

# AC Reactive Sputtering with Inverted Cylindrical Magnetrons

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## ABSTRACT

Inverted cylindrical magnetron sputtering is an excellent way to coat materials at high rates onto complex shapes, such as those used in the biomedical, aerospace, and machine tool industries. Many of these applications require ceramics, which can be deposited most effectively with reactive sputtering. In the past several years, pulsed DC power has become a useful means to avoid arcing when reactively sputtering insulating compounds with planar magnetrons. However, the geometry of inverted cylindrical magnetrons creates an acute "hidden anode" problem that makes pulsed DC reactive sputtering ineffective in these devices. Therefore, we have built a dual-cathode inverted cylindrical magnetron that can be driven with mid-frequency power. Unlike dual-cathode mid-frequency sputtering with two planar magnetrons, this cathode has a common magnetic trap over both electrodes and confines the individual plasmas electrostatically.

We have sputtered aluminum nitride and aluminum oxide at deposition rates that are approximately 40% of the aluminum metal rate in each case. The reactivity of aluminum nitride is such that any desired nitrogen partial pressure is accessible using nitrogen flow control and rapid pumping alone. Aluminum oxide, on the other hand, requires feedback control of the oxygen flow to maintain an arbitrary oxygen partial pressure and we have used the target voltage as the control signal. The index and absorbance of films deposited onto glass were calculated from their spectral transmittance and reflectance and agree with reported bulk values. Preliminary data for titanium oxide are presented as well.

## INTRODUCTION

Inverted cylindrical magnetron sputtering cathodes are efficient devices for coating wires, fibers and complex three-dimensional shapes. In this geometry the target is the inside surface of a cylinder and sputtered material arrives simultaneously from all radial directions. [1,2,3,4] In many applications, such as biomedical components and optical elements for example, electrically insulating coatings are often of interest. However, the enclosed geometry of inverted cylindrical magnetrons presents unique challenges when sputtering dielectrics.

It is well known that RF sputtering relies on a relatively small target area compared to the area of the counterelectrode in order to establish a significant negative self-bias on the target and small sheath potential at the anode. [5] Because the geometry of an inverted cylindrical magnetron confines the plasma to a volume in which the target area is often much larger than the anode area, simply applying an RF bias to a conventional cathode will not work. Penfold and Thornton, the original inventors of inverted cylindrical magnetrons, solved this problem with designs in which the cathode consisted of two symmetric elements driven by RF power. [6] However, to our knowledge such a device was never used. [7]

Recently, bipolar pulsed DC reactive sputtering has become an efficient means for depositing dielectric materials with planar magnetrons. However, our calculations show that depositing insulating materials with pulsed DC power can also lead to problems related to the size of the anode. [8] Specifically, when the ratio of cathode current to anode area becomes large enough, significant potentials can develop on the anodes. We have found that because of the limited anode area in inverted cylindrical magnetrons, using them to reactively sputter dielectrics with pulsed DC power is a problem.

This paper describes the operation of an inverted cylindrical magnetron consisting of two co-axial targets in a common magnetic field, as described by Penfold and Thornton. [6] However, we have used reactive sputtering with mid frequency AC power to drive the cathode rather than RF. One of the purposes of this work was to understand process control and stability using this geometry. Therefore, we studied the system behavior and film properties of aluminum nitride, aluminum oxide and titanium oxide deposited from aluminum and titanium targets under various conditions.

## EXPERIMENTAL PROCEDURE

The design of the dual inverted cylindrical magnetron is shown in Figure 1. Two electrically isolated, coaxial cylindrical targets were arranged in a common axial magnetic field. The magnetic field confined the secondary electrons radially and wings that were at the target potential reflected the electrons at the ends of each target to confine them axially. A floating output 40 kHz AC sputtering power supply (a PE 5K

manufactured by Advanced Energy Industries) was used. Each target had an outside diameter of 19.0 cm, a thickness of 2 mm and a height of 9.8 cm.

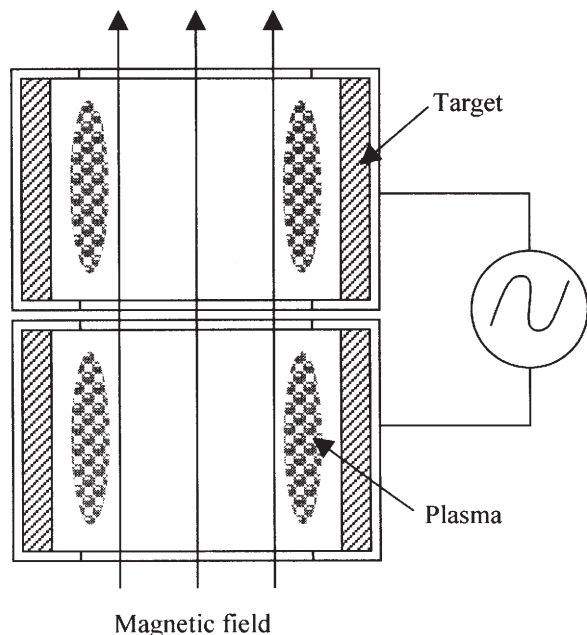


Figure 1. Cross-sectional schematic of the dual inverted cylindrical magnetron cathode used in this work.

This process is obviously similar to mid-frequency sputtering with two planar magnetrons. However, the enclosed geometry leads to a significant amount of gettering on the target surface itself, potentially giving rise to unique process dynamics. Also, in this case a single magnetic trap is associated with both targets, unlike the case for planar magnetrons. Therefore, we would expect the plasma dynamics might be somewhat different.

The two sections were contained in a common housing, which formed the vacuum chamber. The system was a Veeco-Ion Tech Cyclone<sup>R</sup> pumped with a 1600 l/s turbomolecular pump. Pressures of  $3 \times 10^{-7}$  Torr could be reached in 1 hour, and typical base pressures of  $5 \times 10^{-6}$  Torr were achieved in 3 to 5 minutes.

In the case of all three materials, we first measured the target voltage as a function of reactive gas flow using flow control. Our goal was to see if we could eliminate the process hysteresis by using rapid pumping. [9] As we will show, this was possible in the case of aluminum nitride. In order to reactively sputter both aluminum oxide and titanium oxide at high rates,

however, it was necessary to control the reactive gas flow with feedback. We chose to use the target voltage (or current) for this purpose. We did this by comparing the actual target voltage to a voltage setpoint and used the error signal to control the oxygen flow. In this way we could operate at any chosen target voltage, which corresponded to operating at any oxygen partial pressure or level of target poisoning.

In the cases of aluminum nitride and aluminum oxide, the hysteresis was studied and films were made at all four combinations of 1 or 2 kW total sputtering power and 4 or 8 mTorr total sputtering pressure. For titanium oxide, the only operating condition studied in detail was a total pressure of 4 mTorr and power of 2 kW. In all cases the Ar flow was 99 sccm and a pendulum gate valve was used for downstream pressure control. The Ar was always introduced on the pumped side of the targets. The reactive gas could be introduced at either end and the location affected the film properties at various positions, as expected.

Films were made on silicon and glass substrates, which were positioned approximately 5 cm from the target surface with their normals in the radial direction. A cylindrical shutter separated the substrates and targets until the process conditions were established. Small transients were evident in the target voltage as the shutter opened in some cases. Deposition rates were calculated from profilometer thickness measurements and the known run times. The reflectance and transmittance of the films on glass were measured with a UV/Visible spectrophotometer and ellipsometric measurements were made on the aluminum oxide on silicon samples.

## EXPERIMENTAL RESULTS AND DISCUSSION

### Aluminum Nitride

Figure 2 shows the target voltage as a function of nitrogen flow (in flow control) for a total pressure of 8 mTorr and power of 2 kW. There is virtually no hysteresis evident in the flow-voltage curve and any operating voltage could be reproducibly obtained in flow control alone. This behavior was characteristic of aluminum nitride deposited at all conditions. A pressure of 8 mTorr and flow of 99 sccm corresponds to a pumping speed of approximately 160 l/s. This speed is apparently sufficient to eliminate the hysteresis in our aluminum nitride reactive sputtering process.

At 2 kW and 8 mTorr, the transition between absorbing and non-absorbing films occurred between 8 and 9 sccm of nitrogen. At 9 sccm, the measured absorbance (1-R-T) for a 118 nm thick film was less than 1.5% between 400 and 800 nm.

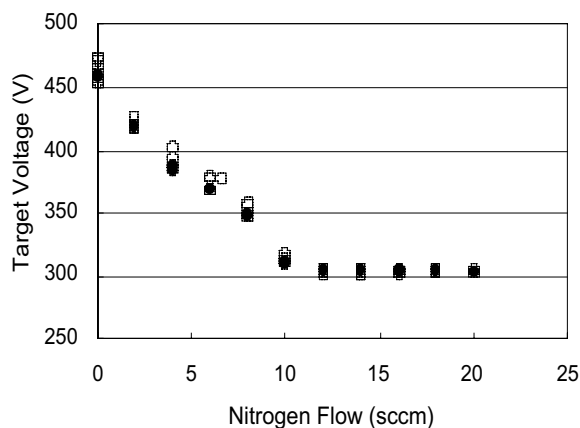


Figure 2. The target voltage as a function of nitrogen flow when reactively sputtering aluminum nitride at 8 mTorr and 2 kW in flow control. The open circles are for increasing flow and the solid circles are for decreasing flow.

Transitions from absorbing to clear films were observed at similar relative voltages for all four powers and pressures studied. At each condition, the minimum flow needed to produce clear films was established. Table I shows the deposition rates of pure Al, as well as the nitrogen flow rate that produced clear films and the deposition rates for those films.

Table I. Deposition rates of pure Al as well as the nitrogen flows and deposition rates for non-absorbing AlN films.

Power (kW)	Pressure (mTorr)	Al Rate (nm/min)	N <sub>2</sub> Flow (sccm)	AlN Rate (nm/min)
1	4	82	8.0	27
1	8	68	7.0	36
2	4	147	12.0	69
2	8	141	9.0	55

The ratio of nitride to metal deposition rates is between 33% and 47%, depending on conditions.

### Aluminum Oxide

In flow control, the target voltage as a function of oxygen flow exhibited the classic hysteretic behavior of reactive sputtering. This is shown in Figure 3, which presents the data for conditions of 1 kW and 4 mTorr. As the flow was increased, we saw a rapid transition from the metallic to the poisoned mode that occurred at a flow of approximately 28 sccm. And as the flow was decreased, the target remained poisoned until the flow was reduced below approximately 10 sccm. However, by using the target current in feedback control to manage the oxygen flow, any target voltage could be reproducibly achieved and maintained.

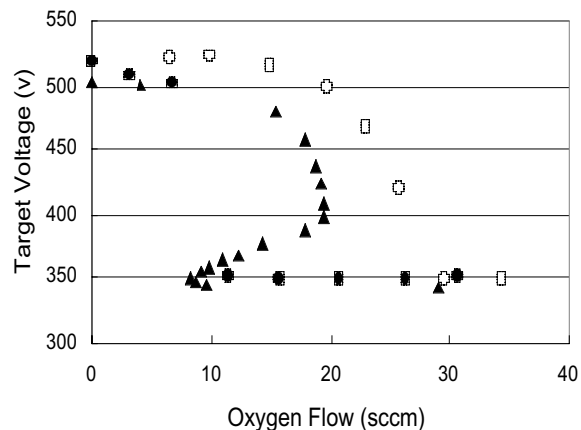


Figure 3. The target voltage as a function of oxygen flow when reactively sputtering aluminum oxide at 4 mTorr and 1 kW. The circles are data taken in flow control, both increasing (open) and decreasing (solid). The triangles are data taken using the target current to control the flow.

Non-absorbing films could be made either in the poisoned mode or by operating approximately at the “nose” of the voltage-flow curve using feedback control (the “high rate” coatings). Ellipsometric measurements were made of high-rate coatings on silicon in order to determine the indices of refraction. Table II shows the deposition rates of aluminum oxide coated in the poisoned mode and for those made in feedback control. Also shown are the measured indices of refraction at 630 nm for the high rate coatings.

Table II. Deposition rates of aluminum oxide made in the poisoned mode and using feedback control as well as the indices of refraction of the high rate coatings. The pure aluminum rates can be found in Table I.

Power (kW)	Pressure (mTorr)	Poisoned (nm/min)	Feedback (nm/min)	Index (630 nm)
1	4	4.8	25	1.63
1	8	5.4	32	1.63
2	4	7.8	38	1.56
2	8	9.0	54	1.49

The coatings made at 1 kW have indices similar to those reported for evaporated Al<sub>2</sub>O<sub>3</sub> [10] and slightly lower than the value of 1.65 reported for reactively sputtered alumina made with ion beam assist. [11] The indices of the films deposited at 2 kW are lower than expected. In the case of the high rate coatings, the oxygen was introduced into the top of the system and some absorption could be seen in films made at the lower (pumped) end of the cathode. This was undoubtedly due to gettering of the oxygen as it flowed from top to bottom.

Although no absorption was evident on the glass substrates, it is possible that at the higher powers there was some absorption in the films on silicon that could have corrupted our index measurements.

The aluminum oxide films made in feedback control had rates between 27% and 38% of the metal rates, which are similar to the values achieved with the aluminum nitride films.

### Titanium Oxide

As Figure 4 shows, flow control produced a complex target voltage vs oxygen flow hysteresis in the case of titanium oxide. These data were taken at a power of 2 kW and pressure of 4 mTorr. As the oxygen flow was increased, at point A the voltage suddenly rose to point B and then fell again to point A. Following that, a further increase in flow produced a small drop in voltage. As the flow was reduced from A to C, the voltage rose slightly until it reached C, at which point it suddenly fell to point D.

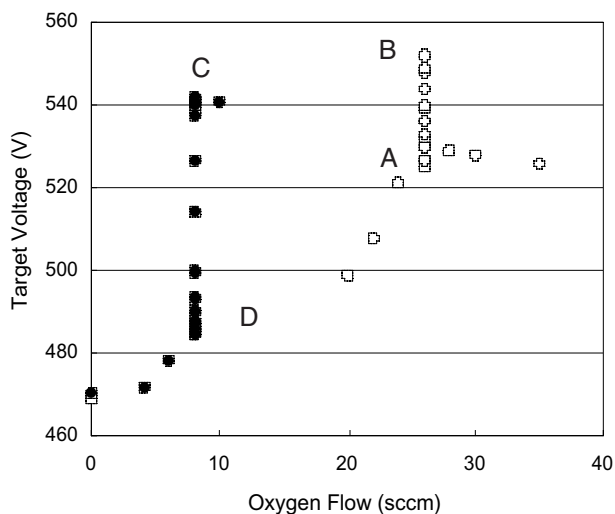


Figure 4. The target voltage as a function of oxygen flow when reactively sputtering titanium oxide at 2 kW and 4 mTorr in flow control. The open circles are for increasing flow and the solid circles are for decreasing flow.

As shown in Figure 5, in feedback control the complex nature of this hysteresis becomes evident. Here we see that the same value of the target voltage occurs at two different flows, unlike the simpler case of aluminum oxide. Point A in Figure 4 corresponds to the vertical slope at an oxygen flow of about 26 sccm in Figure 5. On reaching this point, the target began to poison. The loop in Figure 5 was traversed and the voltage again stabilized at about 535 V, but with the target completely poisoned. The voltage at point B in Figure 4 is that at the top of the loop in Figure 5. Point C in Figure 4 corresponds to the vertical slope at an oxygen flow of about 12 sccm in Figure 5. As the flow was decreased, the voltage first rose until it reached point C, when the loop was again traversed and the

voltage fell to point D in Figure 4. In order to take the data in Figure 5, it was necessary to change the sense of the feedback control depending on whether the target voltage increased or decreased with increasing oxygen flow (at the top of the loop).

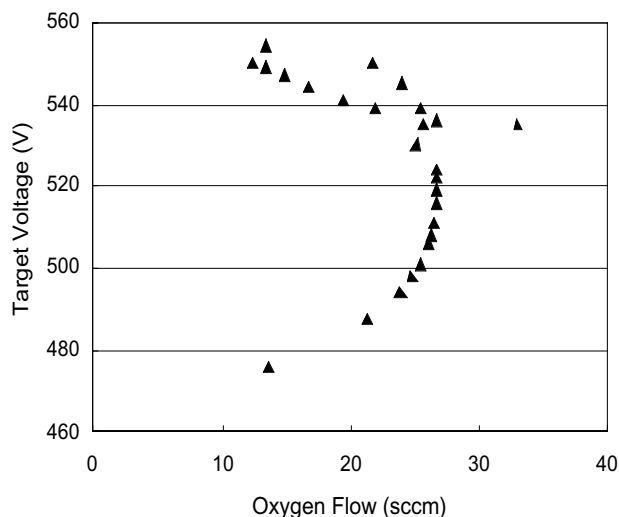


Figure 5. The target voltage as a function of oxygen flow when reactively sputtering titanium oxide at 2 kW and 4 mTorr using feedback control.

Titanium oxide films were made at a power of 2 kW and pressure of 4 mT both in the poisoned mode and in feedback control. Because of the complexity of the hysteresis, the films made using feedback control were started in the poisoned mode. The flow was gradually reduced until the voltage reached the setpoint of 550 V at a flow that averaged about 16 sccm. This point is almost at the peak of the loop in Figure 5. A typical run in feedback control lasted 20 minutes and about 2 minutes were needed to go from the poisoned to the high rate condition. Table III summarizes the deposition rates and compares them to the rate for pure titanium.

Table III. Deposition rate of pure Ti as well as for titanium oxide deposited in the poisoned mode and with feedback control.

Power (kW)	Pressure (mTorr)	Ti Rate (nm/min)	Poisoned (nm/min)	Feedback (nm/min)
2	4	70	7.2	22

All of the titanium oxide films appeared slightly yellow in transmission. The film made in the poisoned mode was 70 nm thick and the absorbance decreased smoothly from 18% at 400 nm to 0% at 800 nm. The film made in feedback control was 440 nm thick and multiple interference fringes made calculation of the absorbance difficult. Reactive sputtering

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can form a number of oxides of titanium and the relative ratio is a function of the oxygen partial pressure. [12] Therefore, we would expect the film composition to depend on the operating point we choose.

## CONCLUSIONS

Reactive sputtering with the dual target inverted cylindrical magnetron used in these experiments is very similar to the behavior of dual planar magnetrons in our experience. The process control issues and operating characteristics are the same and the specific deposition rates are comparable. The enclosed geometry requires careful handling of the reactive gas distribution and higher aspect ratio (length/width) cathodes may present a more difficult challenge.

We were able to deposit aluminum nitride films at high rates using nitrogen flow control alone because of the high pumping speed of the system. High rate aluminum oxide and titanium oxide coatings, on the other hand, required feedback control of the oxygen flow.

These preliminary results suggest that it is possible to deposit insulating films at high rates with inverted cylindrical magnetrons by using conventional reactive sputtering control methods.

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