charged under S3 and S4 conditions; (c) Number of occupied trap levels in different samples

3.2 Characterization of micro sized electret arrays
Charge distribution on electret is an important characteristic for studying the stability of electret. The conventional approach to map charge distribution is to conduct a two-dimensional line scan of discrete points of surface potential on electret thin film by means of a non-contact voltmeter probe[15, 22, 30]. This approach can only obtain a low resolution contour map of charge distribution due to the spatial resolution limited by the probe’s aperture size that is in the range of hundreds of micro meters to millimetres. For a spot electret area of diameter 100µm and less, this scanning method would not be able to provide good measurement of the actual surface potential on the micro sized electret area or the accurate charge distribution.

To overcome the problem of low resolution, SEM (scanning electron microscope), which is a commonly used metrology method to image fine details of surface structure of micromachined parts, is utilized for mapping charge distribution of an micro sized electret array. Electret samples are devoid of sample preparation (e.g. metal coating) for the SEM imaging process. The existing charges in the pre-charged sample will interact with the incident electrons and affect the produced secondary electrons collected for imaging. Figure 8 illustrates the imaging process of a locally positive-charged sample. The incoming incident electrons are neutralized on the charged areas, and therefore the induced secondary electrons are diminished. Figure 9 presents the SEM images of 1cm×1cm×50µm LDPE samples either negative-charged (figure 9 (a) and (b)) or positive-charged (figure 9 (c) and (d)) by a voltage of 900V, using shadow masks with different opening sizes. The dimension of charged areas is very similar to that of designed opening areas.
Figure 8. Schematic diagram of a scanning electron microscope (SEM) applied to map charge distribution on locally positive-charged sample.

Figure 9. SEM images of charge patterns on LDPE thin films (a) opening area designed as 200µm × 200µm, $V_{acc} = 5$ kV; (b) opening area designed as 50µm × 100µm, $V_{acc} = 1$ kV; (c) opening area designed as 200µm × 200µm, $V_{acc} = 5$ kV; (d) opening area designed as 100µm × 100µm, $V_{acc} = 1$ kV.

The images are taken immediately after charging. Acceleration voltage $V_{acc}$ of 5kV is applied in the imaging process of samples of figures on the left side, while $V_{acc}$ of 1kV is applied in figures on the right side. In the SEM imaging process, a bigger difference in the amount of...
secondary electrons induced from charged areas and non charged areas can create a higher contrast. It can be seen that SEM images taken under small $V_{acc}$ of 1kV (figure 9(b) and (d)) have higher contrast between charged and non-charged areas than images taken under relatively higher acceleration voltage $V_{acc}$ of 5kV (figure 9(a) and (c)). Fast disappearance of patterns is also observed when $V_{acc}$ of 5kV is applied. This is because a strong electron beam when high $V_{acc}$ is applied reduces the difference between the amount of secondary electrons generated from charged and non-charged areas and makes it difficult to enhance the contrast of the fine boundary of charged and non charged areas, and the quick electron charge-up can easily destroy the original charge pattern on the sample. Moreover, we also found that acceleration voltage lower than 0.5kV makes it difficult to capture images due to insufficient difference in secondary electrons. Therefore, for characterization of charge patterns on micro sized electrets with surface potential around several hundred volts which is often applied in micro electret power generators, acceleration voltage around 0.5~1kV is suggested. It should be noted that the image must be taken immediately after the focus of electron beam. This is because the focused area will get charged by the incoming electron beam within a few minutes, which destroys charge patterns.

As the aperture of voltmeter covers charged and non-charged areas, the surface potential registered in the voltmeter is the average value over the covered area, and this value is a function of the actual surface potential on charged areas and the area ratio factor which reflects the relation between charged and non-charged areas. To verify this relation, we conduct global charging and localized charging on samples with the same size (1cm × 1cm ×50µm) by the same charging voltage $V_c$ of 900V for 1 minute. Globally charged samples having the whole surface area exposed for charging are used as references. For the locally charged samples, a shadow mask with a total number of 2965 openings (100µm×100µm), spaced 160 µm apart, is used. The area ratio of the total area of locally charged areas to the whole area of LDPE thin film is 3.37. The voltmeter gives an average surface potential reading, $V_{as}$, of 250V from the locally charged samples immediately after charging. The surface potential $V_s$ on samples globally charged is measured to be 800V. Voltage ratio of $V_s$ to $V_{as}$ is equal to 3.2. This similarity between the voltage ratio and the area ratio has proven that the voltmeter registers the average surface potential on the thin film. Area ratio, together with measured $V_{as}$ can be combined to derive the surface potential on the micro sized electret area. The derived $V_s$ on 100µm×100µm area is 842V, and the charging efficiency is therefore 93.6%

Samples charged by 900V for 30 minutes are stored for long-term observation. After 20 days of storage, figure 10(a) shows that the shape of the charge patterns remain in tact but with a reduced length of 95µm. The area ratio becomes 3.8, resulting in a surface potential of 800V on the charged areas, according to a measured $V_{as}$ of 210V. Figure 10 (b) shows the SEM image of a sample taken after 240 days of storage. The charges remain concentrated in the locally charged areas but with a side length of 50µm. The surface potential on the 50µm×50µm area is 730 V, computed by multiplying measured $V_{as}$ of 60V and area ratio of 12.1, representing 87% of its initial surface potential.
Figure 10. SEM images of locally charged samples: (a) 20 days after charging; (b) 240 days after charging [31]

Unlike charges in globally charged electrets where they migrate laterally due to the self-field [32], charges in locally charged samples are not found to have such movement. This could be owing to the distance separation in micro meters between the charged regions; and the electric field distributed within these charged regions prohibits charge migration and leakage at the surface. It is also found that charges retain more at the centre than at the edges of the locally charged areas. This could be explained by the fact that, during charging, moving charges in the centre of the thorough-holes of the shadow mask have higher acceleration energy than moving charges close to the thorough-holes’ sidewall, which exerts a repelling force on charges and results in a loss of the acceleration energy of charges. Therefore, those charges close to the sidewall tend to reside at the shallow parts of the material surface. As such, they are more easily displaced, released and neutralized by atmospheric ions. The results of charges stability provide a motivation for future work to optimize the localized charging system, by considering the influence of the dimension of openings and the charging voltage on the charge motion and charge stability.

4. Conclusion
This work has reported the charging and characterization of micro sized electret arrays for energy harvesting application. Charging efficiency of 93.6% has been achieved by localized charging through the use of a shadow mask to transfer charge pattern, and the use of a triode corona system to facilitate charge penetration. SEM surface topography combined with non-contact measurement of average surface potential is employed to map the charge distribution on locally charged dielectric thin film, observe long-term charge migration, and measure the surface potential on the micro sized area by incorporating the layout characteristic of electret arrays. For a charged micro sized electret array of 100µm×100µm on 1cm×1cm×50µm LDPE thin films, 87% of the initial surface potential remains on the shrunken area of 50µm × 50µm 240 days after charging.

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