The Reactive Sputter Deposition of Aluminum Oxide Coatings Using High Power Pulsed Magnetron Sputtering (HPPMS)

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ABSTRACT

HPPMS combined with partial pressure control of the reactive gas was used for the reactive sputter deposition of aluminum oxide coatings. HPPMS can produce high ionization of the sputtered species, which can promote the formation of a reactive film. In this study, an aluminum target was sputtered in an argon/oxygen atmosphere with two different magnetron configurations. In the first configuration, a circular planar magnetron with a rotating magnet pack located behind the target was used, and the reactive gas was introduced around the substrate holder. Some arcing was observed during the reactive sputtering, but the arc handling capabilities of the experimental HPPMS power supply minimized the effects of this arcing. In the second configuration, a circular planar magnetron with a fixed magnet set was used, and the reactive gas was introduced through a simple delivery system next to the target. Again some arcing was observed, but the arc handling capabilities of the power supply mitigated the effects. Aluminum oxide films were reactively sputtered in both systems, which demonstrates that with arc handling capabilities in the power supply and reactive gas partial pressure control it is possible to reactively deposit a dielectric film with HPPMS power.

INTRODUCTION

Within the past five years, a new technique with the promise of generating highly ionized plasmas using essentially conventional magnetron sputtering equipment has been developed. Very high power pulses with high peak values two orders of magnitude greater than normally used, are applied to the magnetron cathode to achieve a high degree of ionization of the sputtered target material [1-3]. Peak power densities range from 1 to 3 kW/cm^2, at discharge voltages between 500 and 1000 Volts. This technique has been used to demonstrate the advantage of using ions to aid film deposition for semiconductor via fill [1], hard tool coatings [4], and the deposition of a very high density (2.7 g cm^3) protective carbon overcoat for a computer hard disk [5]. There are many more possible applications. The HPPMS process generates considerably fewer macroparticles than the cathodic arc process, and those few that are formed have a smaller size compared to the cathodic arc size.

The high power pulse process has been described as starting in the arc regime before transferring to an arc free region [6]. However, this transition has not been observed in work by these authors [7]. Conventional sputtering at typical power levels has significant issues with arcing, and a good deal of effort has gone into developing techniques for the prevention and handling of arcs [8,9]. Today's sputtering market demands that power supplies detect and handle arcs in a timely manner in order to prevent or lessen the damage done by them.

In order to learn more about HPPMS, an experimental power supply was designed and built [5,7]. It can produce pulses with peak powers of ≤3 MW with a pulse width of 100 - 150 μs. The repetition rate for the pulse is from a single shot to 500 Hz, and the average delivered power is ≤20 kW. A key feature of this experimental power supply is that it can detect and suppress arcs during the HPPMS process in similar manner as is done today for conventional sputtering power supplies. An arc is detected either by a significant drop in the cathode voltage or by a sudden increase in the current supplied to the target. When an arc is detected, the load is first disconnected from the power supply, and then the remainder of the energy stored in the inductor is redirected back to the storage capacitors. Once the arc is cleared, the HPPMS power supply resumes delivering the high power pulses to the sputtering target. More information about this power supply can be found elsewhere [7,10,11].

The great potential of HPPMS is that it can produce an ionized metal plasma similar to the cathodic arc source without the generation of the macroparticles. The challenge now is to learn how to use this ionized metal plasma to improve the structure and properties of sputtered films. Ehiasarian et al. [4] used HPPMS for the deposition of CrN films, and they reported several improvements over the CrN films deposited with their combined cathodic arc and unbalanced magnetron sputtering system. These improvements for the HPPMS CrN films included a reduced corrosion rate and a reduced wear rate (against Al_2O_3) compared to arc deposited CrN.

The work by Ehiasarian et al. also showed that it is possible to use HPPMS for reactive deposition of conducting films. However until now, no work has been reported on HPPMS

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reactive deposition of nonconducting dielectric films. The concern with reactive HPPMS is that the target may become covered with the dielectric film between pulses since the time between pulses is relatively long compared to the pulse time. However, with the development of the experimental HPPMS power supply with arc handling capabilities, it was felt that it should be possible to reactively deposit dielectric films with HPPMS. This paper reports on the efforts to reactively deposit aluminum oxide films using HPPMS in conjunction with partial pressure control of the reactive gas, and another companion paper at this conference reports on the reactive deposition of HPPMS titanium dioxide films [12].

EXPERIMENT CONDITIONS

The aluminum oxide HPPMS reactive deposition experiments were carried out in two different sputtering systems with two different magnetron cathode designs. The first sputtering system was an Applied Materials Centura cluster tool with their 200 mm PVD chamber mounted as one of the modules. An Applied Materials rotating planar magnetron cathode was used with an aluminum target (99.999% purity) with a diameter of 33 cm. The rotational speed of the magnets was 90 rpm. The target to substrate distance was 7.5 cm, and substrates were 200 mm silicon wafers with no oxide layer. Prior to deposition, the sputtering module was pumped down to a base pressure of 4×10^{-5} Pa (3.0×10^{-7} Torr) or better. The Ar sputtering pressure was 0.87 Pa (6.5 mTorr), and the partial pressure of the oxygen reactive gas was controlled with the Advanced Energy® IRESS reactive sputtering controller [13] using an Inficon Transpector 2 to provide the feedback signal for the reactive gas. The oxygen partial pressure is reported as a 0-10 volt signal, which represents the current collected on the Faraday cup in the mass spectrometer. The oxygen was introduced into the sputtering chamber through a circular manifold next to the target in the Centura tool. The average HPPMS power applied to the target varied between 1.0 to 2.2 kW. The average HPPMS power, P_{ave}, is calculated from the following equation:

$$P_{ave} = 1/2CV^2 f_{rep} \tag{1}$$

where C is the capacitance of the bank of capacitors in the HPPMS power supply in μf, V is the voltage to which the capacitors are charged in volts, and f_{rep} is the repetition rate for the high power pulses in Hz.

In addition to the HPPMS power supply, a 10 kW Advanced Energy Pinnacle power supply and a Sparc-LE V pulse generation unit was used to supply conventional bipolar pulsed DC power to the target for a comparison of the deposition rates for aluminum oxide films deposited by HPPMS and pulsed DC power. The bipolar pulsed DC frequency was 100 kHz, and the reverse time was 2.0 μsec. The same argon pressure and average power were used for both the HPPMS and pulsed DC depositions.

The second sputtering system used for the HPPMS experiments was a medium sized, batch, open-volume, cylindrical vacuum chamber (0.75 m diameter x 0.45 m deep) equipped with a 15 cm diameter Kurt Lesker Torus 10 balanced-field magnetron with fixed magnets in the cathode structure. Prior to test depositions, the chamber was turbo-molecular pumped to base pressures below 1.33×10^{-4} Pa (< 1.0×10^{-6} Torr). The HPPMS power was applied to an aluminum target (99.999% purity), and the average power was between 1.0 to 2.0 kW. The argon sputtering pressure was 0.44 Pa (3.3 mTorr), and the Advanced Energy® IRESS partial pressure control system was again used to control the partial pressure of the reactive gas. A simple two-gas inlet system was used to distribute the gas inside the chamber next to the target. Substrates were glass slides (2.54 x 7.62 cm) mounted in a carousel, and up to eight individual slides could be coated during a vacuum cycle. Target to substrate distance was 11.4 cm.

Hysteresis curves were automatically generated for the reactive deposition of AlO_x in both deposition chambers using the IRESS partial pressure controller, and points were chosen from these hysteresis curves for deposition of AlO_x films with different compositions. The thickness of these films was measured with an Alpha Step 250 surface profilometer, and their optical constants, n and k, were measured with a Gaertner L116B ellipsometer at a wavelength of 632.8 nm. A Philips 3100 X-ray diffraction unit using CuKα radiation (40 kV and 40 mA) was used to check the crystallinity of the aluminum oxide films.

RESULTS AND DISCUSSION

In HPPMS the condition of the target is an important factor with respect to the amount of power that can be applied to the target without incurring arcing. Any new target that is placed into the system must be conditioned by starting out at low power and then increasing the power. Due to the higher voltages and currents during the high power pulse, the plasma spreads out over a larger area of the target compared to conventional magnetron sputtering, and this larger area must be cleaned of any contaminants before the target runs smoothly with little or no arcing. The arc handling capability of the experimental HPPMS power supply facilitated this target break-in period and minimized the time needed to arrive at the point where the target is running virtually arc free.

A used target, particularly if it has been run in a reactive process with oxygen, presents a challenging target cleanup problem. The net deposition areas at the center and along the outer edge of the target, in this case, will be covered with an oxide layer that charges up in the presence of the plasma. Arcs will occur usually along the interface between these oxide layers and the metallic part of the target, and the arcing can be very severe. Without arc handling capability, it would be impossible to clean up the target prior to proceeding on with the reactive process. The target break-in and clean-up issues were the same in both deposition systems, but from the work done in this study the target magnet configuration was not a factor.

In the Centura deposition tool, aluminum films were deposited with both the conventional pulsed DC and HPPMS power supplies. Results from these deposition tests are shown in Table 1. For an equivalent average power, the HPPMS deposition rate is lower than the rate for conventional pulsed DC power. The HPPMS rate is typically 25 to 30% of the conventional pulsed DC rate. One reason for this loss of rate is the fact that a portion of the sputtered material is attracted back to the target after it becomes ionized.

Table 1: Comparison of Al HPPMS to Conventional Pulsed DC Deposition Rates.

Power, kW	Rate, nm/min		Ratio, HPPMS to DC
	HPPMS	Conventional Pulsed DC	
1.0	22	70	.31
2.0	37	149	.25

A feedback signal that is representative of the partial pressure of the reactive gas is an important part of a closed-loop control system for reactive sputtering. In the case of the Centura deposition tool with the rotating magnet pack, the motion of the magnets caused a perturbation of this feedback signal. The inlet port for the differentially pumped mass spectrometer was located close to the deposition zone, and as the magnets and plasma passed in front of the port the partial pressure signal dropped due to two factors. The first factor, which was the smaller of the two, was that the magnetic field from the rotating magnets effected the output of the mass spectrometer. This effect, observed when the cathode power was off, was relatively small, on the order of a few percent variation in the feedback signal. The second effect of the rotating magnets came when target power was on. The plasma formed beneath the rotating magnets, and the reaction between the sputtered material and the reactive gas took place on the substrates in the plasma region. As the plasma swept around the chamber, the reactive gas partial pressure was lowered in the plasma region compared to its pressure away from the plasma. Thus, as the plasma passed in front of the mass spectrometer port, the reactive gas partial pressure was momentarily reduced resulting in a fluctuation of the partial pressure feedback signal. The variation was about 10% of the feedback signal, and it resulted in a noisy feedback signal. The control of the partial pressure of the reactive gas was more difficult compared to systems where the magnets are stationary, but control was possible.

The combination of partial pressure control of the reactive gas combined with the arc handling capabilities of the experimental HPPMS power supply made it possible to reactively deposit AlO_x films in both deposition systems. The increasing reactive gas partial pressure curve for AlO_x generated in the Centura tool with HPPMS power is shown in Figure 1. Compared to AlO_x hysteresis curves produced with conventional pulsed DC power [13], the HPPMS hysteresis curve is broader in the transition region. This broadening may be due to the increased ionization from the HPPMS process and to the increased width of the racetrack region resulting from the higher voltages and currents of the HPPMS process.

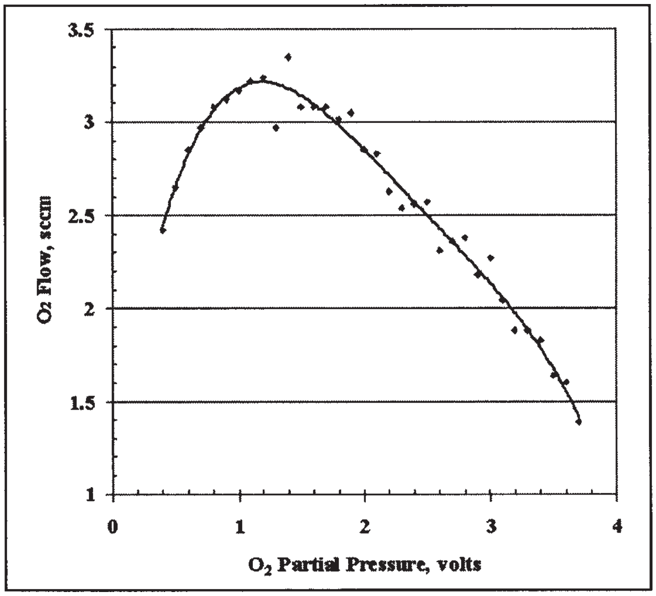

Figure 1: O_2 flow versus partial pressure for HPPMS reactively sputtered AlO_x films deposited in the Centura tool at an average target power of 2.2 kW.

AlO_x samples were deposited with different O_2 partial pressures in the Centura tool with an average HPPMS power of 1.0 kW for 15 minutes, and these films were characterized for thickness, crystallinity, and optical constants. The deposition rates and optical constants for these films as a function of O_2 partial pressure are shown in Table 2. The deposition rate for the AlO_x films decreased as the partial pressure of the reactive gas increased as is also observed for films reactively sputtered with conventional pulsed DC power. Once the O_2 partial pressure was 2 volts or more, the deposition rate did not change much since the target was fully poisoned.

Table 2: AlO_x HPPMS Films Deposited in the Centura Deposition Tool: O_2 Flows, Deposition Rates, and Optical Constants as a function of O_2 partial pressure.

O_2 Partial Pressure, volts	O_2 Flow, sccm	Rate, nm/min	n	k
0.5	1.45	26.1	2.00	-0.114
1.0	1.40	16.0	1.99	-0.021
2.0	1.00	10.3	1.52	-0.128
3.0	0.97	9.5	1.44	-0.137
4.0	1.25	11.0	1.50	-0.064

X-ray diffraction showed no diffraction peaks for any of the above films, which indicates that all of these films are amorphous. The response of the index of refraction (n) as a function of the reactive gas partial pressure for these films is similar to what has been observed for reactively sputtered AlO_x films using conventional pulsed DC power. As the reactive gas partial pressure increases, the index of refraction decreases as does the deposition rate. There is an optimum value for the reactive gas partial pressure that provides both the desired index of refraction with the highest possible deposition. The absorption coefficients for all of the films listed in Table 2 are very high. An optical microscope examination of the surface of the films showed a relatively rough surface, which may account for the high absorption values. Additional work needs to be done with these films to characterize their structure fully.

The results for the deposition of HPPMS AlO_x films in the batch system using the Torus 10 magnetron with non-rotating magnets were similar to those results for the films deposited in the Centura tool. Critical to both systems was the cleanliness of the target prior to the start of deposition runs. Used targets with built-up areas required removal of this material before reaching arc free operation. The arc handling capabilities of the experimental HPPMS power supply made it possible to get through this clean-up issue, and it also made it possible to minimize arcing during the reactive deposition. Without arc control, it would not have been possible to reactively deposit the AlO_x films with HPPMS power.

The O_2 flows, deposition rates, and optical constants for AlO_x films deposited in the batch system with an average HPPMS power of 1.44 kW are shown in Table 3. The deposition rate and the index of refraction dropped as the O_2 partial pressure increased. For these films the absorption coefficient (k) was lower than for those films deposited in the Centura system. The amount of arcing during deposition of the AlO_x films in the batch system was less than that observed in the Centura tool, which may have been partially responsible for the lower absorption coefficient.

Table 3: AlO_x HPPMS Films Deposited in the Batch Deposition Tool: O_2 Flows, Deposition Rates, and Optical Constants as a function of O_2 partial pressure.

O_2 Partial Pressure, volts	O_2 Flow, sccm	Rate, nm/min	n	k
0	0	27.6	-	-
0.8	3.10	18.3	2.35	-0.0002
1.0	3.81	13.1	1.87	-0.005
1.2	3.75	11.1	1.70	-0.005
1.4	3.59	9.4	1.50	-0.004
1.6	3.55	6.9	1.32	-0.002

CONCLUSIONS

HPPMS reactive sputtering of the dielectric film aluminum oxide is possible with partial pressure control of the reactive gas and with arc handling capabilities in the power supply. Target conditioning is essential to minimize arcing during the reactive depositions. For an equivalent amount of power, the deposition rate from an HPPMS process is lower than that done with conventional pulsed DC power. This lower rate can be attributed to the fact that a portion of the sputtered material becomes ionized and is attracted back to the target. Hysteresis curves for HPPMS AlO_x films are similar to those produced in pulsed DC systems. As the partial pressure of the reactive gas is increased, the film deposition rate decreases as does the index of refraction. There is an optimum reactive gas partial pressure that produces the desired index of refraction at the highest possible deposition rate.

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REFERENCES

1. V. Kouznetsov, K. Macák, J.M. Schneider, U. Helmersson, and I. Petrov, “A Novel Pulsed Magnetron Sputter Technique Utilizing Very High Target Power Densities,” Surf. Coat. Technol., 122, 290, 1999.

2. K. Macák, V. Kouznetsov, J. Schneider, and U. Helmersson, “Ionized Sputter Deposition Using an Extremely High Plasma Density Pulsed Magnetron Discharge,” J. Vac. Sci. Technol. A, 18, 1533, 2000.

3. A.P. Ehiasarian, R. New, W.-D. Munz, L. Hultman, U. Helmersson, V. Kouznetsov, “Influence of High Power Densities on The Composition of Pulsed Magnetron Plasmas,” Vacuum 65, 147, 2002.

4. A.P. Ehiasarian, W.-D. Munz, L. Hultman, U. Helmersson, and I. Petrov, “High Power Pulsed Magnetron Sputtered CrN Films,” Surf. Coat. Technol., 163-164, 267, 2003.

5. B. DeKoven, R. Weiss, P. Ward, B. Kakimoto, C. Marion, J. Xie, D. Devir, D.J. Christie, W.D. Sproul, R.A. Scholl, and A. Anders, “Carbon Thin Film Deposition Using High Power Pulsed Magnetron Sputtering,” 46th Annual Technical Conference Proceedings of the Society of Vacuum Coaters, San Francisco, CA, May 3-8, 2003, pages 158-165.

6. V. Kouznetsov, U.S. Patent No. 06,296,742 B1, “Methods and Apparatus for Magnetically Enhanced Sputtering,” October 2, 2001.

7. W.D. Sproul, D.J. Christie, D.C. Carter, F. Tomasel, and T. Linz, “Pulsed Plasmas for Sputtering Applications,” Surface Engineering, 20 No. 3, 1, 2004.

8. G.L. Anderson, P.W. Hammond, D.S. Yotive, U.S. Patent No. 5,241,152, “Circuit for Detecting and Diverting an Electrical Arc in a Glow Discharge Apparatus,” August 31, 1993.

9. G.N. Drummond, U.S. Patent No. 5,427,669, “Thin film DC Plasma Processing System,” June 27, 1995.

10. D.J. Christie, W.D. Sproul, D.C. Carter, and F. Tomasel, “A Novel Pulsed Power Supply with Arc Handling and Leading Edge Control as Enabling Technology for High Power Pulsed Magnetron Sputtering (HPPMS),” 47th Annual Technical Conference Proceedings of the Society of Vacuum Coaters, Dallas, Texas, April 24-29, 2004.

11. D.J. Christie, Ph.D. Thesis, “Power Conversion and Control for Pulsed Magnetron Reactive Sputtering,” Colorado State University, Fort Collins, CO, May 2004.

12. J.A. Davis; W.D. Sproul; D.J. Christie; M. Geisler, “High Power Pulse Reactive Sputtering of TiO2,” 47th Annual Technical Conference Proceedings of the Society of Vacuum Coaters, Dallas, Texas, April 24-29, 2004.

13. W.D. Sproul and B.E. Sylvia, “An Integrated Solution for Reactive Sputtering,” Vacuum Technology and Coating, Vol. 2, No. 8, 32, 2001.