A Study of Sputtering in Microwave Discharge Neutralizer Using Quartz Crystal Microbalance

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Abstract: In order to further develop the microwave discharge ion engine, it is necessary to extend the lifespan. It is the microwave discharge neutralizer that is rate limiting the lifetime of the ion engine. The neutralizer causes deterioration by sputtering of internal ions. So, it should be coated with a material which is difficult to be sputtered. However, sputtering is a very small phenomenon and the influence observation takes a long time. Therefore, material test in a short time using QCM which can measure mass with ng order is proposed in this study. As a result, it was observed that the mass of QCM decreased at a substantially linear speed and the measurement was sufficiently possible in about an hour. By changing the material of the QCM electrode, various materials are probably tested in less time than before. Furthermore, since QCM measures the effect of ions rather than the whole plasma, it may contribute to understanding phenomena inside the neutralizer.

Nomenclature

S = sputtering yield
α = experimental coefficient of target atom in Wilhelm model
N = density of target atom
M = mass of target atom
m = mass of ion
E_1 = ion energy
E_0 = threshold energy of ion
f_0 = fundamental frequency
A = electrode area
ρ = density of crystal

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μ = shear stress of crystal
df = frequency change from fundamental frequency
dm = mass change
Δf_T = frequency variation due to temperature
t = time
f = frequency
f_{real} = temperature calibrated frequency
$\dot{M}$ = mass loss rate
J = current density
A_{hole} = sidewall hole area
C = coulomb number

I. Introduction

These days, space propulsion technology has developed and spacecraft can reach planets or asteroids. Space exploration to them enables us to get some valuable information, for example, the formation of our world. One of the Space exploration projects in Japan is the asteroid sample return project, the asteroid name is Itokawa and the spacecraft which succeeded the project called "HAYABUSA". The spacecraft's most characteristic point is the engine. The engine is the Japanese microwave discharge ion thruster, μ 10. The ion thruster generates plasma by microwave and accelerates ions with grid whose potential difference of 1.5 kV[1]. This makes the engine possible to obtain a high Isp, about 3000s. One of the features of this ion engine is that the cathode is also operated with microwave. The microwave discharge neutralizer accompanies μ 10. It means that microwave oscillator is just one is fine for the ion engine and neutralizer. So the microwave discharge system can reduce its own weight. The lightweight enables the spacecraft to sail to a distant space ocean. That is why μ 10 is selected to the sample return project.

μ 10 is electrodeless discharge system and it does not need the hollow cathode. So the ion engine has an expectation the long life operation. This is an important feature for the deep space exploration. There are two kinds of failures for μ 10, the one is the ion engine breakdown, and the other is the neutralizer breakdown. Unfortunately, the failure is not derived from the ion engine but rather from the neutralizer. Indeed, the majority of the ion engines installed on "HAYABUSA" could not operate due to the neutralizer breakdown[2]. So, in order to enhance the life of the ion engine, it is necessary to extend the lifespan of the neutralizer. One of the methods of extending the lifespan of it is to be coated by some sturdy material inside of it.

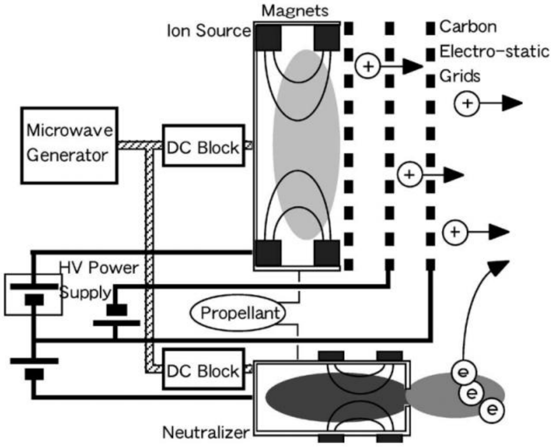

Figure 1. System concept of μ 10[1].

II. Experimental method and setup

A. Microwave Discharge Neutralizer

The neutralizer is a device to emit electrons in order to stabilize the emitted ions. The microwave discharge neutralizer generates plasma by ECR heating like the microwave discharge ion engine. The electrons are extracted by the external positive ion potential, and the inner ions collide with the wall inside the neutralizer, resulting in current loops. Deterioration of the neutralizer depends on the operation time of the ion engine. In fact, three of the four HAYABUSA's ion engines were unable to operate because of the deterioration of the neutralizer. Degradation of the microwave discharge neutralizer is caused by ion sputtering[3]. When ions collide to the wall, sputtering occurs, and atoms on the wall are flushed out. The atoms contaminate the inside of the neutralizer and decrease the microwave transmission efficiency. Then the power supply voltage for operating the neutralizer increases. Finally, the voltage of the neutralizer power supply reaches the upper limit, it becomes impossible to operate the neutralizer.

A method of suppressing deterioration of the neutralizer is not to or less sputter in the neutralizer. The way to do is change the ion energy or material. To change the ion energy, we need to improve the neutralizer, and it will need a big change from a present shape. But to change material doesn't have to change that from now, just to change the surface of it. Then what is the difficulty for the way? It is the time cost. Sputtering is a phenomenon in which atoms are ejected. Therefore, unlike general destruction, it is a minute mass change phenomenon. That means it takes a huge amount of time to measure mass change. For example, the microwave discharge neutralizer coated by the Mo needs 2500hours to measure the mass change and only 17mg declined[4]. In order to select the optimum material, it is necessary to try many materials. However, if thousands of hours on one material spent, there are considerable times to select. Therefore we propose mass measurement in short time using QCM. Quartz Crystal Microbalance

B. Sputtering Estimate

The sputtering phenomenon is complicated and a theoretical equation has not yet been established. However, semi-theoretical equations based on experimental results exist. General models often target energy of keV or more. So, we will refer to Wilhelm model in this theory[5]. This theory models sputtering by ions of small energy whose ion energy is up to 100eV. Since the contact voltage of the neutralizer is about 30 to 40eV, the energy of the internal ion is considered to be on the order of tens of eV. From the Wilhelm model, the sputtering rate due to ion is expressed by Eq. (1).

$$\mathrm{S(E)} = \frac{1}{24}\alpha \mathrm{N}^{\frac{2}{3}}\left(\frac{\left(\frac{M}{m}\right)^2}{1+\frac{2M}{m}}\right)^{\frac{3}{2}}\frac{(E_1-E_0)^2}{\mathrm{E_1}^2} \qquad (E_1 \geq E_0) \tag{1}$$

Since sputtering cannot occur under the threshold, the Eq. (1) considers only energy above it. In order to use this model, α dependent on the target atom must be experimentally known. Wilhelm estimated α for several atoms, for example, Au.

C. Quartz Crystal Microbalance

In order to finish the measurement in a short time, it is necessary to measure the mass with the "ng" order. So we measured the mass decrease by sputtering by the feature of crystal, which was called Quartz Crystal Microbalance, QCM. QCM is a mass sensor that measures an extremely small amount of mass change by utilizing the property that the resonance frequency changes according to its mass when a substance adheres to the electrode surface of the crystal unit. The quartz crystal sandwiched between the electrodes to which the voltage is applied periodically performs a sliding motion in a direction horizontal to the electrode surface. When a substance adheres to this electrode surface, the vibration energy weakens and the frequency decreases. Conversely, when the substance on the electrode surface is detached, the frequency increases. The relationship between frequency and mass change on QCM is expressed by Sauerbrey's equation (Eq.1) [6].

$$\mathrm{df} = \frac{-2\mathrm{f}_0^2}{\mathrm{A}\sqrt{\rho\mu}}dm \tag{2}$$

QCM can measure so small mass and it is sometimes used to investigate air pollution or sputtering phenomena[7]. QCM has temperature characteristics[8], it is necessary to calibrate when using in an environment with temperature change. By fear of temperature change, it is hard to use in environments where a lot of ions are hit.

D. Experimental Method

Since the microwave discharge neutralizer releases electrons to the outside, ions collide with the inner wall to generate a current loop. This collided ion causes sputtering of the inner wall. Therefore, we can consider that the larger the ion current density on the neutralizer wall surface is, the larger the mass loss amount per unit area is. In the microwave discharge neutralizer of the shape used in this study, the current density on the sidewall is the largest. The current density on the sidewall is 0.1 to 0.2mA/mm^2 when the extraction current value of 80 to 170mA. For example, it is 0.15mA/mm^2 during 160mA extraction current[3]. In this study, the sputtering occurring at the sidewall was investigated.

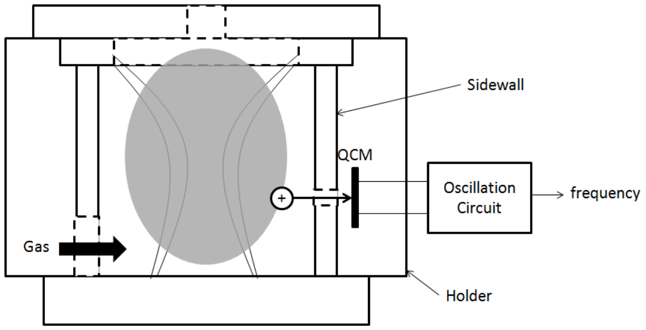

Figure 2QCM arrangements for measurement sputtering.

It is not a good idea to place the QCM in front of the sidewall to measure the sputtering. The neutralizer performance becomes worse if metal pieces enter[3]. It seems that something happens when the performance gets worse. Putting the QCM inside the neutralizer may not be able to reproduce the nominal phenomena inside it. So, we propose to measure the sputtering by the plasma which leaks from the sidewall.

The ions inside the neutralizer reach the QCM outside of the sidewall through holes on the wall. The electric signal from the QCM is sent to the outside through a holder for hermetically closing the neutralizer and the frequency is measured using an oscillation circuit [Fig.2]. The problem is that as the neutralizer is heated by microwaves the temperature inside the holder rises. QCM frequency depends on temperature. Therefore, the QCM frequency when the side hole is covered was measured to calibrate measurement only to measure the sputtering effect.

E. Experimental setup

The neutralizer is basically operated with the ion engine. The ion engine released ions and the electrons from the neutralizer are attracted to the ion potential. In this study, an anode plate is placed instead of ion potential. By applying a voltage between the anode plate and the neutralizer, electrons are extracted. The contact voltage corresponds to the neutralizer voltage. The gas uses Xe. Space is made in a part of the sidewall. The gas was supplied from the outside to the inside of the neutralizer through the holder and space. The gas was supplied at 0.7sccm. The microwave is supplied from the antenna extending from the center of the upstream yoke to the inside of the neutralizer and contributes to ECR heating. The microwave was supplied 8W. On the sidewall opposite to the gas supply space, a hole of 0.3mm in diameter is drilled and QCM is placed in front of the hole. In the study, QCM frequency is 9MHz. According to Eq. (2), the mass change per 1Hz corresponds to 1.07ng. QCM was quartz whose diameter is 8.7mm and two metal electrodes with a diameter of 5mm were stuck on it. QCM vibrates by applying a potential difference to the two electrodes sandwiching the crystal. The electrode was made of Au. The wall of the neutralizer was grounded. In order to simulate the neutralizer sidewall surface, the electrode of the QCM facing the sidewall hole was always grounded. The anode was placed 11mm from the orifice surface of the neutralizer. The anode voltage fluctuated and the neutralizer current was always 180mA [Fig.3].

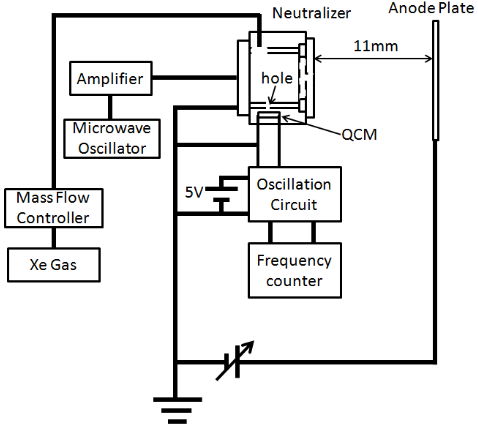

Figure 3 **Experimental setup of neutralizer for sputtering measurement.**

III. Experimental Results and Discussion

The frequency history and the frequency deviation when the hole less neutralizer is operated are shown in Fig.4. The Anode voltage was 44 to 48v.The frequency starts decreasing at the beginning and it starts to increase at around 800s from the operation of the neutralizer. After that, the gradient of frequency change becomes steep with time. This is because the temperature of the QCM continues to increase. There is no actual mass change of the QCM because there is no hole in the neutralizer. Therefore, this frequency change is considered to be due to temperature change. The difference from the frequency with the lapse of time with reference to the frequency before the neutralizer operation Δf_T

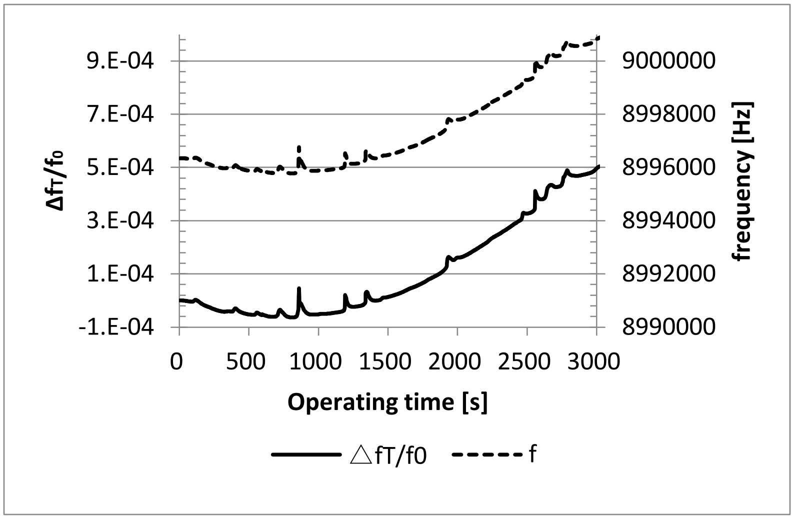

Figure 4 **Frequency change of not sputtered QCM and temperature calibration curve**

is divided by the reference frequency f_0 is shown in Fig.4. This curve is the temperature calibration curve for QCM.

The frequency of QCM at t[s] can be expressed by the temperature calibration variable and the actual frequency as follows.

$$f = f_{real} + \Delta f_T = \left(1 + \frac{\Delta f_T}{f_{real}}\right) f_{real} \qquad (3)$$

From Eq. (3), it is possible to estimate the frequency of the QCM with the influence of the temperature removed, f_{real}. The df is the difference between the frequency of QCM before the neutralizer operation and the current frequency. From the df and Eq. (2), it is possible to calculate the mass change dm amount by the neutralizer operating time. The result of calculating the mass change from the frequency history of QCM when the sidewall perforated neutralizer operates is shown in Fig.5. The anode voltage was 43 to 47V. The results show averages of mass changes in the 50s every 100s. It can be seen that the mass change decreases roughly linearly with respect to the neutralizer operating time. It can be considered that this mass change is due to sputtering caused on the QCM. The error from the primary straight line seems to be derived from the error in temperature calibration. The slope when this mass change history is linearly approximated by the least squares method is the mass change amount per unit time. The mass loss rate in this experiment was 0.64ng/s.

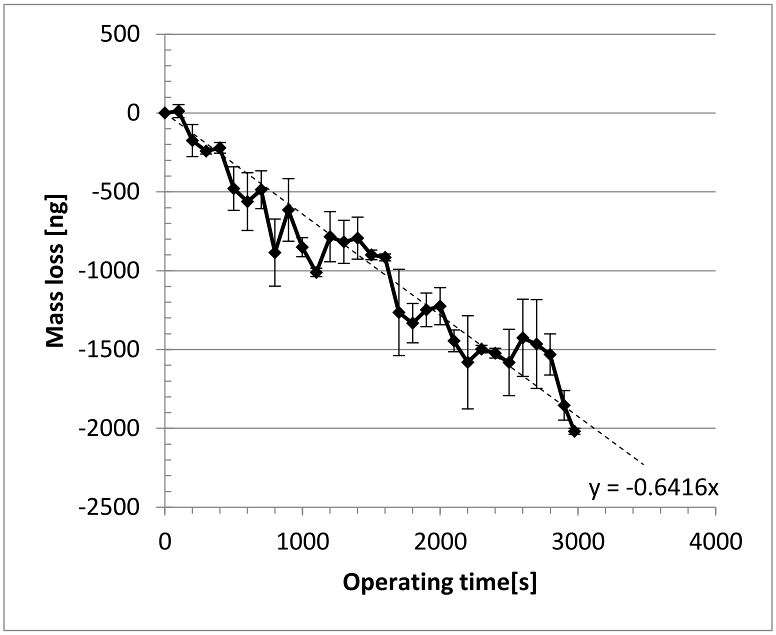

Figure 5. QCM mass reduction by sputtering.

Let us compare with conventional theory. The total amount of ions flowing out of the sidewall holes is estimated from the sidewall current density and the sidewall hole area. The weight loss rate by sputtering of QCM is multiplied as follows.

$$\dot{M} = \frac{JA_{hole}}{C} MS(E) \qquad (4)$$

The weight loss rate due to sputtering Xe ions entering Au from Eq. (1) is shown in the Fig.6. When the mass loss amount reaches 0.64ng/s, the ion energy corresponds to 40eV. This is a value very close to the extraction voltage. On the other hand, the voltage inside the neutralizer is considered to be on the order of 20eV at the anode voltage of 40 to 50 V[4].

Table 1. Specification values of sputtering of Xe ion to Au

α (Au)	4.23×10^{-16}	cm^2
Density(Au)	5.90×10^{22}	cm^{-3}
mass of Xe	2.18×10^{-22}	g
mass of Au	1.79×10^{-22}	g
Threshold energy(Au)	12	eV
Current density	0.2	mA/mm^2
Hole area	7.07×10^{-2}	mm^2

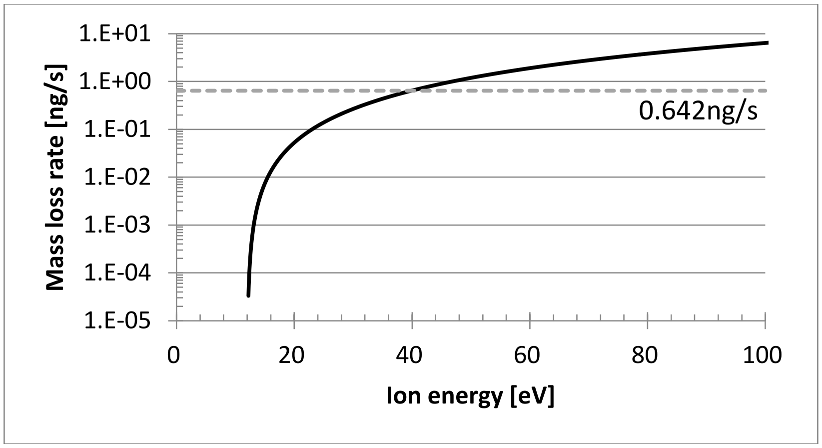

Figure 6. Mass loss rate of Wilhelm model based sputtering of Xe ion to Au

One of the factors of this difference is considered to be the presence of divalent ions. In the case of the same potential difference, the energy of the divalent ion is twice that of the mono ion. As the ratio of divalent ions increases, massive wear increases by that amount. It also depends greatly on the ion energy distribution. The plasma potential basically shows the average value. Sputtering increases exponentially with increasing energy. Even though the average value of the energy is low, mass loss rate may be large if high-energy ions are present. Furthermore, the current density and the total amount of ions don’t exactly match, because current density is only the difference between ion current and electron current. If there are more electron currents flowing to the neutralizer wall, the ion current also larger than measured. Therefore, the total amount of actual ions should be larger than the estimated value and the mass loss rate will be several times larger. These were seen by focusing on the ions rather than the entire plasma. So, it may be possible to observe more detailed phenomena inside the neutralizer using QCM.

IV. Conclusion

We can observe the mass loss rate at an approximately linear rate by measuring the sputtering caused by ions leaking from the side wall to the outside by QCM. The time required for the mass measurement is sufficient for about one hour, which is a considerable time reduction compared to the conventional material test of the microwave discharge neutralizer. Based on the result, it seems possible to compare various materials in a short time by changing the material of the QCM surface. By this method, selection of the optimum material will be prepared. Furthermore, since QCM receives only the ion plasma, it may become a clue to know more detailed information inside the neutralizer.

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