CLOSED LOOP CONTROLLED REACTIVE DUAL MAGNETRON SPUTTERING

By L. Lou, G. MacDonough, H. Walde, D. Carter, G. Roche, and R. Scholl of Advanced Energy Industries, Inc.

Rapid closed loop control of oxygen flow was used to prevent target poisoning and facilitate high growth rate during reactive sputtering. Low frequency ac reactive dual magnetron sputtering of Al_2O_3 was performed, achieving stable film formation with high deposition rates. Thin film properties of deposition rate, refractive index, and film transmission are reported as a function of reactive gas flow rate and target voltage. Results are compared to reactive sputtering of these films by other techniques.

INTRODUCTION

There are numerous approaches to deposit compound thin films, especially dielectric thin films. One classical technique for depositing insulating films is direct sputtering of dielectric targets by the use of radio-frequency (RF) power. However, it is well known that the RF sputtering technique has a relatively low deposition rate, a high cost of power supply equipment, and a strict RF shielding requirement on the chamber system. In order to achieve higher deposition rates, the technique of reactive sputtering has been developed. In this technique, a metallic target or a semiconductor target is sputtered at dc or low frequency ac in the presence of a reactive gas. In addition to higher deposition rates, reactive sputtering has the added benefits of lower cost of power supply equipment and less stringent shielding requirements. Multiple reactive sputtering configurations have been investigated, ranging from simple dc sputtering of a single target, to pulsed dc sputtering on a single target, to dual cathode sputtering using sinusoidal power, to a new technique using both pulsed dc power and dual cathodes[1]. In this paper, low frequency ac dual magnetron reactive sputtering of dielectric thin film will be discussed[2, 3].

Aluminum oxide thin films are widely used throughout the electronics and industrial coatings industries because of their excellent mechanical, optical and dielectric properties[4]. Much work has been done on the dc reactive magnetron sputtering of aluminum oxide[5, 6, 7]. Compared with RF sputtering, a much higher deposition rate can be achieved using dc sputtering. However, there are unresolved problems in dc or pulsed dc reactive sputtering processes such as arcing and the "disappearing anode" problem[8]. During dc reactive sputtering of dielectric thin films, if a single magnetron target is used, the anode will be covered with a layer of sputtered dielectric thin film over time. This has been called the "disappearing anode" problem. In order to solve this problem, the technique of low frequency ac dual magnetron sputtering has been used, which can both eliminate the disappearing anode problem and achieve high deposition rates[9].

Although high deposition rates are possible using dc reactive sputtering, it is only achieved under the condition that the target surface is not severely "poisoned." To this end, closed loop control of the reactive gas flow is described in this paper. The objective of the flow control is to regulate the reactive gas flow rate based on changes in target condition and requirement for film stoichiometry.

EXPERIMENTAL DETAILS

The concept of closed loop controlled low frequency ac dual magnetron reactive sputtering is shown in Figure 1. The dual cathode application requires that the plasma path between the targets be small to reduce the impedance and to avoid unnecessary substrate heating. It is useful to use one plasma shield around both targets surrounding the plasma cloud as a back plate to avoid the reactive gas from going deep in the chamber when no substrate is present. The gas inlet can be placed between both targets. The flow can be directed towards the substrate which is placed close to plasma back plate.

There are several different techniques to control the reactive gas flow. When depositing dielectric films, the target voltage changes as a function of reactive gas partial pressure at a fixed power. This is due to the difference between the secondary emission coefficient of the metal and the reaction product (the dielectric) on the target surface. Because of this, the oxygen gas flow can be regulated as a function of target voltage change. This methodology, however, does not apply to every application. In some cases the difference in the secondary emission coefficient is too small to make this method reliable. When it is not applicable to monitor target voltage change, the most commonly used technique is to regulate the reactive gas flow rate through the measurement of plasma emission spectrum. Compared to the plasma emission spectrum measurement, however, target voltage measurement is easier and cheaper to use. Since in our study there was a large reliable change in secondary emission coefficient, we used target voltage measurement as the parameter to control the oxygen gas flow.

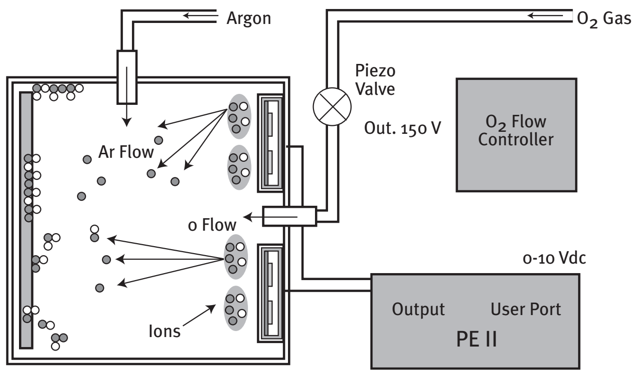

Figure 1. A typical chamber configuration of closed loop controlled dual magnetron reactive sputtering

Since target poisoning can occur very rapidly, the gas flow control valve should be a fast response valve, with speeds on the same order as the chemical reaction times (~1ms). A piezoelectric control valve is normally used on the oxygen gas inlet to regulate oxygen gas flow for these fast response times.

In the system of Figure 1, a desired target voltage setpoint is set on the reactive gas flow controller. The controller then measures the target voltage and compares the measured target voltage to the setpoint voltage. If the setpoint voltage value is higher than the target voltage value, the controller closes the piezoelectric valve to reduce the oxygen flow into the chamber. If the setpoint value is lower than the actual target voltage, the gas flow controller opens the piezoelectric valve to increase the oxygen flow into the chamber. The controller also provides phase lead and gain adjustments to permit the complex response of the controller to be matched to the system for closed loop stability.

The experiments of aluminum oxide thin film sputtering were carried out on a Balzers Pfeiffer PLS 500 chamber system. The actual chamber configuration is shown in Figure 2.

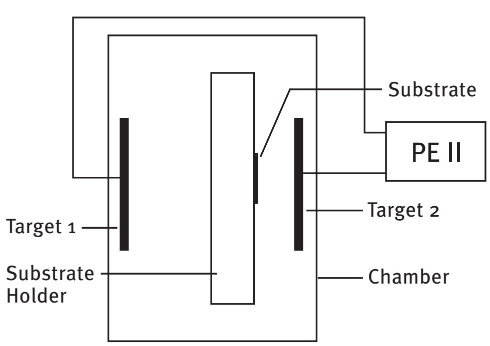

Figure 2. The chamber configuration of the experimental system

The ac power supply used was an Advanced Energy PE II. The power output of this unit is a sinusoidal ac waveform with a frequency of 40 kHz. The ac power output was connected between two targets so that the two targets acted as anode and cathode alternately during the power cycle. In other words, when one target was a cathode, the other target acted as an anode for that cathode. The PE II can be set to regulate current, voltage, or power. During these experiments, the unit was operated in power regulation mode. The two targets were Torrus 10 aluminum targets, which had circular shapes with center anodes grounded to the chamber wall. A rotary substrate fixture was placed vertically in the middle of the chamber, and the substrate could face only one of the targets. The center-to-center distance between the substrate and facing Al target was 15.9 cm. The oxygen gas inlet was on the bottom base plate of the chamber, between the substrate fixture and the target, and was about 5 inches to the substrate.

An MKS 250E reactive gas flow controller was used to control the oxygen gas flow on piezoelectric valve. A Von Ardenne PCV25 piezo valve was used on the oxygen gas inlet as a fast response gas valve.

The substrates were 2.5 by 7.5 cm glass slides. They were cleaned with 2-Propanol and were masked in their middle before being used.

The deposited film thickness was measured by a surface profilometer (a Tencor Alpha-Step 250) on the samples using the step produced by the above-mentioned mask.

Optical properties such as refractive index and transmission of the films were measured using a Film-Metrics F20 instrument.

The chamber was pumped to an approximate base pressure of $2x10^{-5}$ mbar prior to each sputter deposition. When the chamber base pressure was reached, Ar gas was backfilled into the chamber through a separate mass flow controller. The Ar gas flow rate was kept constant at 90 sccm throughout the experiments. Before each sputtering process, the target was presputtered in pure Ar for 30 minutes.

RESULTS AND DISCUSSION

Prior to the film deposition, the hysteresis curve between target voltage and oxygen gas flow rate was investigated with and without closed loop control. The curves are shown in Figure 3 and Figure 4. The sputtering conditions are listed in Table 1.

Target	Aluminum
Target area	173 cm^2
Power range	2.5 kW - 3.5 kW
Ar flow rate	90 sccm
Ar pressure range	5.2E-3 - 6.7E-3 mBar
O_2 flow rate	0 - 200 sccm
Total pressure range	6.7E-3 - 3.9E-2 mBar

Table 1. Sputtering condition for hysteresis curves

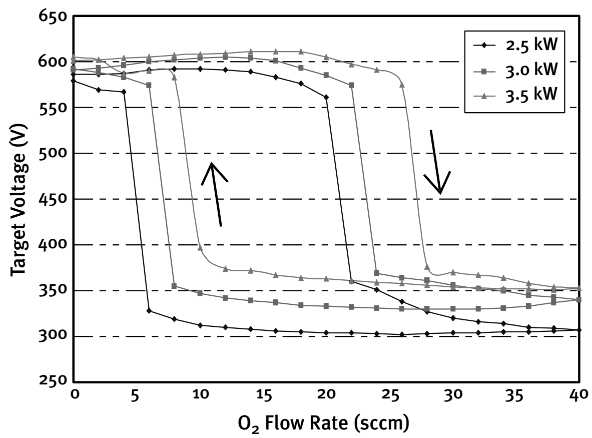

Figure 3. Hysteresis curve with open loop

With the loop open, the change in target voltage was taken as a function of the gas flow rate. With the loop closed, a desired target voltage was set on flow controller, causing the controller to maintain that voltage and automatically adjusting the flow rate. In comparing Figures 3 and 4, it is evident that the closed loop control is much tighter, with only 4 sccm O_2 flow hysteresis, versus 20 sccm O_2 hysteresis in the open loop case. The two turns after the knee in Figure 4 may be caused by unbalanced chamber geometry.

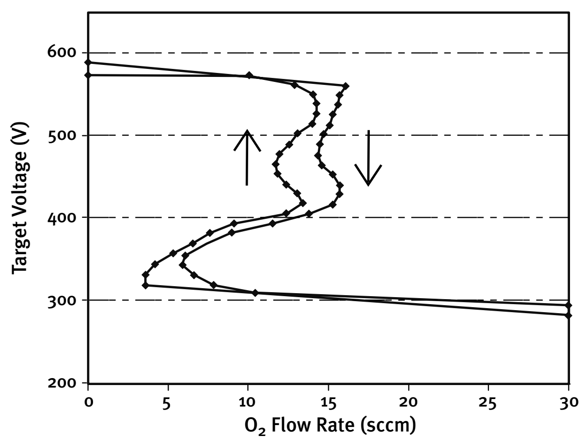

Figure 4. Hysteresis curve with closed loop control at 2.5 kW output

It was observed in both curves that there were two different operating modes of reactive magnetron sputtering: the metallic mode and the oxide, or "poisoned" mode. In the metallic mode the sputtering rate and the reactive gas consumption was high. In the oxide mode the sputtering rate and the reactive gas consumption is low.

As shown in Figure 3, when the loop was open, the transition from metallic mode to oxide mode was fast. In fact, it took only a few seconds for this transition, and it was difficult to maintain any voltage level in the transition region.

Figure 5 shows the forward trace (increasing O_2 flow) for both closed and open loop control configurations. Under closed loop control, the transition region was very controllable.

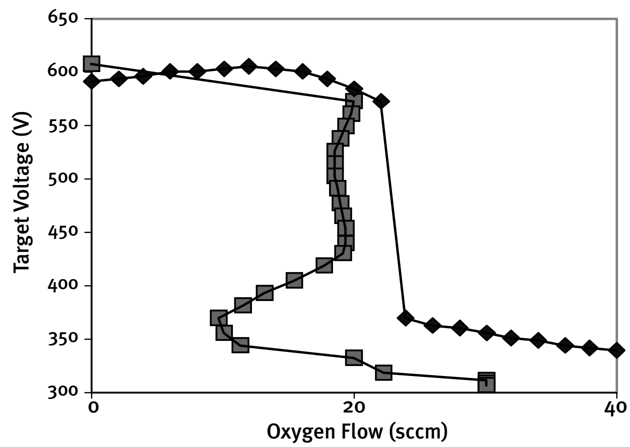

Figure 5. Al_2O_3 target voltage hysteresis curves of open loop versus closed loop

After running the hysteresis curves, three power levels and different voltage levels were chosen to deposit aluminum oxide samples with different composition under closed loop control. Figure 6 shows the trend of deposition rate and transmission as a function of target voltage at different power levels with closed loop control.

At higher voltage, around 570 V, the transmission of samples was very low and the color of the film was dark brown. From a critical voltage below about 550 V, the samples became transparent. This change in optical characteristics suggests that the film composition which was made under high voltage was metal rich. At lower voltages the films became transparent, indicating that the film was stoichiometric aluminum oxide or oxygen rich.

The deposition rate was calculated through the measurement of film thickness and sputtering duration. At voltages higher than 530 V, the deposition rate increased with output power as expected. However, the deposition rate was found nearly constant with power at voltages below 530 V. We speculate that this could be due to the unbalanced target

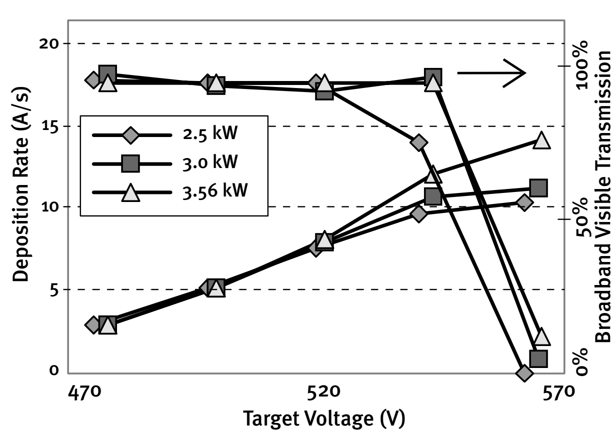

Figure 6. Reactively sputtered aluminum oxide deposition rate and film transmission

configuration, where the excess power was lost on the anode (which was the other target). That is, under low voltage conditions, both target surfaces are at least partially poisoned. The target acting as an anode will have a higher voltage (anode fall), and some of the power from the supply will be lost heating this anode by electron bombardment.

By doing target voltage control under the closed loop, good film composition control was achieved. The voltage was maintained constant while the oxygen gas controller was regulating the gas flow. With this technique, both power and voltage could be fixed parameters.

In order to more closely investigate the transition between the metal rich film and fully oxidized film, and how stable the voltage controlled closed loop works, samples were made between 570 V and 550 V under 3.0 kW output. Figure 7 shows the transmission of the films. The data indicates that the film transmission increased with the decrease of target voltage. Consistent with the transmission measurement, the film color changed from dark brown to light brown. In addition, refractive index data shown in Figure 8 is consistent with this as well.

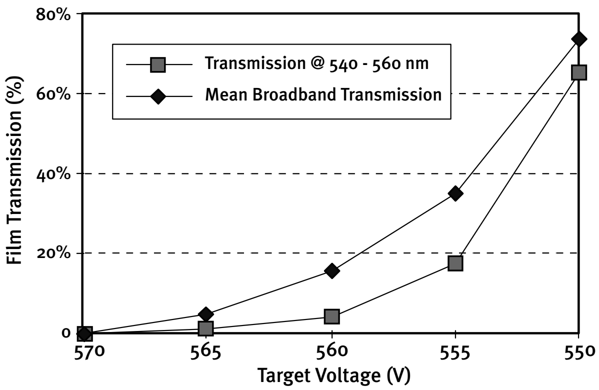

Figure 7. Film transmission at different target voltages under 3.0 kW output power

Figure 8 shows the refractive index of the films under different voltage. The refractive index tends to be 1.63 which is that of the stoichiometric bulk Al_2O_3 material at voltages below 550 V.

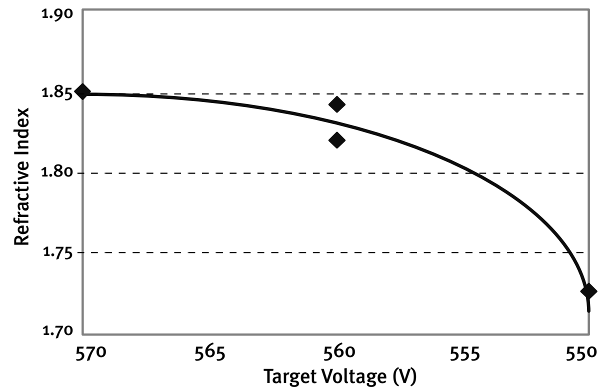

Figure 8. Refractive index of aluminum oxide thin films made under different voltages

CONCLUSIONS

Closed loop control during reactive sputtering is demonstrated in the configuration of dual magnetron ac sputtering. This was accomplished by fixing the sputtering power and regulating the oxygen gas flow so as to maintain a fixed voltage on the sputtering target. Aluminum oxide films ranging from metal rich to fully oxidized were obtained through stable closed loop control. Target surface condition is a critical parameter in the control of the film stoichiometry and deposition rate. The film stoichiometry and structure still needs to be studied, and further investigations of the film properties are also needed.

REFERENCES

1 *R.A. Scholl, Power Systems for Reactive Sputtering of Insulating Films, Advanced Energy Industries, Inc.*

2 *R. Lats, M. Schanz, M. Scherer, J. Szczyrbowski, Patent US5169509, 1991*

3 *R. Lats, Patent US5556520, 1995*

4 *E. Doerre, H. Huebner, Alumina: Processing, Properties and Applications, Springer-Verlag, Berlin, 1984, p.9.*

5 *K. Koski, J. Hoelsa, P. Juliet, Voltage Controlled Reactive Sputtering Process for Aluminum Oxide Thin Films, Thin Solid Films 326, 189-193, 1998.*

6 *M.K. Olsson, B. Hjoervarsson, High Rate Reactive DC Magnetron Sputter Deposition of* Al_2O_3 *Films, J.Vac. Sci. Technol. A 16(2), 639-643, 1998.*

7 *M. Scherer, J. Schmitt, R. Latz, M. Schanz, Reactive Alternating Current Magnetron Sputtering of Dielectric Layers, J. Vac. Sci. Technol. A 10(4), 1772-1776, 1992.*

8 *W. D. Westwood, Reactive Sputter Deposition, Handbook of Plasma Processing technology, 1990.*

9 *G. Este, W. D. Westwood, A Quasi-Direct Current Sputtering Technique for Deposition of Dielectrics at Enhanced Rates, J. Vac. Sci. & Tech. A 6, p1845, 1988.*

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Advanced Energy Industries, Inc.
1625 Sharp Point Drive
Fort Collins, Colorado 80525
800.446.9167
970.221.4670
970.221.5583 (fax)
support@aei.com
www.advanced-energy.com

California	New Jersey	United Kingdom	Germany	Korea	Japan	Taiwan	China
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