Reactively Sputtering High on the Transition Curve Using a Few Inexpensive Components

J. German, Sage Electrochromics, Faribault, MN; and
D. Pelleymounter, Advanced Energy, Northfield, MN

Key Words: Reactive deposition; Sputtering deposition; Mass flow measurement/control; Partial pressure control

ABSTRACT

Riding the "Sweet Spot" of the Transition Curve where reactive properties are achieved while getting higher deposition rates has been the goal for process engineers. There has been lots of time and many papers dedicated to discussion of this topic. These discussions have traditionally resulted in using expensive high tech equipment. Here is an example of a simple and inexpensive way to do the same thing.

DISCUSSION

The purpose of this paper is to show how to run anywhere on the hysteresis curve during reactive sputtering. This is accomplished using components that may already be on your machine and by adding only a couple of inexpensive boxes or PID loops in the machine's control logic. Historically, this same type of process has required components that, while produce the same desired results, are quite expensive and need more than their share of "baby-sitting" and calibration by the operator and/or the process engineer. Those types of components include the OGC and the PEM among others.

For this paper we used a 52-inch long rotatable cathode with a doped silicon target. Our machine is an old one that has been rebuilt and reworked more than a machine should be in its lifetime. We did nothing special to the gas manifold and we used a DC power supply. We wanted this to be the "worst case" that was available.

The components that were already on our machine were: an Advanced Energy Pinnacle II 40 kw DC power supply, an MKS 247 quad MFC readout, an MKS Type 250 pressure regulator combined with a baratron and a 248 control valve. We added an Emco Mach 1 MFC, which was chosen for its fast response time, and we built a cheap signal conditioner box for the Mach 1 setpoint. The schematic for this simple signal conditioner box is shown in Figure 1.

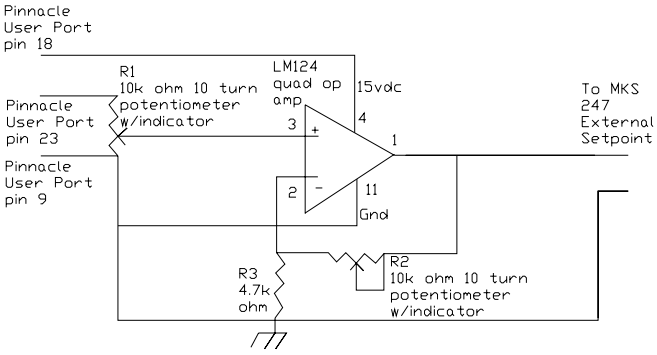

Figure 1: Signal Conditioner Schematic

The sputtering zone is pumped by two high vacuum pumps and is approximately 18 inches x 30 inches x 96 inches in size.

In order to keep the background pressure constant, the baratron senses the pressure in the sputtering zone and sends the signal to the 250. The 250 has a pressure setpoint given by the operator and sends a signal to the 248 control valve to adjust the background argon to satisfy the setpoint. The pressure controller is not necessary but was useful in that it simplified mapping the transition curve at a constant total pressure.

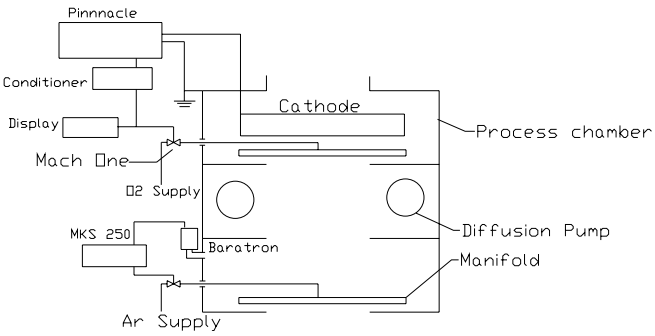

Figure 2: Process Chamber Setup

45th Annual Technical Conference Proceedings (2002) ISSN 0737-5921

We connected the 500 sccm Mach 1 to one of the channels of the 247. We set that channel to look at an external setpoint. The Mach 1 will adjust the total flow of the reactive gas to the sputtering zone. The signal conditioner box sees the voltage output from the user port of the Pinnacle (0-10 V = 0-1500 V). Since the Pinnacle does not go above 800 Vdc the user port output cannot exceed 5.3 Vdc. The Mach 1 uses a 0-5 Vdc input for its setpoint (0-5 V = 0-500 sccm). The operator adjusts this Pinnacle-to-Mach 1 signal using the Level Control R1 on the signal conditioner. This is a 10 kΩ multi-turn potentiometer with a mechanical numerical position readout. This signal goes through the 247 external setpoint input. We should also mention that the 247 provides the +/-15 Vdc for the MFC operation. The Mach 1 provides the reactive gas according to the signal it receives from the conditioner box.

As the reactive gas flows into the sputtering zone, the partial pressure will go up and the voltage on the silicon target will go down. We know from daily production that this particular target will go from 680 Vdc in the full metal mode to 380 Vdc in the fully poisoned mode. Using the Level Control R1 of the signal conditioner we adjusted the voltage for the fully poisoned mode. We then slowly set the signal conditioner to run the process at about 520 Vdc. We noticed that while it was fairly stable it wanted to creep up a volt or two—slowly at first and then speeding up as the voltage got higher. The reactive gas control is shown in Figure 3.

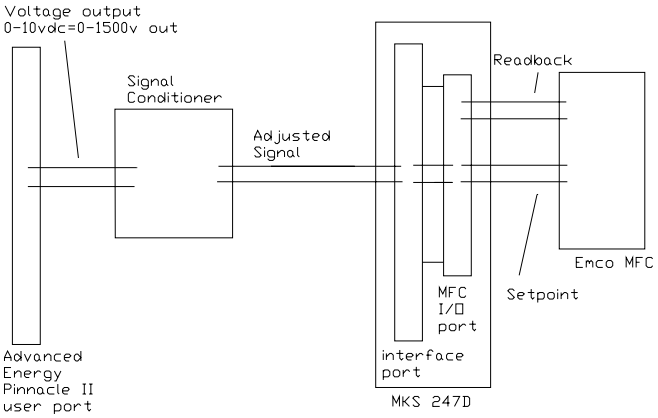

Figure 3: Reactive Gas Control Diagram

We then adjusted the Gain Control on the signal conditioner box. This is R2 on the schematic (Figure 1) which is a 10 kΩ multiturn potentiometer with a mechanical numerical position readout. The active component in the signal conditioner is an LM124 op amp that is used as a non-inverting amplifier. You will notice that R3 is a 4.7 kΩ. The gain is calculated by: Gain=1+R2/R3. As we went up in gain we had to back off the Level Control R1 to keep the process at 520 Vdc. We would adjust the gain up, back the level off and watch for the upward creep in voltage. We found things to be quite stable when R2 equaled 3.6 kΩ. This equates to a gain of about 1.75. This Gain Control was not touched again for the rest of the experiment. The Level Control output voltage was about 0.41 Vdc. We can calculate that 0.41 Vdc x 1.75 = 0.71 Vdc sent to the Mach 1. This equates to about 71 sccm delivery of the reactive gas from the Mach 1 or about 14% of its total flow.

After getting the process to be stable in the steep part of the curve we went to the full metal mode using only the Level Control R1. We slowly tuned the process down through transition and into the fully poisoned mode in small increments making sure that the process was stable and there was no voltage creep at any point. We then went back up the curve in the same manner. The results are shown in Figure 4.

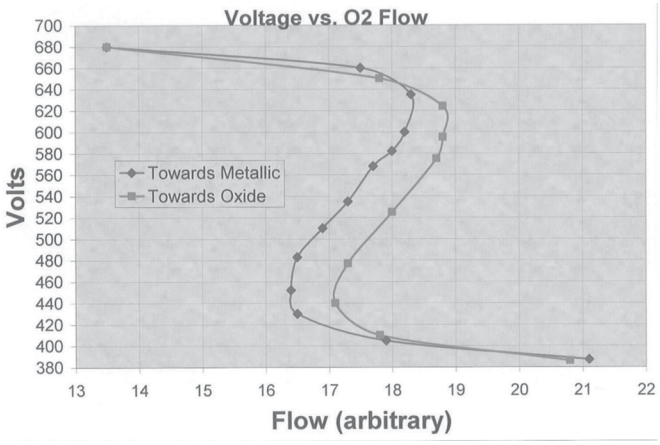

Figure 4: Process Results

We went back to 520 Vdc on the process for stability testing. We wanted to perturb the zone to see how robust and repeatable it is, especially on the transitional part of the curve. We closed a poppet valve on one of the high vacuum pumps. The pressure went up by almost 0.4 mTorr resulting in a voltage decrease to 480 Vdc. The 250 did its job by reducing the pressure to where it was supposed to be and the Mach 1 returned the process to 520 Vdc. We made sure that the process was stable and opened the now closed poppet valve. The pressure went down about 0.3 mTorr and the process voltage went up to 590 Vdc. Again the 250 returned the pressure in the zone to normal and the Mach 1 returned the process voltage to 520 Vdc. In each case, the process was ready to go in less than 5 seconds.

We then let it run there for well over an hour. We saw no signs of voltage creep. Things were stable and life was good! We then turned off the power supply. This effectively gives a 0 V setpoint to the Mach 1 through the signal conditioner. The 250 compensated to bring the pressure back to its setpoint. We turned the Pinnacle output power back on. The voltage was initially about 490 Vdc. The 250 again compensated for the extra reactive gas flow. The Mach 1 brought the process back to 520 Vdc. As arcing occurred, the voltage would collapse

which brought the setpoint to the MFC down accordingly. This helps to keep the target from getting too much oxygen during these events.

We ran samples for rate measurements and reactive film quality at 390 Vdc, 480 Vdc, 540 Vdc and 580 Vdc. The pressure was at 6 mTorr and the DC power was 10 kW. The Pinnacle was in "Power Control" mode. Thickness and optical constant were evaluated by ellipsometry; results are shown in the following table and Figures 5 and 6.

Sputter Voltage	Thickness (nm)
390	286
480	639
540	679
580	760

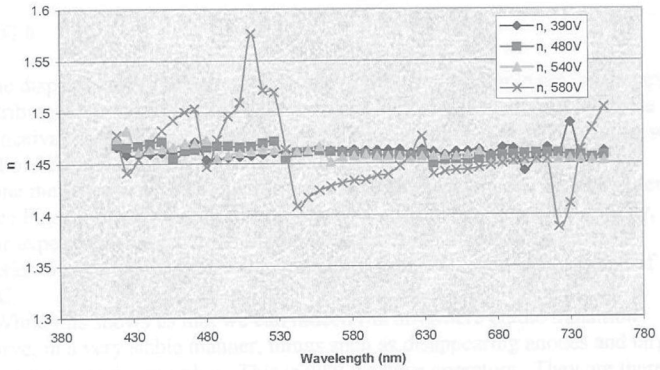

Figure 5: Index of Refraction versus Sputter Voltage

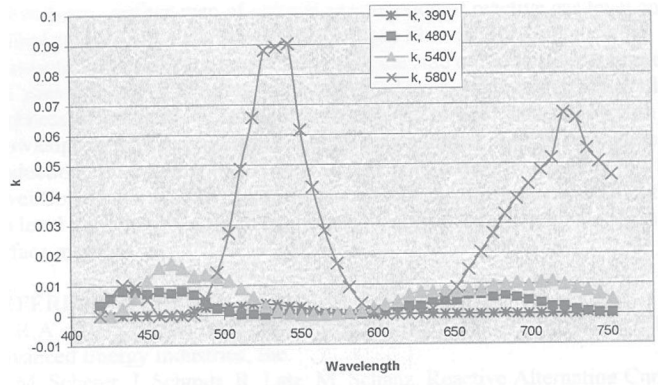

Figure 6: Coefficient of Extinction versus Sputter Voltage

The dispersions of the optical constants for the 580 V sample are, we believe, attributable to the film being slightly sub-stoichiometric. In retrospect we conceivably could have improved this. When re-tuning for each sample, we followed the path from poisoned mode toward metallic mode. If we had gone the other way, we would have had a higher partial pressure of oxygen (see Figure 4), thereby doing a better job of oxidizing the film. Additionally, our experience and results from the literature available indicate that it's easier to keep the film fully stoichiometric using AC sputtering instead of DC. While this shows us that we can indeed run anywhere on the transition curve, in a very stable manner, things such as disappearing anodes and target usage may come into play. This is why we have operators. They are there to keep the machine running and the process in place. A bit of infrequent tweaking on the Level Control R1 ONLY would be needed to keep this process in check.

Without changing the machine configuration and simply by adding the fast MFC and signal conditioner box we are now able to consistently run at a much higher rate with the same film qualities. Since the power supply is running in "Power Control" mode and the voltage is held constant by the fast reaction of the Mach 1, we expect the process will be very stable over the long haul.

Bringing this technique forward, from experiment to production, we would first map out hysteresis curves at several power levels in order to produce a 3-D response surface map of voltage as a function of reactive gas level and applied power. Then we would make sample films at 2 or 3 points in the transition regions of each power level. On the side of the hysteresis curve that goes from metal to oxide, we would produce a response surface of film thickness as a function of sputtering voltage and power. This, along with knowledge of required line speeds, will indicate initial process settings for production. Fine-tuning the process can be done simply by resetting the Level Control R1. Larger changes can be made by adjusting power, but the gas level may also need a corresponding change as indicated by the response surface maps.

REFERENCES

1. R.A. Scholl, Power Systems for Reactive Sputtering of Insulating Films, Advanced Energy Industries, Inc., Advanced Energy White Paper Series, November 2001.

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

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