The design of the emission layer for electron multipliers

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Abstract

The electron multipliers gain is closely related to the secondary electron emission coefficient (SEE) of the emission layer materials. The SEE is closely related to the thickness of the emission layer. If the emission layer is thin, the low SEE causes the low gain of electron multipliers. If the emission layer is thick, the conductive layer can't timely supplement charge to the emission layer, the electronic amplifier gain is low too. The electron multipliers usually choose Al\textsubscript{2}O\textsubscript{3} and MgO film as the emission layer because of the high SEE level. MgO easy deliquescence into Mg(OH)\textsubscript{2} resulting in the lower SEE level. The SEE level of Al\textsubscript{2}O\textsubscript{3} is lower than MgO, but Al\textsubscript{2}O\textsubscript{3} is stable. We designed a spherical system for testing the SEE level of materials, and proposed to use low-energy secondary electrons instead of low-energy electron beam for neutralization to measuring the SEE level of Al\textsubscript{2}O\textsubscript{3}, MgO, MgO/Al\textsubscript{2}O\textsubscript{3}, Al\textsubscript{2}O\textsubscript{3}/MgO, and precisely control the film thickness by using atomic layer deposition (ALD). We propose to compare the SEE under the adjacent incident electrons energy to partition the SEE value of the material, and obtain four empirical formula for the relationship between SEE and thickness. Through experiments and calculations, we put forward a new emission layer for electron multipliers, including 2–3 nm Al\textsubscript{2}O\textsubscript{3} buffer layer, 9nm MgO main-body layer, 1nm protective layer or 0.3nm enhancement layer. We can apply this new emission layer to channel electron multiplier (CEM), microchannel plate (MCP), separate electron multiplier.

Key words: electron multipliers; SEE; Al\textsubscript{2}O\textsubscript{3}; MgO; ALD

Introduction

The secondary electron emission coefficient (SEE) of a material is defined as the ratio of the emitted secondary electrons number to the incident electrons number on the material. The application field of secondary electrons is very wide, mainly divided into the field of electron multiplication, the field of material surface composition and structure analysis, and the field of suppressing micro-discharge. The field of electron multiplication includes channel electron multiplier (CEM), microchannel plate (MCP), separate electron multiplier, micro-pulse gun (MPG), dielectric window, atomic clocks, etc [1-9]. The field of material surface composition and structure analysis includes transmission electron microscope (TEM), scanning electron microscope (SEM), auger electron spectrometer (AES), electron diffractometer, etc. The field of suppressing micro-discharge includes the electron cloud problem on the inner surface of the ring-accelerator, the reliability and life of high-power microwave vacuum devices in space, the dielectric window breakdown of high-power microwave sources, the charging/discharging problems on the surface of the spacecraft, etc [1].

Our main research area is the application field of electron multiplication. Electron multipliers consist of the substrate, the conductive layer and the emission layer. The incident electron hitting the emission layer lead to the generation of secondary electron from the emission layer. The secondary electron will be further accelerated by bias voltage to hit the emission layer and lead to more and more secondary electron, resulting in an electron avalanche and the emission of a cloud of electrons from the output. The emission layer lost a
large amount of electric charge due to more and more secondary electron, so the conductive layer for the loss of the electron emission continuously provide the charge [10].

The SEE is closely related to the thickness of the emission layer. If the emission layer is thin, the low SEE causes the low gain of electron multipliers. If the emission layer is thick, the conductive layer can't timely supplement charge to the loss charge of the emission layer due to the electron avalanche, resulting in the low gain of the electron multipliers. The experiment experience that the emission layer between 5 nm ~ 15 nm is appropriate. Therefore, the gain of electron multipliers is closely related to the SEE level of the materials and the thickness of the emission layer. It becomes very important to study the thickness of the emission layer and the SEE level of the materials.

It is known that the SEE level of Al₂O₃ is very high [11]. Therefore, Al₂O₃ is usually selected as the emission layer film in the electron multipliers. But the SEE level of MgO is much higher than Al₂O₃ [2,12]. There are four reasons why MgO was not selected. First, MgO is easy to deliquesce into Mg(OH)₂, which causes the SEE level to become as low as that of Al₂O₃; second, the film will be very thick (35nm) under the saturated SEE level of MgO, the conductive layer cannot replenish charge to the surface of the emission layer in time; third, the properties of Al₂O₃ are stable for a long time in the atmosphere; fourth, the preparation process of Al₂O₃ is simpler than that of MgO.

In order to measure the SEE level of materials, many laboratories around the world have built their own dedicated measuring devices, including the University of Utah [13], Princeton University [14]; University of Science and Technology of China, Xi’an Jiaotong University, 504 Institute of Aerospace, Dongguan Hash Neutron Source, University of Electronic Science and Technology, etc. We designed a spherical system for testing the SEE level of materials, and proposed to use low-energy secondary electrons instead of low-energy electron beam for neutralization, for measuring the SEE level of insulation materials, such as MgO and Al₂O₃. It avoids the disadvantages of neutralization dose and neutralization time.

We compare the SEE value under the incident electron energy of the neighbors, and use this as a standard to divide the material into a low-energy region, a medium-energy region and a high-energy region. It is found that the middle energy region can eliminate the interference of incident electron energy on the SEE value. Therefore, the middle energy region is selected as the standard to measure the SEE level of the material, and Al₂O₃, MgO, MgO/Al₂O₃, Al₂O₃/MgO are studied to obtain the empirical formula. Our experiments and calculations found that after growing Al₂O₃ and then growing MgO, the saturated SEE level of MgO can be revealed when this film is thinner than the MgO film. This solves the problem that the MgO film is too thick and the conductive layer cannot supplement the charge for the emission layer. And we found that after growing MgO and then growing Al₂O₃, Al₂O₃ above 3nm no longer shows the SEE level of MgO; the 1nm Al₂O₃ can resist the damage of the external environment to MgO, and keep the SEE level of MgO for a long time; the 0.3nm Al₂O₃ can raise the saturated SEE level of MgO. Therefore, we propose that the preparation process of the new emission layer is to grow a 9nm MgO main layer on the 2nm Al₂O₃ buffer layer, and then grow 1nm Al₂O₃ protective layer or 0.3nm Al₂O₃ enhancement layer on it, which can solve the problem of the MgO shortcomings of as the emission layer in the electron multipliers. The design thickness of this new emission layer is of great significance for improving the gain and stability of the electron multiplier.

**Experimental and Methods**

**The emission layer using atomic layer deposition**

Atomic layer deposition (ALD) is a kind of technology, which is the precursor gas and reaction gas
alternately enter the basal surface at a controlled rate, physical or chemical adsorption on the surface or surface saturated reaction occurs on the surface, the material is deposited layer by layer in the form of a single atom film on the surface. ALD can produce continuous no pin-microchannel film, have excellent coverage, and can control the atomic film thickness and composition. Therefore, we choose ALD as an important preparation method for studying the thickness of the emission layer. [15,16,17]

The following is the chemical reaction equation of using ALD to grow Al₂O₃:
A: Substrate − OH⁺ + Al(CH₃)₃ → Substrate − O − Al(CH₃)₂⁺ + CH₄ ↑
B: Substrate − O − Al(CH₃)₂⁺ + 2H₂O → Substrate − O − Al(OH)₂⁺ + 2CH₄ ↑
C: Al − OH⁺ + Al(CH₃)₃ → Al − O − Al(CH₃)₂⁺ + CH₄ ↑
D: Al − CH₃⁺ + H₂O → Al − OH⁺ + 2CH₄ ↑

As the equation of A and B or C and D shown, the basal surface was originally covered with −OH. The chemical reaction of −OH and Al(CH₃)₃ (TMA) formed the new −CH₃ surface, and released CH₄ (byproduct). The new −CH₃ surface exposed to water vapor, their reaction generated the new −OH surface and released CH₄ again. The temperature of the reaction is 200 °C. As shown in the figure 1, the time and the order of growing a layer of Al₂O₃ atom:

TMA/N₂/H₂O/N₂ = 0.1~1s/5~45s/0.1~1s /5~45s.

The following is the chemical reaction equation of using ALD to grow MgO:
E: Substrate − OH⁺ + Mg(C₅H₅)₂ → Substrate − O − MgC₅H₅⁺ + C₅H₆ ↑
F: Substrate − O − MgC₅H₅⁺ + H₂O → Substrate − OH⁺ + C₅H₆ ↑
G: Mg − OH⁺ + Mg(C₅H₅)₂ → Mg − O − MgC₅H₅⁺ + C₅H₆ ↑
H: Mg − C₅H₅⁺ + H₂O → Mg − OH⁺ + C₅H₆ ↑

As the equation of A and B or C and D shown, the basal surface was originally covered with −OH. The chemical reaction of −OH and Mg(C₅H₅)₂ (Mg(C₅H₅)₂) formed the new −C₅H₅ surface, and released C₅H₆ (byproduct). The new −C₅H₅ surface exposed to water vapor, their reaction generated the new −OH surface and released C₅H₆ again.

We heat Mg(C₅H₅)₂ at 60 °C to turn it into dust. Temperature of the reaction chamber is 200 °C. As shown in the figure 1, the time and order of growing a layer of MgO atom using Atomic layer deposition:

Mg(C₅H₅)₂/N₂/H₂O/N₂ = 0.1~1s/5~45s/0.1~1s /
The design of emission layer

As shown in the figure 2, the samples are prepared in four ways: grow different thicknesses of Al₂O₃ on Si wafer; grow different thicknesses of MgO on Si wafer; grow different thicknesses of Al₂O₃ on Si wafer and then grow fixed thicknesses of MgO; grow a fixed thickness of MgO on the Si wafer and then grow a different thickness of Al₂O₃.

We have grown different thicknesses of Al₂O₃ on Si wafer (1nm, 3nm, 7nm, 9nm, 30nm, 50nm).

We have grown different thicknesses of MgO on Si wafer (1nm, 3nm, 5nm, 9nm, 15nm, 20nm, 35nm).

We grow different thicknesses of Al₂O₃ on Si wafer (0.6nm, 1nm, 3nm, 30nm) and then grow fixed thicknesses of MgO (9nm).
We grow a fixed thickness of MgO on the Si wafer (35nm) and then grow a different thickness of Al$_2$O$_3$ (0.3nm, 0.6nm, 1nm, 3nm, 5nm, 7nm, 10nm, 20nm).

**The new test method for SEE**

As shown in the figure 3, we use the collector method to measure: first connect the sample stage to the collector, the current measured by the picoammeter is the incident electron current, denoted as $I_p$; under the same incident conditions, disconnect the sample and collector, at this time the measured current on the collector is the secondary electron current, denoted as $I_s$.

$$\text{SEE} = \frac{I_s}{I_p}$$

We designed the device into a global-shaped structure to ensure the full collection of secondary electrons and help improve the accuracy of the measurement results [18].

When the insulating material is bombarded by incident electrons, the surface of the material emits secondary electrons and accumulates positive charges due to the loss of electrons. The positive charge makes the potential rise. Because the secondary electrons are generated within a few nanometers of the material surface and have low energy (~eV). The secondary electrons are very susceptible to the positive potential. The positive potential will affect the next secondary electron emission process leading to a decline in secondary electron yield.

In order to eliminate the effect of charge accumulation on the measurement result of the SEE of the insulating sample and accurately measure the SEE of the insulating sample, the traditional method directly uses a low-energy electron beam to irradiate the insulating sample, and the positive charge on the surface of the sample is neutralized by the low-energy electron. The traditional method has two disadvantages. First, it needs to accurately calculate the neutralization dose, it is easy to have positive charge on the sample surface due to insufficient neutralization dose, or negative charge on the sample surface due to excessive neutralization; second, it needs to be equipped with another one low energy electron gun.

As shown in the figure 4, we propose to use low-energy secondary electrons instead of low-energy electron beam for neutralization, which overcomes the shortcomings of traditional methods and obtains accurate secondary electrons [19]. We place the insulation sample to be tested on half of the sample stage, and leave the other half empty. The sample table is made of 304 stainless steel, and the electric potential is 0V. As shown in the figure 4(a), when testing an insulating sample, the electrons generated by the electron gun bombard the surface of the insulating sample, resulting
in a positive charge area. As shown in the figure 4(b), when neutralizing the surface charge of the insulating sample, the half empty area of the sample stage is bombarded by adjusting the electron gun to make the sample stage emit secondary electrons. Due to the mutual attraction of positive charges and electrons, secondary electrons are attracted to the sample surface for charge neutralization. As the positive charge decreases, fewer electrons are attracted. When the positive charge on the sample surface is neutralized, the surface of the insulating sample returns to its original state. Because there is no positive charge, it will not continue to attract the low-energy secondary electrons generated by the sample stage, so there will be no excessive neutralization that causes the sample surface to be negatively charged.

Result and Discussion

We compare the SEE value under the adjacent incident electrons energy to describe the change of SEE with the energy of incident electrons and define it as

$$R_{\text{SEE}} = \frac{\text{SEE}(x+b)}{\text{SEE}(x)}$$

and the SEE of the material is divided into three areas by the size of the \(R_{\text{SEE}}\) value, namely the low energy region of the incident electron, the medium energy region of the incident electron and the high energy region of the incident electron.

As shown in the figure 5, it can be seen that the \(\text{Al}_2\text{O}_3\) SEE basically remains unchanged after 9nm. As shown in figure 5(a) and 5(b), the low energy region of \(\text{Al}_2\text{O}_3\) is between 100 eV and 250 eV, the \(R_{\text{SEE}}\) decreases from 1.25 to 1, indicating that as the incident electron energy increases, the SEE increases and finally stabilizes. As shown in figure 5(c) and 5(d), the medium energy region of \(\text{Al}_2\text{O}_3\) is between 250 eV and 500 eV,
the $R_{\text{SEE}}$ is considered constant within the interval of $[0.98, 1.02]$, that is, the $R_{\text{SEE}}$ is approximately equal to 1, indicating that the SEE is basically unchanged as the incident electron energy increases. As shown in figure 5(e) and 5(f), the high energy region of $\text{Al}_2\text{O}_3$ is between 500 eV and 1500 eV, for every increase of 200 eV of incident electron energy, the SEE decreases by about 0.9 times.

Because the $\text{Al}_2\text{O}_3$ SEE is stable in the medium energy region, the incident electron energy can be excluded as a variable factor. As shown in figure 6, we choose the medium incident electron energy 300 eV as the standard to measure the SEE level of $\text{Al}_2\text{O}_3$, the empirical formula for the thickness of $\text{Al}_2\text{O}_3$ and the best SEE is obtained by fitting.

$$B_{\text{SEE}}^{\text{Al}_2\text{O}_3} = 3.99 - 2.5 \times e^{-\frac{\text{thickness}}{1.73}}$$ (1)

As shown in the figure 7, it can be seen that the MgO SEE basically remains unchanged after 20nm. As shown in figure 7(a) and 7(b), the low energy region of MgO is between 100 eV and 500 eV, the $R_{\text{SEE}}$ decreases from 1.3 to 1, indicating that as the incident electron energy increases, the SEE increases and finally stabilizes. As shown in figure 7(c) and 7(d), the medium energy region of MgO is between 500 eV and 1000 eV, the $R_{\text{SEE}}$ is considered constant within the interval of $[0.98, 1.02]$, that is, the $R_{\text{SEE}}$ is approximately equal to 1, indicating that the SEE is basically unchanged as the incident electron energy increases. As shown in figure 7(e) and 7(f), the high energy region of MgO is between 1000 eV and 1500 eV, for every increase of 100 eV of incident electron energy, the SEE decreases by about 0.94 times.

Because the MgO SEE is stable in the medium energy region, the incident electron energy can be excluded as a variable factor. As shown in figure 8, we choose the medium incident electron energy 700 eV as the standard

Figure 7, after dividing the incident electron energy by $R_{\text{SEE}} = \frac{\text{SEE}(\times \text{b})}{\text{SEE}(\times \text{c})}$, the change of MgO SEE with thickness.

Figure 8, under the incident electron energy 700ev, the relationship between the SEE of MgO and the thickness.
to measure the SEE level of MgO, the empirical formula for the thickness of alumina material and the best SEE is obtained by fitting.

\[
\text{BSEE}_{\text{MgO}} = 9.56 - 8.64 \times e^{-\frac{\text{thickness}}{7.39}}
\]  

(2)

As shown in the figure 9, The SEE of Al₂O₃/MgO and MgO have similar incident electron energy partition, it can be seen that the SEE of Al₂O₃/MgO basically remains unchanged after 3nm. As shown in figure 9(a) and 9(b), the low energy region of Al₂O₃/MgO is between 100 eV and 450 eV, the RSEE decreases from 1.4 to 1.05, indicating that as the incident electron energy increases, the SEE increases and finally stabilizes. As shown in figure 9(c) and 9(d), the medium energy region of Al₂O₃/MgO is between 500 eV and 1000 eV, the RSEE is considered constant within the interval of [0.98, 1.02], that is, the RSEE is approximately equal to 1, indicating that the SEE is basically unchanged as the incident electron energy increases. As shown in figure 9(e) and 9(f), the high energy region of Al₂O₃/MgO is between 1000 eV and 1500 eV, for every increase of 100 eV of incident electron energy, the SEE decreases by about 0.95 times. Because the SEE of Al₂O₃/MgO is stable in the medium energy region, the incident electron energy can be excluded as a variable factor.

As shown in figure 10, we choose the medium incident electron energy 700 eV as the standard to measure the SEE level of Al₂O₃/MgO, the empirical formula for the thickness of alumina material and the best SEE is obtained by fitting.

\[
\text{BSEE}_{\text{Al₂O₃/MgO}} = 7.94 - 1.21 \times e^{-\frac{\text{thickness}}{1.05}}
\]  

(3)

As shown in the figure 11, The SEE of MgO/Al₂O₃ and Al₂O₃ have similar incident electron energy partition, it can be seen that the SEE of MgO/Al₂O₃ basically remains unchanged after 3nm. As shown in Figure 11(a) and 11(b), the low energy region of
MgO/Al₂O₃ is between 100 eV and 300 eV, the $R_{\text{SEE}}$ decreases from 1.8 to 1, indicating that as the incident electron energy increases, the SEE increases and finally stabilizes. As shown in Figure 11(c) and 11(d), the medium energy region of MgO/Al₂O₃ is between 300 eV and 500 eV, the $R_{\text{SEE}}$ is considered constant within the interval of [0.98, 1.03], when Al₂O₃ is thin, $R_{\text{SEE}}$ deviates from 1, and the difference in SEE under different incident electron energies is obvious; when Al₂O₃ is thick, $R_{\text{SEE}}$ is close to 1, and the difference is not obvious. As shown in Figure 11(e) and 11(f), the high energy region of MgO/Al₂O₃ is between 500 eV and 1500 eV, when Al₂O₃ is thin, $R_{\text{SEE}}$ is close to 1, and the difference in SEE under different incident electron energies is not obvious; when Al₂O₃ is thick, $R_{\text{SEE}}$ deviates from 1, and the difference is obvious; for every increase of 200 eV of incident electron energy, the SEE decreases by about 0.9 times.

Because the SEE of MgO/Al₂O₃ is stable in the medium energy region, the incident electron energy can be excluded as a variable factor. As shown in Figure 12, we choose the medium incident electron energy 450 eV as the standard to measure the SEE level of MgO/Al₂O₃, the empirical formula for the thickness of alumina material and the best SEE is obtained by fitting.

$$B_{\text{SEE}}_{\text{MgO}} = 4.69 + 3.64 * e^{-\frac{\text{thickness}}{2.11}}$$

$$B_{\text{SEE}}_{\text{Al₂O₃/MgO}} = \frac{9.56 - 8.64 * e^{-\frac{9}{7.75}}}{3.99 - 2.5 * e^{-\frac{30}{1.73}}} \approx 1.755$$

According to formula 1 and formula 2, the SEE level of 9nm MgO is 1.755 times higher than that of 30nm Al₂O₃.

$$B_{\text{SEE}}_{\text{Al₂O₃}} = \frac{7.94 - 1.21 * e^{-\frac{3}{1.63}}}{3.99 - 2.5 * e^{-\frac{30}{1.73}}} \approx 1.973$$
As shown in Figure 13(a), we deposit 0~30 nm Al₂O₃ and redeposit 9 nm MgO on the Si wafer as the film. Formula 1 and formula 3 show that the SEE level of 9 nm MgO grown on 3 nm Al₂O₃ is 1.973 times higher than that of Al₂O₃. Formulas 2 and 3 show that the SEE level of 9 nm MgO grown on 3 nm Al₂O₃ is 1.124 times higher than that of 9 nm MgO.

As shown in Figure 13(b), the SEE level of MgO after deliquescent drops significantly. Then we deposit 35 nm MgO and redeposit 1 nm Al₂O₃ on the Si wafer as the film. We found the SEE of this film exposed to the air 7 months is close to the SEE without exposed to the air.

Formula 1 and formula 3 show that the SEE level of 1 nm Al₂O₃ grown on MgO is 1.743 times higher than that of MgO.

As shown in Figure 13(c), we deposit 35 nm MgO and redeposit 0.3 nm Al₂O₃ on the Si wafer as the film. It can be seen from formula 1, formula 2, and formula 4 that the SEE level of 0.3 nm Al₂O₃ grown on MgO is 1.967 times higher than that of Al₂O₃ and 1.12 times higher than that of MgO:

\[
\frac{B_{\text{SEE}}_{\text{MgO}}}{B_{\text{SEE}}_{\text{Al₂O₃}}} = \frac{4.69 + 3.64 \times e^{-\frac{0.3}{39.9} - 2.5 \times e^{-\frac{1}{30.7}}}}{3.99 - 2.5 \times e^{-\frac{1}{30.7}}} 
\approx 1.743
\]

As shown in Figure 13(c), we deposit 35 nm MgO and redeposit 0.3 nm Al₂O₃ on the Si wafer as the film. It can be seen from formula 1, formula 2, and formula 4 that the SEE level of 0.3 nm Al₂O₃ grown on MgO is 1.967 times higher than that of Al₂O₃ and 1.12 times higher than that of MgO:

\[
\frac{B_{\text{SEE}}_{\text{MgO}}}{B_{\text{SEE}}_{\text{Al₂O₃}}} = \frac{4.69 + 3.64 \times e^{-\frac{0.3}{39.9} - 2.5 \times e^{-\frac{1}{30.7}}}}{3.99 - 2.5 \times e^{-\frac{1}{30.7}}} 
\approx 1.743
\]

As shown in Figure 13(b), the SEE level of MgO after deliquescent drops significantly. Then we deposit 35 nm MgO and redeposit 1 nm Al₂O₃ on the Si wafer as the film. We found the SEE of this film exposed to the air 7 months is close to the SEE without exposed to the air. Formula 1 and formula 3 show that the SEE level of 1 nm Al₂O₃ grown on MgO is 1.743 times higher than that of MgO.
the SEE of Al₂O₃, and can be long-term maintain a high SEE level (no obvious deliquescence in 7 months).

As shown in Figure 14, we propose to grow 2nm~3nm Al₂O₃ as a buffer layer, grow 9nm MgO as the main layer, and grow 0.3nm Al₂O₃ as an enhancement layer or 1nm Al₂O₃ as a protective layer as the emissive layer of electron multipliers.

**Conclusion**

We designed a global-shaped structure device for testing the SEE of the material and propose to use low-energy secondary electrons instead of low-energy electron beam for neutralization to measure the insulating material. And we used atomic layer deposition technology to precisely control the growth thickness of the material to study the relationship between different thickness and SEE. We propose the nearest neighbor SEE ratio and use this to divide the SEE incident electron energy of the material into the high-energy region, the middle-energy region and the low-energy region. We have obtained four empirical formulas for SEE and thickness by studying Al₂O₃, MgO, MgO/Al₂O₃, Al₂O₃/MgO, and obtained the optimal Al₂O₃/MgO/Al₂O₃ three-layer structure thickness suitable for electron multiplier through formula analysis. The film with this structure can maintain a high SEE level for a long time.

**Competing interests**
The authors declare that they have no competing interests.

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**Authors’ Contributions**
YMW was the first author and was a major contributor in writing the manuscript. The main idea, experiment, and analysis of this paper are raised by YMW. SL was the corresponding author and YMW’s PhD Tutor, he gave a lot of experimental experience. MQ was the corresponding author and YMW’s PhD Tutor, he gave a lot of data analysis experience. BY and KW gave a lot of experience in testing SEE methods and instrument design. BZ, JG, WY join in the measurements of SEE. All authors read and approved the final manuscript.

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