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PROCESS: DEPOSITION

An Economical Method for Process Control in Pulsed-DC Magnetron Reactive Sputtering

By D. Carter, D. Madsen and W. D. Sproul, *Advanced Energy Industries, Inc.*

Low deposition rates and poor process stability often plague DC and pulsed-DC reactive magnetron sputtering of insulating films. But high-rate deposition of dielectrics by reactive sputtering can be achieved using a relatively simple and economical approach.

Using pulsed-DC power to manage arc activity and employing a low-cost, easy-to-implement closed-loop control scheme for reactive gas can maintain target conditions and hold the process in the high deposition rate transition region. The capabilities of this approach were evaluated for reactive deposition of aluminum oxide at various powers, and pulsing conditions and performance were verified throughout the target transition region. When properly adjusted, the process was shown to deliver long-term stability and to produce clear, dense, dielectric films.

INTRODUCTION

The sputter deposition of dielectrics has been an active area of research for many years. High-frequency AC sputtering using RF power and dielectric targets has historically been the reluctant choice, but the high cost of power and low deposition rates typically make this approach less favorable for volume-driven applications. Dielectrics can also be deposited by *reactive sputtering*, which is performed with metallic targets (such as aluminum or silicon) in the presence of a reactive gas such as oxygen. A DC-based power delivery system and metallic targets are less expensive, but this method often presents new problems for the process engineer.

A common issue in reactive sputtering is target condition control. Most reactive processes today are operated in the "poisoned" condition, whereby most of the target is insulating,

to avoid repeatability and instability issues associated with target transitions (see insert). In recent years, however, various techniques for active control of target condition have been gaining acceptance. Optical emission, partial pressure and target voltage control techniques have all been successfully incorporated in reactive sputtering, but each approach carries the risk of cost and complexity. There is a straightforward and economical approach for incorporating active control in a reactive sputtering process using target voltage feedback, and while this approach lacks some of the capabilities offered by some of the 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 transition control, allowing the user to establish stable transition region operation in many difficult-to-control reactive processes.

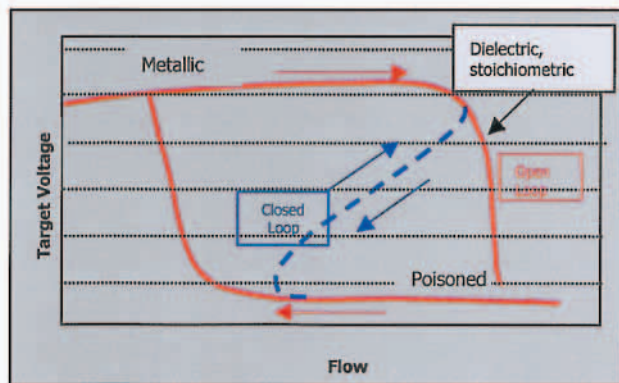
KEY ELEMENTS FOR CONTROL

Pulsed-DC is commonly used for arc suppression during reactive sputtering of dielectrics. Arcing results from charge buildup on insulating layers reaching a point of breakdown. This is a major issue in a process where the principal objective is to form an insulating deposit. To combat this, reverse voltage pulsing has been shown to be quite effective at dissipating accumulated charge. The prospect of optimizing the reverse voltage pulse has been studied to show that, under certain conditions, arc-free operation can be achieved by defining a "critical" reverse time to provide the necessary amount of charge scrubbing to eliminate build-up and thus the breakdown event.

Pulsing alone is only one element in a successful reactive sputtering implementation. To control the process in the transition region an active control loop is required to regulate introduction of reactive gas to the process. Partial pressure and optical emission techniques provide direct feedback of the plasma composition and thereby offer a means to control reactive gas introduction. An alternative to this is the direct monitoring of the target voltage for this purpose. Target voltage (as shown in the Target Voltage Hysteresis insert) can be

TARGET CONDITION AND VOLTAGE HYSTERESIS

A major challenge in many reactive sputtering processes is associated with "target condition" control. Target condition refers to the electrical state of the immediate surface of the target. This condition can be critical to the stability of the sputtering process. To achieve highest deposition rates in a reactive sputtering process, the target must remain largely in a "metallic" or electrically conductive state. When reactive gas is introduced into the sputtering environment (see graph below), the surface of the target will transition to become insulating. Typically the target voltage drops as the target surface becomes insulating. As gas flow increases, target voltage continues to drop, which causes sputtering rates to decrease, thereby causing the target to become further coated with the insulating film. Arcing becomes more prevalent. In the limit, the target surface is insulated to the point where the target voltage is at a minimum. This is the "poisoned" condition. To return to a metallic condition, the gas flow needs to be reduced significantly. This is shown in the hysteresis of the target voltage/gas flow curve. A stable, high rate reactive deposition represents a delicate balance between the point of complete reaction at the substrate (to the desired stoichiometry) and the point of complete transition of the target surface chemistry. By eliminating arcs, pulsed-DC power aids in achieving this balance, but pulsing alone is not adequate to provide necessary stability for long-term operation and target condition control. To achieve this level of control, a dedicated feedback loop providing target condition information and direct regulation of reactive gas introduction is required.



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dramatically affected by reactive gas partial pressure. For the deposition of aluminum-oxygen and silicon-oxygen compounds, this technique provides rapid feedback of the electrical condition of the target.

The key to successful implementation of all these techniques is in the response time of the feedback and gas flow regulation. The speed of the control loop must exceed the reaction rate experienced on the target surface. Fast and accurate response of the reactive gas regulation is critical. It is commonly understood that thermal mass flow controllers (MFC) lack both the response and accuracy needed for such applications. A common approach used in the methods described above is the incorporation of a high-voltage, fast-acting valve (usually a piezo-electric) 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 a high-voltage control signal. Another shortcoming is the lack any feedback mechanism for monitoring purposes.

For this implementation, we used a new type of MFC that regulates flow through a sonic orifice using a built-in piezo-electric valve. The Mach One™ MFC, manufactured by Advanced Energy, is well-suited for this application because it offers fast response without the overshoot or "ringing" often associated with thermal flow devices. This eliminates the need for a separate piezo valve, and since it operates on a 0-5 V input it provides a drop-in replacement for many of today's main-stream controllers. The details of this new flow device are shown in the "Sonic MFC" insert.

TECHNIQUE

Data were generated in a large, open-volume, cylindrical (about 30 inches in diameter by 18 inches deep) vacuum chamber. The chamber was pumped with a Leybold Turbovac 1000 turbo molecular pump backed by an Edwards EH250/E2M40 blower stack. A six-inch aluminum (99.995 percent) target mounted on a Torus 10 balanced-field magnetron served as the sputter source. Power to the cathode was applied using an Advanced Energy Pinnacle™ Plus pulsed-DC power supply.

Control loop and deposition tests were performed from 2.0 – 6.0 mTorr after pumping to a base of 5.0×10^{-6} Torr or less. 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.

During all tests, power to the cathode was held constant 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 control loop.

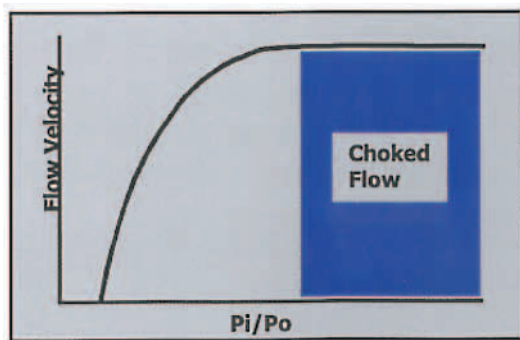
Argon was fed to the chamber via a dedicated gas line positioned behind the chamber shielding, and the flow of Argon was regulated using a standard, thermal mass flow controller.

Oxygen was delivered to the process using the sonic MFC receiving its control signal from a PID controller. The flow signal was based on the error value between measured target voltage and the target voltage set point. Set point was the user-defined voltage, selected according to the desired process result

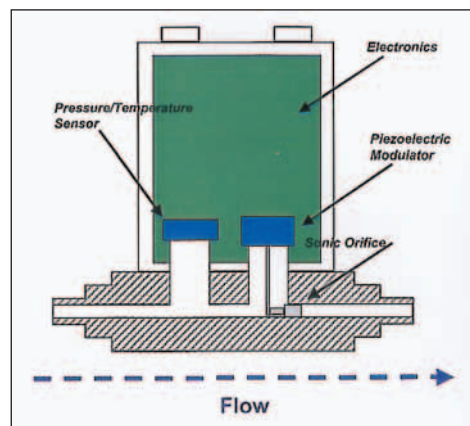
SONIC MFC – A NEW TECHNIQUE FOR REACTIVE GAS FLOW REGULATION

The Mach One™ sonic MFC used in this study features a high-speed piezoelectric element to regulate gas flow through a sonic orifice. Pressure and temperature monitoring of the gas stream provide active feedback of input conditions. Operation in the “choked flow” regime allows for precise flow regulation using pulse with modulation (see below). Compared to thermal MFCs, these devices offer much faster response (<200 ms), no overshoot and wider dynamic range, all of which are critical elements for successful gas flow regulation to a reactive sputtering process.

The Mach One™ uses well-established physical principles for calculating and regulating mass flow. As the pressure drop (P_{input}/P_{output}) across a nozzle increases, flow velocity through the element increases exponentially until the speed of sound is reached.



Once sonic velocity is reached, the flow becomes “choked,” or flow limited. Further increases in pressure drop have no effect on the mass flow rate. For a particular gas, the mass flow rate becomes directly proportional to the square root of the absolute temperature. The Mach One™ generates the desired flow rate by modulating the flow through the nozzle between two flow states: No Flow and Choked Flow. By varying the on/off time ratio between states – pulse width modulation – a means of mass flow control is achieved.



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and the point on the transition curve desired for operation. Set point can be represented as a “turndown” percentage defined as the proportional amount of the voltage drop measured from metallic to dielectric target condition. A process operated at 0 percent turndown would therefore be operating in the metallic (high voltage) target condition while 100 percent turndown would be at the dielectric (low voltage) condition. Clear films resulted from set points ranging from approximately 20 percent turndown to 100 percent turndown.

DEPOSITION RESULTS AND CONTROL LOOP PERFORMANCE

The process and control loop were tested across a wide range of process conditions and pulsed power set points. Pulsing parameters (combination of pulse frequency and duty factor) were chosen to minimize arcing based on previous work. PID control loop parameters were established to provide visibility of the process stabilization dynamic. Of course, in a production process the control loop PID parameters would also be optimized to reduce overshoot and increase response time.

Figure 1 shows aluminum oxide process stabilization and control at a set point of 50 percent turndown. Initially 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 an initial overshoot in flow, control at the voltage set point is established at approximately 25 seconds into the run. Power for this test was regulated at 1kW, pulsed at 100 kHz and 90 percent duty factor.

Argon pressure was 3.5 mT. In this case the 50 percent turndown voltage was approximately 340 volts. As shown, target voltage in pure argon (metallic condition) is approximately 410 volts; the dielectric condition in this case was 270 volts. After the initial stabilization, control was maintained for over 30 minutes before power to the process is turned off.

Through the extended run shown in Figure 1, aside from minor control adjustments, the nominal oxygen flow falls slowly during the first 15 to 20 minutes while target voltage remains level. This behavior 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 2 shows the capability of this approach for allowing operation throughout the target transition providing access to the otherwise forbidden regions of process space not achievable in open-loop operation. In the open loop case, oxygen must flow at a critical point, near the transition, to form the reacted deposit. Without active control to regulate the process, at this point there is no protection against a spontaneous transition leading to a poisoned target in the dielectric condition. A severe hysteresis also exists requiring significant reduction in oxygen partial pressure to force a transition back to the metallic condition.

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 precise point to maintain the target condition and avoid poisoning. Control points can then be selected such that the entire “S” curve can be mapped out from metal to dielectric target condition. While some hysteresis is still present in the transition curve, it is much reduced over the open loop case.

The ability to control not only at a single point but also throughout the target transition region ensures composition control of compound depositions. This capability is confirmation that the voltage feedback is effective for control and that the control scheme 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 3 shows a family of transition curves generated as power is varied from 750 watts to 1.5 kilowatts to show its effect on transition behavior and to demonstrate control capability throughout the transition in each case. The pulse frequency here was 350 kHz at 86 percent duty factor. Argon pressure again was 3.5 mT.

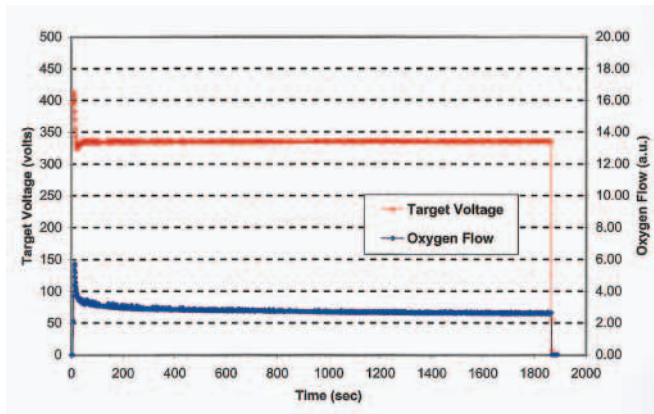
As power increases, nominal voltages for both the metallic and dielectric condition increase. Furthermore, as expected, the amount of oxygen needed to transition the target increases. This is due to the increased metal sputtering taking place at the elevated powers and an associated increase in oxygen required

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to react with the sputtered metal.

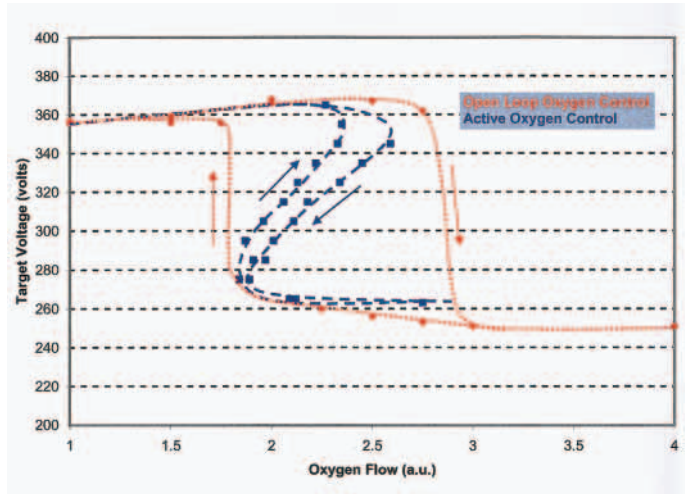
In addition to process stability and stoichiometry control, closed-loop transition region sputtering also offers significant rate improvements over RF sputtering and DC sputtering from poisoned targets. Figure 4 illustrates this with deposition rate

FIGURE 1



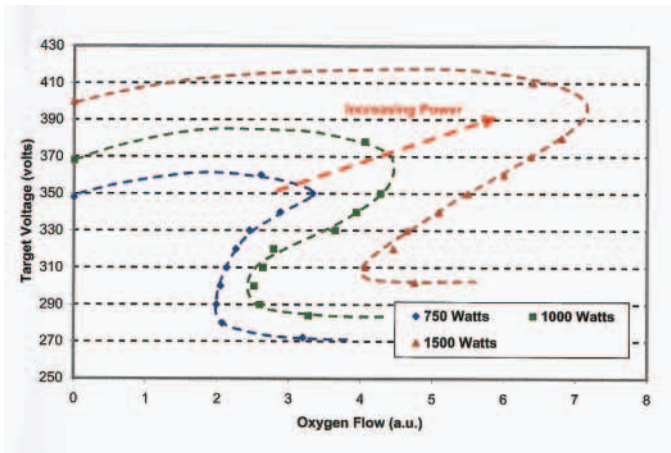
Active control dynamic at 1000 W, 100 kHz, 90% duty factor.

FIGURE 2



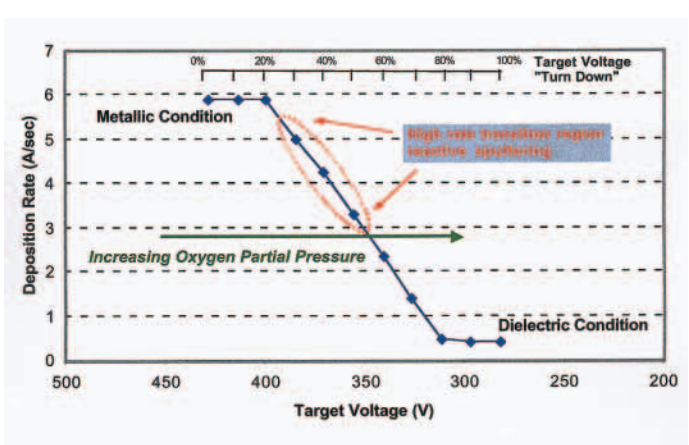
Open-loop versus closed-loop at 1000 W, 20 kHz, 90% duty factor.

FIGURE 3



Closed-loop transition region operation at 750 W, 1000 W and 1500 W @ 350 kHz, 86% duty factor.

FIGURE 4




AlO_x deposition rate changes through target transition. 750 watts, 50 kHz, 80% duty factor.

plotted against target voltage at different points through the transition region of the aluminum target. Transition of the target, driven by oxygen addition, is again represented by the turn-down percentage. The fully metallic condition is 0 percent turned-down at approximately 430 volts while the fully poisoned target is 100 percent turned-down at 280 volts. Maximum rates are observed in the metallic condition. As oxygen is added, the target voltage begins to drop and 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 more than ten-fold.

Along with deposition rate, film properties also vary through the target transition but, as shown in Figures 5 and 6, films with good optical quality and a dense glassy appearance are achieved well before the target transitions to the dielectric condition. In fact, fully clear films in this case occur at only 20 percent turn-down. As shown in Figure 4, deposition rates in this region are nearly 10 times that observed when the target becomes poisoned. While the appropriate operating position used will vary by application and desired film properties, these charts illustrate the economic value in this approach being the ability to produce highly transparent compound films at many times the deposition rate available without active control.

SUMMARY

By combining pulsed-DC power for arc reduction with an active closed-loop control system for gas regulation, process stability can be achieved throughout the transition region for

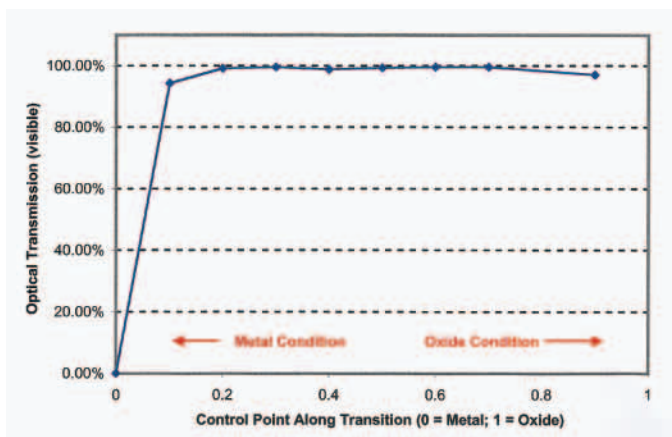
difficult-to-produce dielectric oxides. An economical approach to accomplishing this involves the use of a high-speed sonic MFC driven by using a PID controller based on target voltage feedback. Aluminum oxide deposition was successfully demonstrated using this approach, which is applicable for other compound dielectrics possessing similar electrical behavior. The high-speed sonic MFC used was effective in providing response and accuracy necessary for control throughout the target transition curve while providing stable operation during long deposition cycles. Transition-region control provides significant deposition rate improvement over operation in the dielectric (or "poisoned") condition. Transparent films produced in the transition region were deposited at nearly 10 times the rate measured from a poisoned target operated under similar power conditions. 

Dan Carter is Applications Manager for the Applications Research and Development group at Advanced Energy Industries Inc. He received BS and MS degrees in material science and engineering from Purdue and Northwestern Universities.

David Madsen is an AE Staff Engineer in the Applications Research and Development group. Madsen earned a BS degree from Brigham Young University in mechanical engineering and physics.

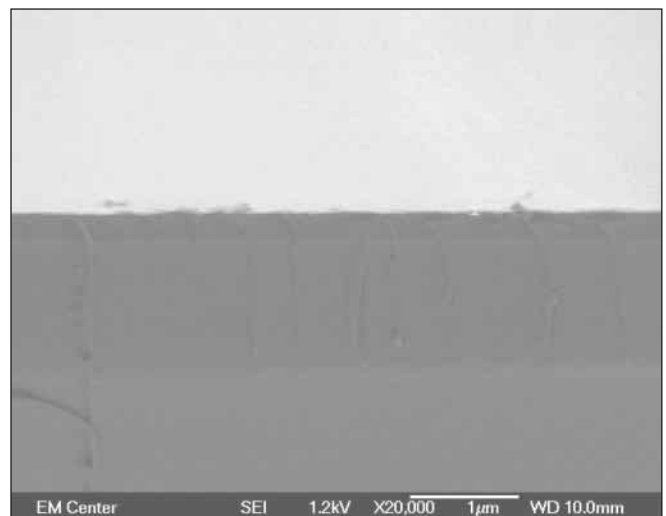
William Sproul, Ph.D., joined AE in 2002 as a Senior Scientist for Power Systems Research and Development. He received his Ph.D. degree in materials science engineering from Brown University.

FIGURE 5



AIOx film transmission through target transition.

FIGURE 6



Aluminum oxide film deposited in target transition using closed loop reactive gas control.

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