Plasma diagnostic by optical emission spectroscopy on reactive magnetron sputtering plasma – A Brief Introduction

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Abstract. Optical emission spectroscopy has been employed as non-invasive plasma diagnostics method and it is to detect the light emission during titanium nitride deposition using magnetron sputtering plasma discharge. The optical emission from argon and titanium were used to observe the ionization ratio of each element during the discharge. Then, the optical emission of nitrogen spectrum was used for the calculation of gas rotational temperature by using a simple formula and it was evaluated at various discharge conditions. A comparative of two different spectrometers resolution have been used for the determination of gas temperature. The results showed that the evaluated temperatures were almost the same as expected in high-pressure magnetron sputtering plasmas even when using low-resolution of spectrometer.

1. Introduction
Reactive magnetron sputtering is a common technique in microelectronic industries to fabricate oxide and nitride thin films. This technique produced thin films with good quality of adhesion and surface morphology [1-3]. The other advantage of reactive magnetron sputtering technique is that one could control the composition of deposited thin film by controlling the composition of sputter target and reactive gases introduce into the discharge chamber. During the fabrication of integrated circuit, titanium nitride thin film is used as barrier layer to prevent leakage current of the interconnects.

In order to optimize the plasma processing, it is essential to diagnose the plasma behaviour and understands the fundamental process during the thin film deposition. There are several of plasma diagnostics techniques have been discovered for many years. Optical emission spectroscopy is one of the plasma diagnostic tools that has been used for industrial applications [4-8]. In addition, optical emission spectroscopy (OES) measurement has been recognized as one of the common plasma diagnostics techniques due to its simplicity and low cost. Subsequently, another benefit of using OES
diagnostics is that one could able to acquire the data without perturbing the plasma during the diagnostic process.

2. Experimental Setup
Reactive magnetron sputtering plasmas were produced using water-cooled conventional balance sputter source. The sputter target was titanium and the discharge gas was argon. Nitrogen was introduced into the chamber as reactive gas to produce titanium nitride thin film. The size of solid titanium sputter target is 75 mm in diameter and 15 mm in thickness. The size of the chamber is 1 m in diameter and 1 m in height. Sputtering chamber consist of six view ports using quartz glass window for plasma monitoring. The vacuum was created using turbo molecular pump backed by rotary pump. Maximum vacuum condition was achieved at 8 x 10⁻⁶ Torr.

Figure 1 shows the experimental setup to capture the optical emission from sputtering plasmas during the deposition of titanium nitride thin film. An optical lens was used and placed in front of the quartz window chamber as shown in Fig. 1. The purpose of this configuration is to ensure that the emission from specific position in plasma discharge is focused onto the fiber entrance. The ultraviolet and visible emission from the plasma discharge passed through a quartz window chamber. The spectrometer from Ocean Optics HR4000 was used to capture the emission from the plasma discharge. The system is microcontroller-controlled spectrometer and all the operating parameters are implemented through Spectra Suite software interfacing to the unit. The spectrometer able to responsive to the wavelength in the range of 200 nm to 1100 nm. The optical emission of titanium, argon and nitrogen were studied at various of discharge conditions.

![Figure 1. Experimental setup for optical emission spectroscopy measurement at specific position in magnetron sputtering plasma system.](image)

3. Results and Discussion
The discharge power and argon flow rate was kept at 200 W and 36 sccm, respectively. The total gas pressure was kept high at 100 mTorr. The major reason of this high-pressure condition is to enhance the ionization ratio of titanium atom in the sputtering plasma as we have presented it elsewhere [9].

Figure 2 shows the typical emission for magnetron sputtering plasma employing titanium target [10]. The nitrogen flow was varied between 0 and 40 sccm. The assigning of the elemental emission spectrum was confirmed from NIST database [11]. It is clearly shown that the emission from titanium atom and ions between 300-500 nm are significant when no the nitrogen flow into the chamber (nitrogen flow rate = 0 sccm). The emission from argon metastables and ions between 700-850 nm are also clearly shown in Figure 2.
Figure 2. Widerange emission of magnetron sputtering plasma using titanium target and various nitrogen flow rates[9].

Figure 3 shows the emission of argon atoms and ions that has been assigned as Ar I and Ar II, respectively. As shown in Figure 3, the ratio of Ar I / Ar II decreased with the nitrogen gas flow rate. This result indicates two situations: (1) the relative intensity of Ar II increased with the nitrogen gas flow rate and (2) the relative intensity of Ar I decreased with the nitrogen gas flow. The experimental result was not consistent with the global model simulation on titanium nitride deposition using reactive magnetron sputtering plasmas [12-14]. The main reason of this inconsistency is may be due to the high-pressure condition in the present experiment. To date, there is no simulation report on the high-pressure magnetron sputtering plasmas.

Figure 4 show the relative intensities of titanium atom (Ti I) and titanium ion (Ti II) at various nitrogen flow rates. As shown on Figure 4, the characteristics of titanium emission was almost the same with argon emission. The Ti II increased with the nitrogen flow rate. This understanding is very important for titanium nitride thin film deposition using ionized physical vapor deposition technique. In general cases, the electron-impact excitation from titanium ground states of species is considered to be dominant excitation mechanism to increase the density of titanium ions in the plasmas. The intensity of Ti I is directly proportional to the concentration of titanium atoms sputtered from the cathode.
Another advantage of optical emission spectroscopy is to evaluate plasma gas temperature from the rotational temperature of nitrogen gas. The determination of rotational temperature of nitrogen gas was done by using any rotational band of the emission spectrum of nitrogen and it is applicable for any other molecular gas such as oxygen and hydrogen. In the case of nitrogen, one could use either the band from the first negative system at 391.4 nm or second positive system bands at 337.4 or 380.4 nm. The spectrum calculation procedure for molecular gas temperature is based on the basic principles of optical emission and it requires knowledge of the electronic, vibrational and rotational constants of particular molecular spectrum. In our previous work, data of the nitrogen spectrum was captured at 775 nm as shown in Fig.5 [15]. For simplicity, N.Britun et al in his previous works had calculated the rotational temperature based on the theoretical formula after some correlation of experimental data [16]. The formula is given by:

\[ T_{rot}[K] = \frac{195}{(P_1/P_2)^{-0.52}} \]  

(1)
Figure 5 show emission of nitrogen spectrum measured using two different type of spectrophotometers. The objective was to compare the evaluation of gas temperature when measured using low- and high-resolution spectrophotometers. If the results are comparable, one should able to use simple and low cost spectrophotometer for gas temperature evaluation. As shown in Figure 5, the peak at two positions that required for equation 1 was clearly detected for both low- and high-resolution spectrophotometers.

Figure 6 shows the nitrogen gas rotational temperature evaluated from Figure 5 and equation 1. The rotational temperature was evaluated at plasma discharge power of 100W, 150W and 200W. As shown in Fig. 6, the temperature increased with the discharge power. The fluctuation of gas temperature evaluated from low- and high-resolution spectrophotometers were small, indicating that the gas temperature at the range of 410-460 K. In other words, by using low resolution spectrometer is sufficient to detect the nitrogen emission spectrum and peak values that are required to calculate the nitrogen gas rotational temperature. In addition, Figure 6 indicates that the deposition enviroment of titanium nitride thin film was at low temperature. Therefore, the process parameters of the present experiment are suitable for coating of heat sensitive materials such as polymers.

![Figure 5](image1.jpg)

**Figure 5.** Nitrogen emission captures using low- and high-resolution spectrophotometer[15].

![Figure 6](image2.jpg)

**Figure 6.** Rotational temperature of nitrogen gas evaluated from the emission spectrum of nitrogen[15].
4. Conclusion
In conclusion, plasma diagnostic using optical emission spectroscopy technique is a simple method to understand the fundamental of plasma processing applications. The ratio of ions and atoms emission peaks were used to evaluate the ionization process at various discharge conditions. The changes were significantly observed. Then the optical emission have been detected and performed by using different resolution of spectrometer. Variation of plasma discharge powers at 100 W, 150 W and 200 W using low-resolution and high-resolution spectrometers were reported. The nitrogen gas temperature evaluated using both spectrometers were almost the same at the range of 410-460 K. The influence of discharge power is almost negligible. Therefore, we concluded that low-resolution spectrometer is adequate when measuring gas rotational temperature in certain region.

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