Integrated Process Control for Reactive Sputter Deposition of Dielectric Thin Films

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ABSTRACT

Pulsed-DC reactive sputter deposition of dielectric films is a growing practice in the industrial and functional coatings industries. Transition region sputtering offers compositional control and high deposition rates but is possible only with active process feedback and appropriate in-situ instrumentation. Successful techniques use one or more process feedback signals, typically optical emission, partial pressure and/or target voltage. Ease of implementation and cost are highly dependent on system configuration and scale. Effective control systems for such applications usually require significant engineering effort involving sensing hardware, high-speed flow regulation, communication electronics, control electronics and software to interface and control all of the equipment. We have addressed these issues by developing an integrated approach to reactive sputtering process control. All electronics and algorithms with necessary I/O requirements can be incorporated directly into a pulsed-DC power supply. A key feature of our approach is piezo-driven mass flow controllers that replace the usual high voltage piezo-electric valves for fast gas flow control. We demonstrate transition mode control of aluminum-oxygen and silicon-oxygen reactive deposition processes with a novel controller incorporated into a pulsed-DC power supply. We show robustness of this process control scheme with real time data and deposited film properties.

INTRODUCTION

The sputter deposition of dielectrics has been gaining popularity in the industrial and functional coatings industries for many years. High frequency AC sputtering using RF power and dielectric targets is gradually being replaced by more economical and production friendly mid-frequency AC and pulsed-DC reactive sputtering techniques, but issues with process control, deposition rate and film quality still plague many who are trying to exploit this promising approach.

Dielectric deposits can be produced using metallic targets (such as aluminum or silicon) sputtered in the presence of a reactive gas such as oxygen. Power delivery and target materials used in reactive sputtering are typically less expensive than the RF alternatives, but for optimal performance the reactive approach requires additional monitoring and control provisions not normally found in the basic sputtering system.

A common challenge in many reactive sputtering processes is target condition control (see Figure 1). As reactive gas is added to the deposition environment the behavior of the target transitions from being representative of the metal to being more representative of the compound being formed in the reaction. For most dielectric deposits, this transition can dramatically change the deposition behavior and lead to decreased deposition rates along with process instabilities such as arcing.

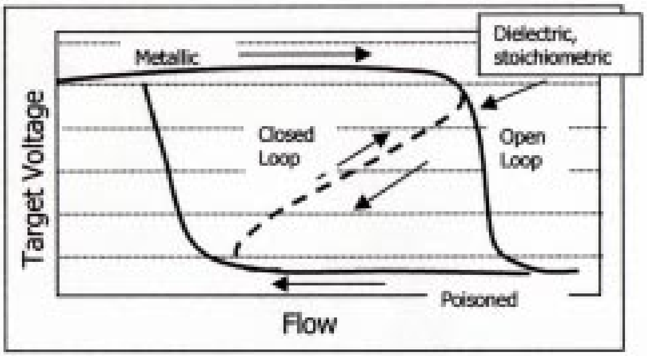

Figure 1: Typical, open-loop, hysteresis behavior seen in reactive deposition of dielectric films.

To guard against this transition, most reactive processes historically have operated in the “poisoned” regime, whereby most of the target is insulating. While this approach avoids the transition event the result can be a low deposition rate and limited composition control. In recent years, various techniques for active control of target condition have been gaining acceptance offering dramatic rate enhancement along with improved composition control. Optical emission, partial pressure and target voltage [1,2,3] have all been demonstrated as viable feedback mechanisms for control of such processes, but each approach adds cost and complexity so expense and ease of implementation are keys to their ultimate acceptance.

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This study explores a straightforward and economical approach for incorporating active process control in a reactive sputtering deposition using target voltage feedback. While our approach lacks some capabilities offered by more elaborate techniques it excels in its simplicity and ease of implementation. It employs pulsed-DC power for arc management and an active gas flow control system for target condition control. We show how stable transition region operation can be accomplished in difficult-to-control reactive processes using very conventional hardware and limited system modifications.

APPROACH AND APPARATUS

Pulsed-DC power is commonly used for arc suppression during reactive sputtering of dielectrics [4]. Arcing results from charge buildup on insulating layers reaching a point of voltage breakdown. Besides its adverse effects on film quality and particulate levels, arcing can also create severe instabilities in a deposition process making control and repeatability difficult. Reverse voltage pulsing has been shown to be quite effective at dissipating accumulated charge during a reactive dielectric deposition. Proper optimization of pulsing parameters [3,5,6,7] has been shown to virtually eliminate the occurrence of arcs allowing for long-term stable process operation.

Pulsing alone, however, is only one element in a successful implementation of reactive dielectric sputtering. To control the process in the high rate, transition region, an active control loop is required to regulate the flow of reactive gas to the process and maintain the deposition conditions in the desired range [6,8]. Partial pressure and optical emission techniques provide a means for compositional feedback from the bulk of the plasma. These techniques both employ external sensors to monitor either the reactive gas or metal constituency in the plasma and feed a signal to a high-speed proportional valve. The alternative discussed here is the direct monitoring of the target voltage for this purpose. Target voltage (as shown Figure 1) can be dramatically affected by surface target condition. For the deposition of aluminum-oxygen and silicon-oxygen compounds, target transition from metal to insulator often represents a drop in sputtering voltage of greater than 100 Volts. This signal provides a direct and extremely fast feedback of the electrical condition of the target offering a convenient means for controlling the deposition process itself.

The key to successful implementation of an active control scheme often lies in two key elements: timely feedback from the process and high-speed response of the regulating variable. In our case the speed of the control loop must exceed the reaction rate experienced on the target surface. Voltage control provides real-time feedback from the process with response limited only by the sampling electronics used to monitor the signal. Fast and accurate reactive gas flow regulation is the other critical element required. It is commonly understood that thermal mass flow controllers (MFCs) lack both the response and accuracy needed for such applications. A common approach used in the methods described above is the incorporation is a high voltage, fast-acting proportional valve (usually a piezoelectric) to give rapid response to the control loop. Although very fast and accurate, these valves tend to be quite expensive and can add complexity to the system by requiring high voltage control signals. Another shortcoming is the lack of any feedback mechanism for flow monitoring purposes.

For this implementation we use two types of MFCs that both regulate flow through piezoelectric valves. The Mach One™ MFC manufactured by Advanced Energy is well suited for this application because it offers fast response without overshoot or “ringing” often associated with thermal flow devices. This eliminates the need for a separate piezo valve and because it operates on a 0-5 V input it provides a drop-in replacement for many of today’s mainstream controllers.

In the second implementation we used a modified Aera PrimAera™ thermal MFC for reactive gas regulation. This device is equipped with a high-speed piezoelectric control valve. The controller was modified for this work to accept a 0-5 V control signal directly to the piezo drive. This modification allowed for direct control of the fast valve by bypassing the slower thermal feedback loop. In both cases, control signals are kept in a manageable range (0 to 5 Volts) and separate feedback is available so monitoring actual flow during operation is possible.

The final piece of the system is the controller electronics itself. In this study a simple PID controller was used with a single input channel (target voltage) and single output (flow signal). Sputtering voltage was fed directly from the pulsed-DC power supply to the input of the controller where an error value and flow signals were calculated and sent to the flow device. Input signals were sampled at > 1000 Hz and output was updated every 150 ms. Figure 2 shows schematically the layout of the control system used.

Data were generated in two separate systems. One was a medium sized, open-volume, cylindrical (~30” dia. x 18” deep) vacuum chamber with a single 6” Torus 10 balanced-field magnetron. The other was a larger volume (~48”x36”x30”) open chamber with a 12”x44” planar magnetron. Uniform distribution of reactive gas was achieved using rather simple diffusers, examples are shown in Figure 3. Both chambers were turbomolecular pumped to base pressures below $5x10^{-6}$ Torr prior to deposition runs. Aluminum and silicon targets were sputtered using an Advanced Energy “Pinnacle™ *plus*” pulsed-DC power supply.

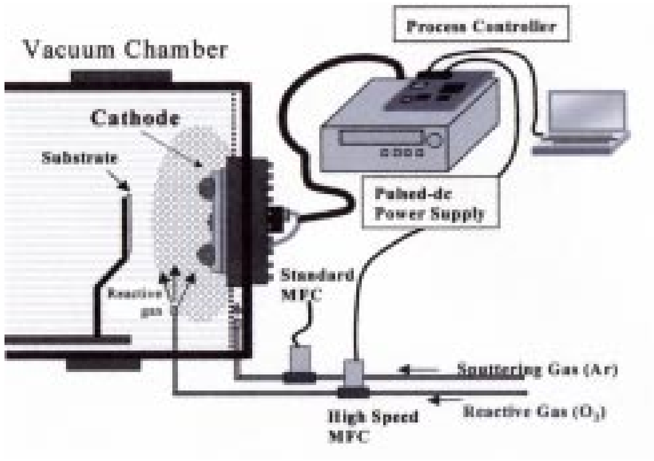

Figure 2: Key elements of the control system used in this study.

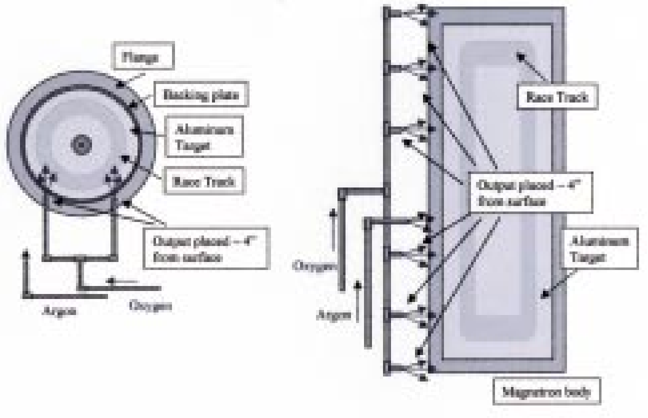

Figure 3: Reactive gas was introduced using simple manifolds directing flow across the plane of the targets, at ~ 4" from the target surface.

Deposition rates were measured in-situ using a Sycon STM-100 quartz crystal monitor and on glass substrates using a Tencor P200 profilometer. Optical properties were measured on a Gaertner L116 ellipsometer and a Filmetrics F20 spectrometer.

Power to the cathode was held constant during all tests by power regulation in the Pinnacle™*plus*. Power regulation is chosen to allow the current and voltage to vary depending on the target condition giving mechanism to the active voltage-based control loop. The performance of the control loop was evaluated under various sputtering conditions on both aluminum oxide and silicon oxide processes. Pulsing parameters (the combination of pulse frequency and duty factor) were chosen to minimize arcing based on previous work [3]. The two magnetrons allowed the scalability of the control scheme to be tested at low to moderately high power levels.

PID control loop parameters were established to provide visibility of the process stabilization dynamic. In a production process the control loop PID parameters would also be optimized to reduce overshoot and improve response time.

RESULTS

The first set of tests was performed using the small 6" diameter magnetron sputtering aluminum in the presence of oxygen. The Mach One™ flow controller was used in this set-up and the stabilization shown in Figure 4 illustrates mid-transition control achieved and maintained over an extended period of operation. In the initial portion of the chart power is on but no oxygen is yet present. The control loop is initiated at time = 10 seconds and the PID loop introduces oxygen flow to drive target voltage toward the set point. To achieve control the sonic MFC is rapidly driven by the PID loop to balance the process within the transition region. After a brief overshoot in flow, control at the voltage setpoint is established at approximately 25 seconds into the run.

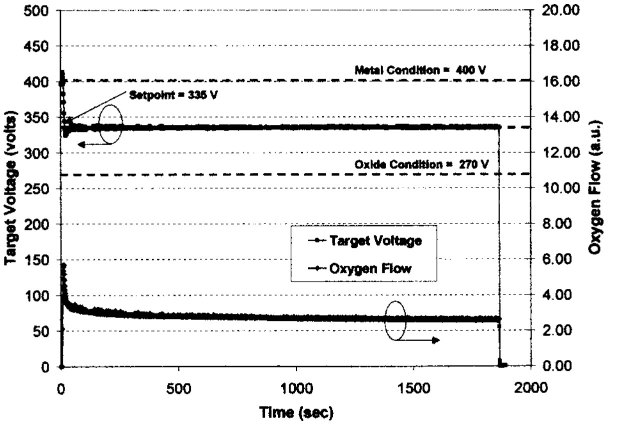

Figure 4: Stabilization behavior observed on 6" magnetron in 3.5 mTorr Argon, 1000 Watts, 100 kHz, 90% duty factor depositing AlO_x.

Power for this test was regulated at 1 kW, pulsed at 100 kHz and 90% duty factor. Argon pressure was 3.5 mTorr. As shown, target voltage in pure argon (metallic condition) is approximately 400 Volts; and the dielectric condition was at 270 Volts. In this case the voltage setpoint was approximately 335 Volts representing 50% into the target transition. After the initial stabilization, control was maintained for a 30-minute period before power to the process was turned off.

Aside from minor control adjustments, it was noted that the nominal oxygen flow falls slowly during the first 15 to 20 minutes of a deposition cycle. During this time the control loop maintains a stable operating voltage. This was a commonly observed behavior and is attributed to the control loop responding to a combination of thermal and surface gettering effects in the chamber that tend to have long stabilization times.

Figure 5 shows the classical "S" curve generated in this set-up. We compare the active loop results to data generated in an open-loop mode. In the case where active control is enabled, the complete transition region is accessible for stable operation. By selecting the target voltage desired, the active control regulates oxygen flow at the appropriate point to maintain the target condition and avoid poisoning. Control points were selected such that the entire curve was mapped from the metal to the dielectric target condition. While some hysteresis is still present in the closed-loop curve, it is much reduced over the open-loop case. This illustrates the ability, using this technique, to access otherwise forbidden regions of process space from metal rich to oxygen rich and all points between. These results are confirmation that the voltage feedback is effective for control and that the methodology used including the PID controller and the sonic MFC have adequate response and accuracy to achieve and maintain stability at each point in the transition.

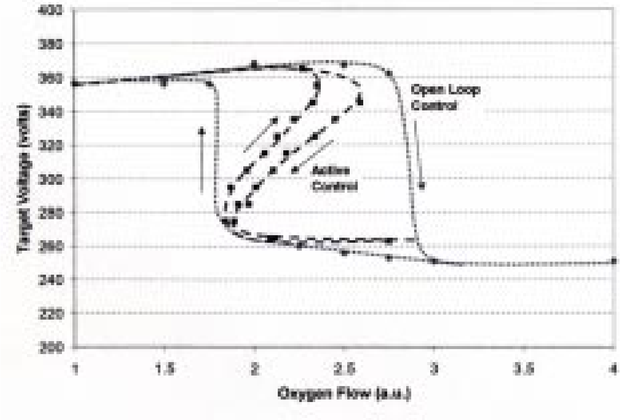

Figure 5: Target transition in open and closed loop control; 1000 Watts pulsed-DC at 20 kHz, 90% duty factor, 3.5 mTorr Ar depositing AlO_x.

The effects of power are shown in Figure 6 in a family of transition curves generated from 750 Watts to 1.5 kiloWatts. Pulsing frequency was 350 kHz at 86% duty factor; argon pressure was again 3.5 mTorr. As power increases, nominal voltages increase. Higher powers result in increased aluminum sputtering and, as shown, the amount of oxygen needed to transition the target increases as well.

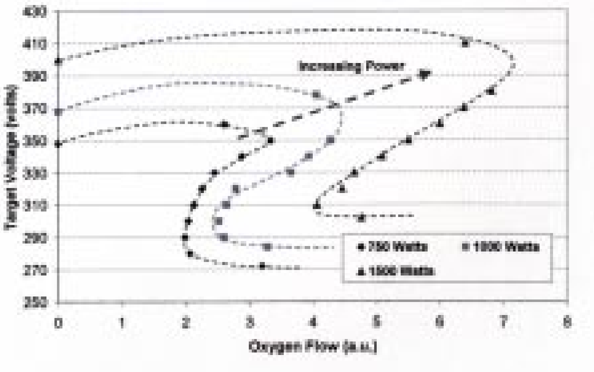

Figure 6: Target transition at 750 Watts; 1000 Watts and 1500 Watts at 350 kHz; 86% duty factor depositing AlO_x.

Optical transmission of deposited alumina films is given in Figure 7. As expected, films deposited in the metal rich (high voltage) region of the transition curve had poor transmission properties. A wide process window producing optically clear films was observed from approximately 20% to 70% down the transition curve. Transparency falls off slightly at the 90% condition due to the film likely becoming hyper-stoichiometric. A sampling of refractive index measurements was also taken on the clear films yielding values from 1.58 to 1.62.

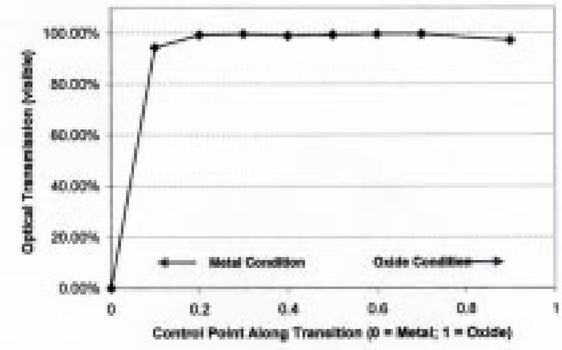

Figure 7: Optical transmission data from aluminum oixide films deposited at 1000 Watts; 100 kHz; 90% duty factor.

In addition to process stability and stoichiometry control, closed-loop transition region sputtering also offers significant rate improvements over RF sputtering and DC sputtering from poisoned targets. Figure 8 illustrates this with deposition rate plotted against target voltage at different points through the transition region of the aluminum target. The fully metallic target operates at approximately 400 Volts while the fully poisoned target is found at 270 Volts. Maximum rates are observed toward the metallic condition and as the target transitions to lower voltages deposition rate falls in a proportional fashion. Minimum rates are observed when the target is fully poisoned. From the metallic to the poisoned condition rates are seen to fall nearly ten-fold.

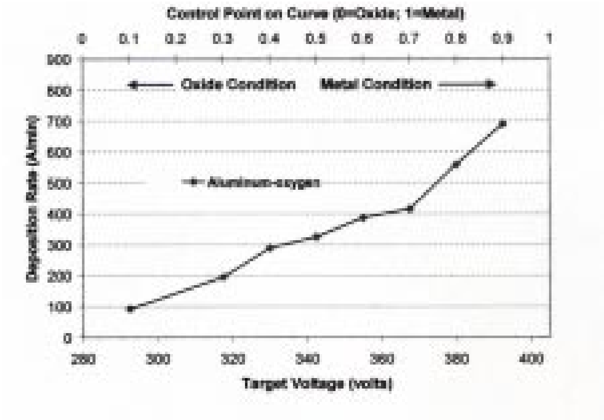

Figure 8: Deposition rate as a function of position on transition curve for aluminum oxide film deposited at 1000 Watts; 100 kHz; 90% duty factor.

Aluminum oxide deposition is only one of many hard-to-control reactive processes. Silicon oxide reactive sputtering is another. Figure 9 shows the transition curve generated from the 6" magnetron loaded with a silicon target sputtered at 500 Watts under active voltage control. Due to cooling issues on this target, higher power testing was not possible. In the range tested, control was stable throughout the transition region allowing the complete curve to be generated.

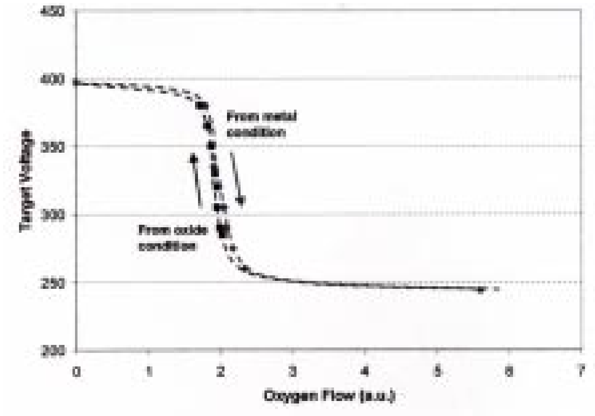

Figure 9: Transition curve generated from silicon target on small magnetron at 500 Watts pulsed at 100 kHz, 60% duty factor.

The ability to scale this technique is important as industrial users drive toward larger systems and employ higher power densities. Figure 10 shows stabilization curves for aluminum oxide sputtered from our large area (12"x44") cathode, this time using the modified PrimAera flow controller. Here 10 kW was used to sputter at 2.0 mTorr. Despite significant arcing (forcing the low duty factor) stable operation was achieved at a voltage deep into the transition region. The complete "S" curve was then generated at 17.5 kW to again show access to the entire process space (Figure 11) afforded by this technique.

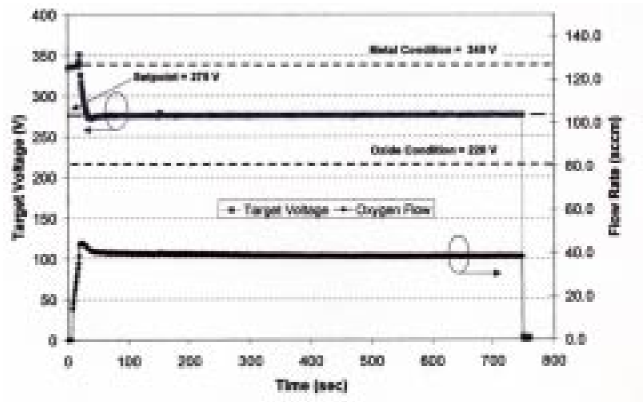

Figure 10: Large cathode transition region control for aluminum oxide at 10 kW; 100 kHz; 60% duty factor and 2.0 mTorr.

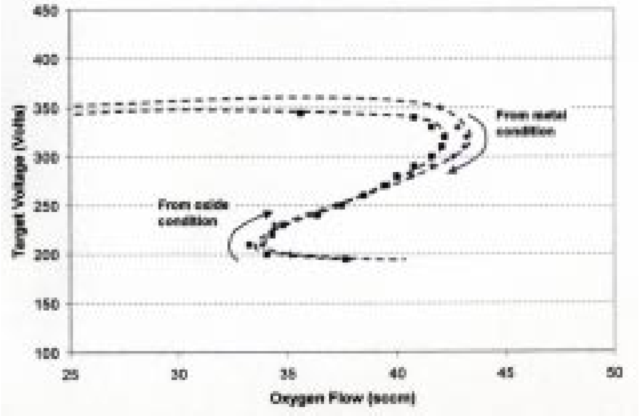

Figure 11: Large cathode aluminum oxide transition curve mapped at 17.5 kW; 75 kHz; 63% duty factor and 2.0 mTorr.

CONCLUSION

Target voltage control can offer an uncomplicated but effective means for closed-loop control of reactive dielectric sputtering. The technique can be implemented without the addition of sophisticated diagnostics or instrumentation and is shown to be readily scalable. Stable control throughout the transition region of both silicon-oxygen and aluminum-oxygen processes were demonstrated under a variety of pulsed-DC conditions on both small and large-scale planar magnetron sources.

In this method, pulsed-DC power minimizes the effects of arcing and target voltage feedback is taken directly from the power supply output. A PID loop compares voltage feedback with user-defined setpoint and output is directed to fast-acting mass flow controllers. All communications are accomplished utilizing low voltage analog signals and piezoelectric drivers in two different MFCs are shown to provide adequately fast response. Simple gas distribution manifolds are demonstrated to offer stable control on both small and large-scale magnetron targets.

The simplicity of this method lends itself well to a fully integrated approach whereby the process control electronics are incorporated directly into the power supply itself. Such a system will monitor voltage internal to the supply and deliver appropriate control signals in the form of analog output to the reactive gas flow controllers. The expandability of such an approach could also include analog inputs to monitor other external sensors and additional outputs to allow for zonal control in even larger scale systems.

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