Effect of working power and pressure on plasma properties during the deposition of TiN films in reactive magnetron sputtering plasma measured using Langmuir probe measurement

Soo Ren How¹, Nafarizal Nayan², Mohd Khairul Ahmad³, Chin Fhong Soon⁴, Mohd Zainizan Sahdan⁵, Jais Lias⁶, Ahmad Shuhaimi Abu Bakar⁷, Mohd Khairuddin Md Arshad⁸, Uda Hashim⁹ and Mohd Yazid Ahmad¹⁰

¹,²,³,⁴,⁵,⁶ Microelectronic and Nanotechnology- Shamsuddin Research Centre (MiNT-SRC) & Faculty of Electrical and Electronic Engineering, Universiti Tun Hussein Onn Malaysia, Batu Pahat, 86400 Johor, Malaysia
⁷ Low Dimensional Materials Research Centre, Department of Physics, Faculty of Science, University of Malaya, 50603 Kuala Lumpur, Malaysia
⁸,⁹ Institute of Nano Electronic Engineering (INEE), Universiti Malaysia Perlis, 01000 Kangar, Perlis, Malaysia
¹⁰ Nanorian Technologies Sdn Bhd, No. 40 & 40-1, Jalan Kajang Perdana 3/2, Taman Kajang Perdana, 43000 Kajang. Selangor, Malaysia

E-mail: nafa@uthm.edu.my

Abstract. The ion, electron density and electron temperature during formation of TiN films in reactive magnetron sputtering system have been investigated for various settings of radio frequency (RF) power and working pressure by using Langmuir probe measurements. The RF power and working pressure able to affect the densities and plasma properties during the deposition process. In this work, a working pressure (100 and 20 mTorr) and RF power (100, 150 and 200 W) have been used for data acquisition of probe measurement. Fundamental of studied on sputter deposition is very important for improvement of film quality and deposition rate. Higher working pressure and RF power able to produce a higher ion density and reduction of electron temperature.

1. Introduction

Attention for the development of metal-nitride applications in various fields has been attracted by the community. One of the examples of metal nitrides is titanium nitride, TiN films. TiN coatings commonly used as wear resistant for cutting tools due to its excellent physical, chemical, electrical and mechanical properties[1-2]. Moreover, TiN films also applied in the microelectronics industry as a diffusion barrier in integrated circuit [3-4]. Deposition of TiN films could be prepared by physical vapor deposition (PVD) or chemical vapor deposition (CVD) techniques which depends on the properties of the application. In fact, PVD method is one of the most extensively used for formation of
TiN such as reactively magnetron sputtering [5-6]. By using this method, it is possible to deposit compound films with controlled microstructure and composition. However, one of the disadvantages is the presence of reactive gas during the sputtering of the metallic target interacts with the target and fall down at the surface of the target.

Up until now, the deposition of TiN films still kept improving in term of its properties especially in integrated circuit (IC). As we can know that IC chips had evolved from a micro to a few nano size. Thus, TiN as a barrier diffusion in IC quite hard to deposited on trench with a few nano sizes of chips[5]. Therefore, there is a new challenge arise in microelectronics industry [7-8]. In order to overcome this problem, we have well understand about the sputtered deposition mechanism of TiN films in reactive sputtering plasma. Therefore, plasma diagnostics technique is necessary needed to know the basic of the sputtered deposition mechanism of films.

Previously, we had conducted the plasma diagnostics using optical emission spectroscopy (OES) method to characterize the plasma during sputtering. We use this OES method to study about the effect of RF power and N2 flow rate on the deposition of TiN films. For this experiment, we will use Langmuir probe as the plasma diagnostics technique that used to investigate and studied the behavior of mechanism sputter deposition as well as characterize the plasma in the chamber at different working pressure with various of RF power.

2. Experimental details

Figure 1 demonstrated the configuration of Langmuir probe (Espion, Hiden Analytical, UK) with tungsten tip was used as plasma diagnostics technique in the sputtering chamber. The probe was inserted inside the chamber through the viewport of the chamber. The sputtering chamber was evacuated by high vacuum pumping system with a turbo-molecular pump and able to create a base pressure of less than 10⁻⁶ mTorr. A titanium target (3” diameter, 0.125”W thick, 99.995% pure) was used as the target of sputtering. The chamber was pre-sputtered first before running the experiments. A tungsten tip with diameter of 0.15 mm and length of 10 mm and encapsulated with a ceramic insulator as protection from energetic plasma.

ESPion software used to interfaced with the Langmuir probe system for collecting the results and analyze under different plasma parameters. The probe bias voltage was swept from -100 V to 50 V. The probe that used to measure the plasma parameters was placed at center of the substrate location approximately 10.5 cm above of the target. The probe tip was cleaned with the suitable cleaning solution before start data acquisition process. For this experiment, the working pressure was varied to the range of 100 mTorr and 20 mTorr in the chamber during the data acquisition. Meanwhile, working power of 100, 150 and 200 W were used for each set of working pressure. Flow rate of argon and nitrogen gas were set and varied according to working pressure that using for this experiment. Five scans of I-V characteristics curve were acquired and averaged for each parameter. For analyze the result part, semi-automatics data analysis mode was prefered to determine and calculate the plasma properties such as ion, electron densities and electron temperature.
3. Result & Discussion

Figure 2 shows the typical representative of I-V characteristic curve that collected using Langmuir probe device for 100 mTorr of pressure, 200 W RF power, 36 sccm of Ar gas and 18 sccm of N₂ gas flow rate. This I-V curve representative has divided into three main parts to determine the plasma parameters. The first one is ion saturation region where it only covers the positive ions. Another part of an electron saturation region where it collects electrons only. Then, the last part of transition region is used to calculate the electron energy density and plasma potential[9-10]. From this curve, we will focus on certain plasma parameter including electron temperature, ion and electron density at different pressure and power.
Figure 3 shows the measurement of ion density obtained in Langmuir probe at different working pressure in Ar and N\textsubscript{2} gas mixture. It could be observed that the ion density increasing with higher RF power for both 100 and 20 mTorr. However, the trend of the ion density at 100 mTorr was higher than 20 mTorr regardless of RF power. As RF power increase, the ion density at 100 mTorr rapidly increase while the ion density at 20 mTorr nearly remain constant. By using the higher RF power, it is able to providing more energy to atoms and ions to increase the ease of breakdown of chemical bonding of the compound.

At higher pressure (100mTorr), all the reaction happening at the very confined space, causing the rate of collision between the atoms and electrons become higher. Thus, almost all of the electrons would always condense and circulating at a region of target and these electron not easily been escaped. As a result, the rate of ionization and dissociation of atoms increases compared at lower pressure. Therefore, the electron density decrease with increasing of working pressure.

![Figure 3](image.png)

**Figure 3.** Measurement of ion density as function of RF power at different working pressure in (Ar + N\textsubscript{2}) gas mixture.

Figure 4 shows the measurement of electron density as a function of RF power obtained at pressure 100 mTorr and 20 mTorr respectively. The fluctuation of the trend electron density at high pressure significantly changes whereas there was nearly constant for 20 mTorr. However, it still has a slight change even the trend seem not to fluctuate in Figure 4. At 100 mTorr, the electron density decreases as the RF power increases. Significant change could be found from power of 150 to 200 W where the electron density decreases steeply. It was indicated that the rate of collision is higher, causing the reduction of concentration of electron and thus the plasma could be sustained. Moreover, the deposition rate also increases in higher working pressure (100 mTorr) and RF power (200 W).
In contrast, the electron density at 20 mTorr did not exhibit the significance changes even there was a slight increase as increasing of RF power and shown in Figure 4. It is expected that electron still able to escape from the plasma region, which these electrons supposedly used for reaction purpose. Thus, the increment of electron density at lower pressure would be detected by probe although using higher RF power. Applying higher working power in the deposition still could not give much effect if using lower pressure.

Figure 5 illustrated the measurement of electron temperature obtained at a variety of RF power and working pressure. The electron temperature at 100 mTorr decrease with increasing of RF power. One of the possible explanation was that rate of collision between electrons decrease due to lower electron density that discussed above and resulting the electron temperature become lower. On the other hand, the electron temperature seem increases at 20 mTorr. Initially, there is not much difference at RF power of 100 and 150 W. It then started to increase when using 200 W. High amount electrons could be collected at working power of 200 W resulting the more collision between electron in the plasma occur in this region. Consequence, the electron temperature become higher.

![Figure 4](image_url)  
**Figure 4.** Measurement of electron density as function of RF power at different working pressure in (Ar + N\textsubscript{2}) gas mixture.
Figure 5. The electron temperature as function of RF power at different working pressure in (Ar + N₂) gas mixture.

4. Conclusion

Effect of working pressure and power on the formation of TiN films in Ar + N₂ gas mixture could be measured by using the Langmuir probe. It is possible to determine the ion, electron density and electron temperature at different working pressure (100 mTorr and 20 mTorr) and power (100, 150 and 200 W) present in the reactive magnetron sputtering system. It is found out that different plasma parameter able to obtain different results during the data acquisition from the Langmuir probe. These findings of this works could be used for better understanding on fundamental of sputtered deposition mechanism during the formation TiN films. It could be conclude that using higher working pressure (100 mTorr) and higher power (200W) during the sputtered deposition able to produce greater of ion density in the chamber that able to improve the rate of deposition.

Acknowledgments

The present work was partially supported by Fundamental Research Grant Scheme FRGS/UTHM/1207, Exploratory Research Grant Scheme ERGS E025 of Ministry of Higher Education Malaysia and Contract Grant UTHM U566 and U675.
References

[1] S. Carpenter and P. J. Kelly, 2009 Surf. Coatings Technol., 204, pp. 923–926.
[2] P. W. Shum, W. C. Tam, K. Y. Li, Z. F. Zhou, and Y. G. Shen, 2004 Wear, 257, pp. 1030–1040.
[3] N. Jiang, H. J. Zhang, S. N. Bao, Y. G. Shen, and Z. F. Zhou, 2004 Phys. B Condens. Matter, 352, pp. 118–126.
[4] H. Liang, J. Xu, D. Zhou, X. Sun, S. Chu, and Y. Bai, 2016 Ceram. Int., 42, 2, pp. 2642–2647.
[5] Y. L. Jeyachandran, S. K. Narayandass, D. Mangalaraj, S. Areva, and J. A. Mielczarski, 2007 Mater. Sci. Eng. A, 445–446, pp. 223–236.
[6] P. Roquiny, F. Bodart, and G. Terwagne, 1999 Surf. Coatings Technol., 116–119, pp. 278–283.
[7] U. Helmersson, M. Lattemann, J. Bohlmark, A. P. Ehiasarian, and J. Tomas, 2006 Thin Solid Films 513, pp. 1–24.
[8] E. Kusano, N. Kashiwagi, T. Kobayashi, H. Nanto, and a. Kinbara, 1998 Surf. Coatings Technol., 108–109, pp. 177–181.
[9] B. B. Sahu, J. G. Han, M. Hori, and K. Takeda, 2015 J. Appl. Phys., 117, 2, pp. 1–14.
[10] S. P. Koirala, H. H. Abu-safe, S. L. Mensah, H. A. Naseem, and M. H. Gordon, 2008 Surf. Coatings Technol., 203, 5–7, pp. 602–605.