# Improvement of the Performance of the PECVD SiO<sub>2</sub>/Si<sub>3</sub>N<sub>4</sub> Double-layer Electrets

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## ABSTRACT

In this paper, multiple approaches were attempted to improve the performance of PECVD SiO<sub>2</sub>/Si<sub>3</sub>N<sub>4</sub> double-layer electrets on glass, including different electrode structures, as well as various corona charging conditions. Both chargeability and charge stability of the PECVD SiO<sub>2</sub>/Si<sub>3</sub>N<sub>4</sub> double-layer electret at high temperature and high humidity were investigated. It is proven that samples with both lower and back electrodes has simpler process but exhibits similar chargeability and charge stability to those having lower electrode contact with ground directly by welding lead. High charging temperature can increase chargeability but has no effect on charge stability melioration. Long-term observation implies that if the electrets have to work outside properly, heat treatment will be necessary, although hermetic packaging is the best option. PECVD SiO<sub>2</sub>/Si<sub>3</sub>N<sub>4</sub> double-layer electrets can endure high temperature lower than 250°C and shows good charge stability under high humidity.

Index Terms —PECVD, SiO<sub>2</sub>/Si<sub>3</sub>N<sub>4</sub> double-layer, electret.

#### **1 INTRODUCTION**

**DUE** to their stable charge storage and other useful properties, electrets are widely applied in electric-acoustic transducers [1], biomedicine [2], electrostatic power-generators [3], and etc. Silicon-based inorganic electrets, such as SiO<sub>2</sub>, Si<sub>3</sub>N<sub>4</sub> and SiO<sub>2</sub>/Si<sub>3</sub>N<sub>4</sub> double–layer, have been well studied [4-6] for their high charge stability and compatibility to IC (integrated circuits) and micromachining technologies. Especially, SiO<sub>2</sub>/Si<sub>3</sub>N<sub>4</sub> double–layer electrets have been proven to have better performance [7].

Commonly SiO<sub>2</sub>/Si<sub>3</sub>N<sub>4</sub> double–layer is prepared by thermal oxidation or LPCVD on silicon substrates. Compared to these high temperature deposition methods, PECVD has its own advantages of relatively high deposition speed, low temperature, low residual stress and compatibility with both silicon and non-silicon substrates. In the past, several works have reported the PECVD silicon-compound single layer (silicon nitride, oxide, and oxynitrides) electrets, mainly focusing on the influence of the deposition parameters and the mechanical stress [8], the chemical composition [9], and the storage and transport of charges [10]. They have proved the good chargeability and stable charge storage of PECVD silicon-based electrets.

In all of those documents, whether the silicon-based inorganic electrets were prepared by LPCVD, thermal oxidation or PECVD, semiconductive silicon was the only substrate. Our previous paper firstly proposed  $SiO_2/Si_3N_4$  double-layer both prepared by PECVD on insulating glass

substrates [11], where the sample structure was glass/Cr/Au/SiO<sub>2</sub>/Si<sub>3</sub>N<sub>4</sub> (as shown in figure 1 (a)). However, our former studies shown that the surface potential was not uniform among the whole sample and lack of regularity. Afterward, it was found that the surface potential decreased significantly when SiO<sub>2</sub> was thicker than  $2\mu m$ , which was contrary to our expectation. Careful observation shown that thick SiO<sub>2</sub> layer make the substrate buckle due to large



Figure 1. Schematic graphs of samples with different electrode structures: (a) floating lower electrode only; (b) floating lower and grounded back electrodes; (c) grounded back electrode only; (d) grounded lower electrode only.

residual stress forming a small gap between the substrate and ground plate of the charging setup. This indicates that the bad ground contact induced the random non-uniform surface potential and reduced the surface potential further. Therefore, an Al thin film was added as back electrode (i.e. Al/glass/Cr/Au/SiO<sub>2</sub>/Si<sub>3</sub>N<sub>4</sub> as new sample structure shown in Figure 1 (b)) to achieve better contact between the sample and

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the ground in our following papers. The basic properties and charging process optimization of sample with this new electrode structure were studied and reported in this paper.

# 2 PREPARATION OF SAMPLES

10 cm (4 inch) Pyrex-7740 glass wafers (whose resistivity is  $1.2 \times 10^9 \ \Omega$  cm at 350 °C and  $1.6 \times 10^{11} \ \Omega$  cm at 250 °C indicating they are insulator when the temperature is lower than 350 °C) were used as substrates. Cr (30 nm) and Au (100 nm) as lower electrode were sputtered at first. Then, SiO<sub>2</sub> (1  $\mu$ m) and Si<sub>3</sub>N<sub>4</sub> (50 nm) were deposited by PECVD. SiO<sub>2</sub> was deposited at a speed of 120 nm/min and Si<sub>3</sub>N<sub>4</sub> at a speed of 15 nm/min with substrate temperature of 300 °C. Finally, Al (500 nm) as back electrode was sputtered on the back of the substrates. All these wafers were cut to 3cm×3cm pieces as charging samples. For comparison, the samples with different electrode structures as shown in Figures 1a, 1c and 1d were also prepared using part of above processes. For sample as shown in Figure 1d, the  $Si_3N_4$  and  $SiO_2$  at the corner were removed by wet-etching, and then a lead line was soldered on Au lower electrode and connected to the ground directly.

Before charging, all samples were ultrasonically cleaned and dehydrated for one hour at 150 °C. Then, they were stored in silica gel desiccators at room temperature to be insulated from moisture and contaminants. The negatively corona charging equipment is as schematically shown in Figure 2. As for a traditional tip-grid scheme, the grid voltage is used to limit the surface potential. All samples were charged with -5.5 kV tip voltage and -400 V grid voltage except for those which were charged with different tip and grid voltages for optimization. The distance between the tip and sample surface is about 15mm, and the grid is 2 mm above the sample. The lower electrode was floating but the back electrode contacted to the ground during the corona charge process. Most of samples were charged at 80 °C for 30min except those for optimizing charging temperature and time.



Figure 2. Schematic view of corona charging equipment.

# **3 MEASUREMENT METHODS**

Surface potential is a commonly used parameter to characterize the charge storage in electrets. In this paper, it was measured by Trek 347 voltmeter. Five points of every sample (as shown in Figure 3) were measured to derive average surface potential.



Figure 3. Schematic view of five measurement points of the sample.

Initial surface potential ( $V_0$ ) just after charging process is usually used to exhibit the chargeability of electrets. However, the floating charges which are not really trapped in the electrets vanish rapidly right after corona charging and then slowly. This phenomenon makes the initial surface potential data less reliable and comparable. According to our previous experiments [11], it was found that 30 min heat treatment at 250 °C just after charging process is enough to neutralize the floating charges. So in this paper, the average surface potential after 30 min heat treatment ( $V_{30}$ ) at 250 °C was commonly referred to as initial surface potential without otherwise specified.

The isothermal charge decay is another common method to observe the charge stability of electrets in a relative short time. In this paper, all samples went through 250°C heat-treatment for 5 h in the oven and the surface potential was measured once every 30mins (V<sub>t</sub>). Normalized data (V<sub>t</sub>/V<sub>30</sub>) were applied to exclude the influence of the initial surface potential.

Charge stability under high humidity condition is also very important to guarantee the reliability of electrets working at different environmental conditions. High humidity charge decaying was operated at 95%RH, 25 °C and the surface potential was measured once every 30mins. According to our previous research [11], the performance of  $SiO_2/Si_3N_4$  electrets in high humidity condition can be significantly improved by heat treatment. Therefore, all the samples were pre-treated at 250 °C for 3h before put into 95%RH chamber. And so the normalized data were obtained by V<sub>t</sub>/V<sub>3hr</sub>.

# **4 RESULTS AND DISCUSSIONS**

## 4.1 COMPARISON OF SAMPLES WITH DIFFERENT ELECTRODE STRUCTURES

In this paper, the substrate was insulating glass instead of semiconductor silicon which is usually used in other works. Electrode plays an important role in corona charging method, especially for insulating substrate. Four different electrode structures as shown in Figure 1 have been prepared and studied: (a) floating lower electrode only; (b) floating lower and grounded back electrodes; (c) grounded back electrode only; (d) grounded lower electrode only. Figure 4 exhibits their isothermal charge of these four types of electrode structures. The sample with neither the lower nor the back electrode was also prepared and tested. It's starting surface potential (V<sub>0</sub>) was much lower than others and faded away rapidly, so its isothermal charge decaying curve is not included in Figure 4.



(b)

Figure 4. Isothermal charge decaying curves of samples with different electrode structures: (a) average surface potential & time; (b) normalized average surface potential & time.

Theoretically the starting surface potential ( $V_0$ ) should be equal to the grid voltage which was 400V in this paper. Samples (b), (c) and (d) had similar  $V_0$  of about 350 V, very close to 400V, but the  $V_0$  of sample (a) was just about 150 V. The back electrodes of sample (b) and (c) and the lower electrode of sample (d) were grounded well during charging process. Whereas, Sample (a) having no electrode directly being grounded brings on charging electrical field decreasing at poor grounded micro-scale areas, so the mean of starting surface potential is much lower than the grid voltage. Therefore, good back or lower electrode grounding can help the electret on the insulating substrate to achieve starting surface potential close to the grid voltage.

The surface potential of sample (c) precipitated from 328 V to 15 V after just 30min isothermal treatment at 250 °C indicating that almost all of charges in sample (c) are floating. Sample (c) only has the back electrode, which means the 500 $\mu$ m glass substrate also accumulates with the SiO<sub>2</sub>/Si<sub>3</sub>N<sub>4</sub> as the whole electret, resulting in much lower charging electrical field at the surface region than (a) and (d). And so the charges couldn't be accelerated and implanted deeply enough into the surface and get trapped there. Providing charging voltage increasing enough, the sample (c) should achieve as good chargeability and charge stability as sample (b) and (d).

Otherwise, sample (a) has initial surface potential  $(V_{30})$  lower than that of (b) and (d) but much higher than (c) and its charge stability is as good as that of sample (b) and (d). It may deduce that the floating lower electrode may elevate the ground position and act as the ground. The sample (b) and (d) have the similar chargeability and charge stability demonstrates this further.

Above all, all samples in the following have the electrode structure of sample (b) whose process is much simpler than that of structure (d).

## 4.2 EFFECT OF CORONA CHARGING CONDITIONS

The corona charging conditions mainly include tip and grid voltages, tip and grid heights over the sample surface, charging time and temperature. For application, it is very important for the electret having not only the high surface potential value but also the good surface potential uniformity. The tip and grid should be as close as possible to the sample surface for achieving the highest possible electrical field under same voltage and avoiding breakdown happened between the grid and the ground at same time. And the tip has also to be far enough from the sample area to achieve the symmetrical electrical field all over. In this paper, the distance between the tip and sample surface is about 15 mm for 10 cm (4 inch) wafer although our sample is just  $3\times3$  cm for economical concerning, and the grid is 2 mm above the sample. The tip and grid voltages, charging time and temperature were studied as follows.

## 4.2.1 CORONA CHARGING VOLTAGE AND CHARGING TIME

Several samples were charged at different tip and grid voltages and different charging times. Their five-point surface potentials were measured as shown in Figure 5. The higher the grid voltage is, the higher the surface potential is (as shown in Figures 5a-5d). The center area potential is higher than the area around, especially at low tip voltage, because the tip electrical field is cone-shaped with the highest strength in the center. As the charging time extended the surface potential had some increase. While, excessive charging time is useless.

Although extending charging time can improve the potential uniformity a little, tip voltage is more crucial. High tip voltage



Figure 5. Surface potential at five points of samples charged for 5mins, 15mins and 25mins with different tip voltages/grid voltages: (a) -4.5kV/-300V; (b) -5kV/350V; (c) -5kV/400V; (d) -5.5kV/400V; (e) -6kV/500V.

can make enough air ionized supplying adequate effective charges with enough kinetic energy to plunge into and diffuse further in the sample surface region. When the tip voltage was up to 5.5 kV, 25 min charging can realize very good potential uniformity, i.e. potential variance of five points is lower than 2 V. However, the break-down property of dielectric thin film draws the upper-limit of grid voltage and that of tip voltage too, because the grid voltage is directly proportional to the tip voltage. When the tip and grid voltages went up to 6 kV and 500 V respectively (as shown in Figure 5e), the center area exhibited lower potential than the margin revealing breakdown happened and spreading from center to margin area.

Thus, all samples in other sections were charged with 5.5 kV tip voltage and 400 V grid voltage for 30min.

#### **4.2.2 CHARGING TEMPERATURE**

Samples were charged at different temperatures of room temperature (RT), 80 and 120 °C for 30min. Their charge decaying was measured at 250 °C and 95%RH respectively. A higher charging temperature resulted in better chargeability (as shown in Figure 6a). This is possibly because the elevated charging temperatures can increase the charge mobility during



**Figure 6.** Charge decay curves of samples charged at different temperatures: (a) isothermal charge decaying at 250°C; (b) high humidity charge decaying at 95%RH.

corona charging process, thus promoting more charges diffusing and captured in traps. Whereas, these samples have same charge stability as shown in the small attached figure of normalized surface potential in Figure 6a implying that higher charging temperature do not lead charges moving to and captured by energetically deeper traps. Additionally, charging temperature has no impact on the charge stability at high humidity too (as shown in Figure 6b). This Phenomenon is similar to the result reported by [13]. Considering our equipment condition and safety, 80 °C was chosen as the standard charging temperature for other samples.

# 4.3 PERFORMANCE OF PECVD SIO<sub>2</sub>/SI<sub>3</sub>N<sub>4</sub> ELECTRET

## 4.3.1 SURFACE POTENTIAL DURING LONG-TERM OBSERVATION

The surface potential of samples charged at typical conditions was observed at room temperature for 38 days and measured once several days. There were three kinds of samples: (I) always kept in silica gel desiccators (about 15%RH) except measuring time; (II) always kept outside without any treatment; (III) always kept outside having 250 °C 3hrs heat treatment after corona charging. The results are shown in Figure 7.



Figure 7. Long-term observation of three kinds of samples: (a) surface potential vs. time; (b) normalized surface potential vs. time.

After 38 days, samples (I), (II) and (III) still have 89%, 61.5% and 95.5% of their initial surface potential. The surface potentials of both sample (I) and (III) descended slowly and linearly, but that of sample (II) decreased rapidly at first 5 days and then the trend slowed down and became linear. The

surface potential of sample (II) dropped at the speed up to 40 V/day, much larger than that of sample (I) and (III). This reveals that being kept in outside environment can impair the charge stability effectively because of large amount of ions and water molecules existing. After several days, its dropping velocity decreased and stabilized at about 1.3V/day. Although sample (III) has the surface potential dropped just 0.3 V/day, much slower than 1V/day of sample (I), it paid more than 150V surface potential decreasing during 250 °C 3hrs heat treatment as a cost. Sample (II) always shows the fastest charge losing than others and lower surface potential than sample (III) after one month. Therefore, if the PECVD SiO<sub>2</sub>/Si<sub>3</sub>N<sub>4</sub> electret has to work outside, proper heat treatment will be necessary.

#### **4.3.2 ISOTHERMAL CHARGE DECAYING**

Due to the excellent stability of  $SiO_2/Si_3N_4$  electrets, the surface potential usually decays too slowly to be observed during a short interval. In order to evaluate the charge stability in a reasonably short time span, isothermal charge decay at



**Figure 8.** Curves of isothermal charge decaying at different temperature: (a) surface potential; (b) normalized surface potential.

high temperatures is often applied, since charge decay can be accelerated by increasing temperatures as charge mobility is increased [12].

Isothermal charge decay at different temperatures, such as 200°C, 250°C and 300°C, was observed here. The results are shown in Figure 8. All three samples show abruptly surface

potentials reducing during the first 30min heat treatment. They lost 21.5%, 36.4% and 64.2% of the surface potential respectively. Provided they have same amount of floating charges, higher heating temperature may enhance the ohmic conduction of the dielectric and accelerate the drift and diffusion of excess charges [14]. Temperature above 300°C can lead to quick and large charge decay to lower than 100V in 90min. There is no big difference of decaying percentages at temperature 200 and 250 °C, but 250 °C brought on much more surface potential decaying. In the other sections, 250 °C isothermal charge-decaying is used to compare charge stability at different conditions.

#### 4.3.3 CHARGE DECAYING UNDER HIGH HUMIDITY ENVIRONMENT

As for electrets materials, there are several discharging mechanisms in high humidity, such as the adhesion of water molecules making the surface conductance increase, penetration of water into the layers causing the neutralization of locally deeper traps and so on [15]. The above discussions are all based on the precondition that the samples are stored in silica gel desiccators, i.e. at a very low humidity environment. However, if not having hermetic package, the adhesion of water molecules to the electrets cannot be avoided. In order to guarantee the performance of electrets devices under different environmental conditions, the charge stability under high humidity conditions was studied and methods to improve it were explored here. After corona charging, the sample was put in a humidistat whose humidity was controlled at 95%RH and the temperature was 25 °C. Surface potential was measured once every half an hour as shown in Figure 9.

Common methods like heat treatment and HMDS process are usually applied to improve the charge stability of inorganic electrets [16]. Therefore, in this section, these methods were also attempted to improve the charge stability of PECVD  $SiO_2/Si_3N_4$  double-layer electret under high humidity conditions: 1. heat treatment after corona charging at 250 °C for various time, such as 30 min, 90 min and 3 h; 2. HMDS treatment after corona charging.

It can be seen in figure 9 that the sample without any treatment would lose about 20% of the surface potential after 6 h at 95% RH. It is well known that there naturally form about 2 nm oxidization thin film at the surface of silicon at room temperature after fresh silicon exposing to atmosphere for 2hr. The composition of the  $Si_3N_4$  was measured by AES (PHI-700) and the results show that the PECVD  $Si_3N_4$  is silicon rich and there is high concentration of O in the beginning, i.e. about 1-2 nm SiO<sub>2</sub> at surface. This naturally generated silicon dioxide layer could have chemical reactions with water molecules adhering to the surface and then would increase the surface conductance.

Both heat treatment at 250 °C and HMDS process would significantly improve the charge stability under high humidity. As heat treatment time increases from 0min to 3hrs the charge decaying percentage decreases significantly from 20% to lower than 1% after 6hrs at 95% RH. HMDS process sample shows just about 6% charge decaying. However, heat treatment leads surface potential losing very much at same time (as shown in the small attached graph of Figure 9). Therefore, 90 min heat treatment at 250 °C for improving its stability is optimum.



**Figure 9.** Normalized charge decaying at 95%RH curves of samples treated by different conditions (attached small graph is charge decaying curves at 95% of samples heat treated at 250°C for different time).

## 5 CONCLUSION

In this paper, multiple approaches were attempted to improve the performance of PECVD  $SiO_2/Si_3N_4$  double-layer electrets on glass, including different electrode structures, as well as various corona charging conditions. With the optimized electrode structure, both the chargeability and charge stability of the PECVD  $SiO_2/Si_3N_4$  double-layer electret at high temperature and high humidity were investigated.

On insulating glass substrates, samples with different electrode structures were prepared and their corona charging performances were compared. Good ground contacting of the electret is the key factor for obtaining the highest possible starting surface potential, i.e. equaling to the grid voltage. The sample with both lower and back electrodes exhibits similar chargeability and charge stability to that having lower electrode contact with ground directly by welding lead. The floating lower electrode between electret and glass may elevate the ground side and act as the ground by itself.

Various corona charging conditions, such as tip and grid voltages, charging time and temperature, were experimented and optimized for better charging performance and uniformity. Higher grid voltage leads to higher surface potential. However, the break-down property of electret dielectric thin film draws the up-limit of grid voltage and that of tip voltage too. As for PECVD  $SiO_2/Si_3N_4$  (1µm/50nm) double-layer electret, grid voltage should be lower than 500V. Although extending charging time can improve the surface potential uniformity, tip voltage is the more crucial factor. When the tip voltage increased up to 5.5 kV 25 min charging can realize very good potential uniformity, i.e. potential variance of five points is lower than 2 V. High charging temperature can increase chargeability while has no effect on charge stability melioration.

At last, the chargeability and charge stability of PECVD  $SiO_2/Si_3N_4$  double-layer electret at high temperature and humidity were experimented and studied. Long-term observation implies that if the electret has to work outside properly, heat treatment will be necessary, although hermetic packaging is the best option. PECVD  $SiO_2/Si_3N_4$  double-layer electrets can endure high temperature lower than 250 °C and shows good charge stability under high humidity. Both the heat treatment and HMDS can improve the charge stability under high humidity but reduce the surface potential at cost.

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