

Versatile Reactive Sputtering Batch Drum Coater with Auxiliary Plasma



Deposition Sciences, Inc.

Quality Coating Solutions

Versatile Reactive Sputtering Batch Drum Coater with Auxiliary Plasma

M.A. George, H.R. Gray, N. Boling, and E. Krisl, Deposition Sciences Incorporated, Santa Rosa, California

Key Words:

Magnetron Sputtering
Microwave Plasma

Optical Coatings
Multilayer Deposition

ABSTRACT

Reactive sputtering processes have been utilized for 30 years to deposit dielectric thin films of a quality superior to that achieved by conventional evaporation technologies. The reactive sputtering process creates higher energy adatoms and the magnetron plasma releases ions of high energy to the depositing surface. Both of these features of reactive sputtering result in improved film quality. Use of a rotating drum configuration in batch processing lends to a versatile extension of this basic reactive sputtering process. Auxiliary plasmas are readily incorporated, acting on sub-monolayer deposits with every rotation of the drum and interacting with the target plasmas. Microwave driven plasmas are particularly effective in this regard. Further, the activation of the reactive gas permits the coater to operate lower on the reactive sputtering hysteresis while retaining improved stoichiometry, resulting in better thin film thickness control.

We will discuss further development of the MicroDyn® process for several applications of dielectric thin film filters showing preferred operating points on the hysteresis and the resulting coating performance [1]. The versatility of the process is demonstrated by reviewing optical thin film applications of hot mirrors, cold mirrors and band pass filters. We also discuss the uniformity control realized for coating highly curved objects.

All thin films discussed are processed on a batch drum coater equipped with 12.5 cm wide targets and lengths of 37.5 cm or 1 meter, powered in either the DC or AC configuration. The auxiliary plasma is sustained by a planar 10 cm wide by 37.5 cm or 1 meter long microwave plasma applicator. Multi-layer thin films are characterized by transmittance and reflectance, and transmission electron microscopy.

INTRODUCTION

The reactive sputtering drum coater is presented schematically in Figure 1. The chamber is laid out as an octagon with a 1.27 meter maximum diameter. Each face of the octagon can support a process zone. A process zone can be equipped with one of the following of hardware; DC Magnetron, AC Magnetron Pair, Microwave Plasma Applicator or Heater. Mid-frequency pulsed DC power supplies power the DC magnetrons. Pulsed DC power supplies have been shown in previous work to dramatically improve film quality [2-3]. Mid-frequency AC power supplies power the AC magnetrons. AC sputtering eliminates the anode loss sometimes associated with DC sputtering to form dielectrics and is selected when exceptional film quality is required [4]. The coater is equipped with a 1.2 meter diameter drum supported with spokes connected to a hub that engages a rotary vacuum feed through. Substrates are mounted on the drum with the coated surface facing outward. The drum is rotated between 0.75 and 2 revolutions per second depending on the application.

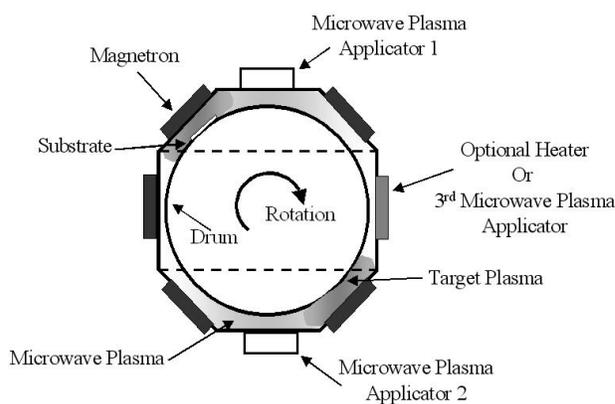


Figure 1. Schematic of the MicroDyn™ reactive sputtering drum coater.

A 2000 liter/sec turbo molecular pump pumps the 37.5 cm height drum coater and the 1meter drum coater is pumped by two 1800 liter/sec turbo molecular pumps. A Meissner trap located on the top and bottom of the chamber is chilled by an external refrigeration system to pump water.

The coater is equipped with two opposed doors for easy access to the drum. Process control is accomplished through a PC based control solution selected for its ability to implement several options for feedback control depending on process requirements. A differentially pumped RGA quadrupole mass spectrometer is utilized as the gas sensor for feedback of reactive gas concentrations and as an in-board internal leak check device. Substrates are pre-cleaned before coating by exposure to the microwave plasma. The plasma pre-clean reduces carbonation and water contamination and prepares the interface for adhesion of the thin film. To begin the reactive sputtering process one or more magnetrons are energized and the substrate receives a flux of material from the target at each pass in front of the target(s). A novel process control scheme developed specifically for the reactive sputtering drum coater is used to stabilize the target plasmas within 500 milliseconds resulting in excellent intra-layer rate stability [5]. Sufficient rotations of the drum are made to build up the necessary thickness of an individual optical layer. It is important to note that the microwave plasmas are active during the reactive sputtering deposition. The microwave plasma envelops the annular space between the drum and the chamber. There is no attempt to baffle or reduce the interactions between the target and microwave plasmas. The benefits of the microwave plasmas for the reactive sputtering process are demonstrated below.

Microwave Plasma Improvement

Microwave driven plasmas efficiently dissociate diatomic molecules like oxygen and nitrogen that are often used in reactive sputtering. The energy imparted to the monatomic species created by the dissociation is transferred to the growing thin film. This dissociation energy of the diatomics are in the range of a few eV and ideal for bond formation of the metal oxides and nitrides of interest for reactive sputtering processes. Other auxiliary plasma sources like ion guns have typical energies much higher, in the range of 50 to 100 eV, which can lead to bond breakage and interface damage. In our application we made use of several features of the microwave plasma. First the microwave plasma envelops the annular space between the drum and the chamber and strongly interacts with the target plasmas.

This interaction tends to stabilize the target plasma and leads to the suppression of arcs [6,7]. The suppression of arcs leads to better film quality by eliminating the particles and surface damage created by the arcing process.

The mechanism by which the microwave improves the reactive sputtering process can be attributed to two phenomena both related to the increased reactivity of the monatomic species generated in the microwave plasma. If we follow the trajectory of the drum around the chamber the substrate is exposed to a flux of monatomic species in the region of the microwave plasma. Monatomic species tend to have high reactivity for both metals and metal oxides. The monatomic species tend to oxidize any metal on the surface and saturate the surface to lower the surface energy creating an excess of reactive species on the substrate surface. As the substrate passes in front of the target it is exposed to both a flux of metal, monatomic reactive gas and diatomic reactive gas. The arriving metal flux can react with the chemisorbed reactive monatomic gas already present on the surface creating a more fully reacted metal. As the substrate continues past the target more metal and reactive material is deposited consuming the chemisorbed monatomic species and leaving the substrate surface with some partially reacted metal.

As the substrate with its partially reacted metal passes by the microwave plasma the flux of highly reactive monatomic species to the surface reacts with the partially reacted metal to complete its reaction. The excess monatomic flux then saturates the surface to repeat the process.

The final mechanism that plays a significant role in the efficacy of the microwave plasma is the impinging flux of Argon ions through the plasma sheath to the substrate surface. These argon ions arrive with 10 to 25 eV of energy that tend to help rearrange surface atoms, lowering the surface energy, without causing further damage.

The effect of the microwave plasma on the reactive sputtering process can be appreciated by comparing the hysteresis curves of reactively sputtering the same material with and without the microwave plasma. In Figure 2 the hysteresis curve of oxygen flow and thin film oxygen consumption is presented. The thin film oxygen consumption is calculated from the known pumping curve of the machine without a target energized and the measured pressure in the chamber during acquisition of the hysteresis data. Here the hysteresis for 8 kW of power applied to a pair of 12.5 x 37.5 cm Si targets powered at 40 kHz is shown. Two microwave plasmas are each supported by a 10 x 37.5 cm microwave wave guide

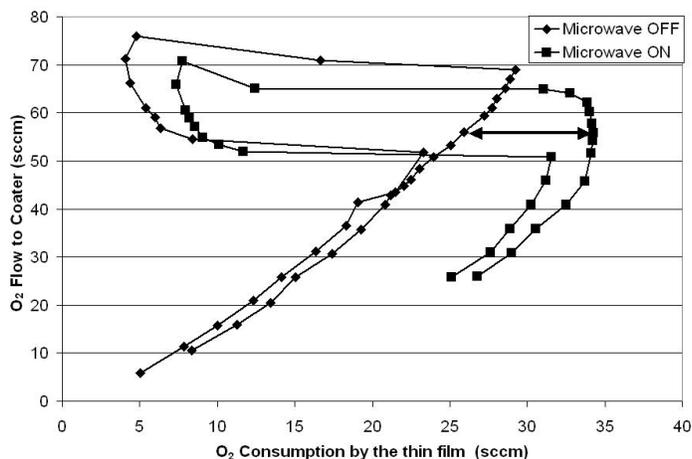


Figure 2. Hysteresis curves for reactive sputtering of Si with and without the microwave plasma. The consumption of oxygen is markedly improved with the microwave plasma.

driven at 2.5 kW and 2.54 GHz. The oxygen flow is increased incrementally and the consumption of oxygen measured.

By comparing the two hysteresis loops it is immediately apparent to the observer that for the same oxygen flow the effect of the microwave plasmas is to increase the oxygen consumed by the thin film. The double-ended arrow in Figure 2 highlights this difference. As the knee of the hysteresis is approached the microwave plasmas tend to permit a more gradual poisoning rather than the sharp discontinuity observed by the hysteresis with the microwave off. This gradual poisoning provides a more forgiving process that permits operation at maximum oxygen consumption over a larger process window.

Deposition Rates

The MicroDyn process has been used to deposit a rather extensive list of dielectric materials. Table 1 contains a partial list of some metal oxides and metal nitrides for which processes have been developed, proven deposition rates and refractive indices for each. The deposition rates reported are on flat substrates secured to the 1.2 meter diameter drum rotating at 1 rps. The deposition rates reported are those for proven processes, higher rates may be possible for each material with further development. All of these processes are used in multi-layer optical coatings and therefore have absorption coefficients sufficient for those applications.

The excellent film quality and morphology of the sputtering process is demonstrated by the transmission electron micrograph of Ta₂O₅ – SiO₂ multilayers shown in Figure 3. Interfacial quality is ascertained by the transmission electron micrograph shown in Figure 4. The higher energy processes developed by the midfrequency pulsed DC or AC power supplies results in exceptional bulk film and interfacial quality. The smooth transition at the interface, in Figure 4, reflects the optimal energy flux delivered by the species present in the microwave and target plasmas.

Table 1. Refractive Index and Deposition Rates for Selected Metal Oxides and Metal Nitrides

Material	Refractive Index @ 550 nm	Deposition Rate (Å/min)
SiO ₂	1.47	600
Ta ₂ O ₅	2.19	310
Nb ₂ O ₅	2.39	750
ZrO ₂	2.06	162
HfO ₂	2.10	200
Al ₂ O ₃	1.66	195
AlN ₃	2.00	100
Si ₃ N ₄	2.05	125

OPTICAL INTERFERENCE FILTER APPLICATION

Multilayer thin films consisting of alternating layers of high and low index materials are readily applied for optical interference applications. For these optical applications the most commonly used low index material commonly is SiO₂ and the high index material consists of one of the other metal oxides or metal nitrides shown in Table 1. Deposition rate stability of the reactive sputtering process permits individual layer thickness to be applied by control of time alone. Rate stability for the process is typically better than ± 1%. The versatile nature of the coater does permit the use of an optical monitor for the most demanding applications.

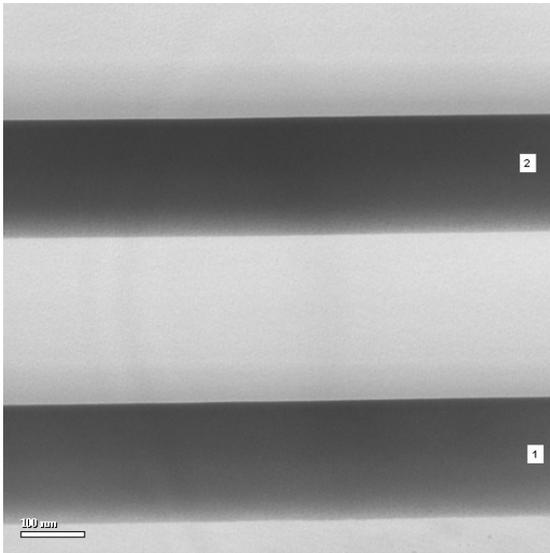


Figure 3. TEM of a multi-layer coating of SiO₂ and Ta₂O₅. Ta₂O₅ is the dark layer.

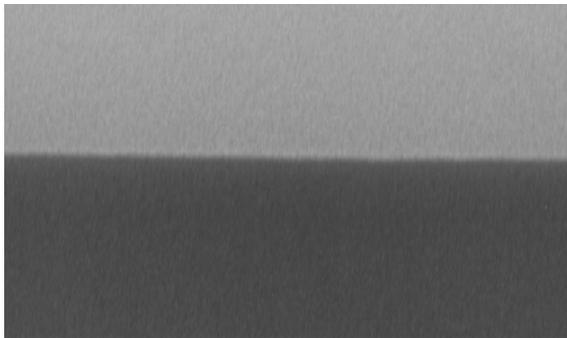


Figure 4. TEM of interfacial region between SiO₂ and Ta₂O₅ layers.

Color Filter

Color filters for RGB color control are utilized in projection display light engines and other digital imaging applications. A 38 layer green filter composed of SiO₂ and Nb₂O₅ is shown in Figure 5. The repeatability of these runs at the 50% cutoff at 560 nm is $\pm 0.5\%$.

One of the advantages of the reactive sputtering drum coater is the throughput capability. For this particular application a 1 meter high drum is used. The usable

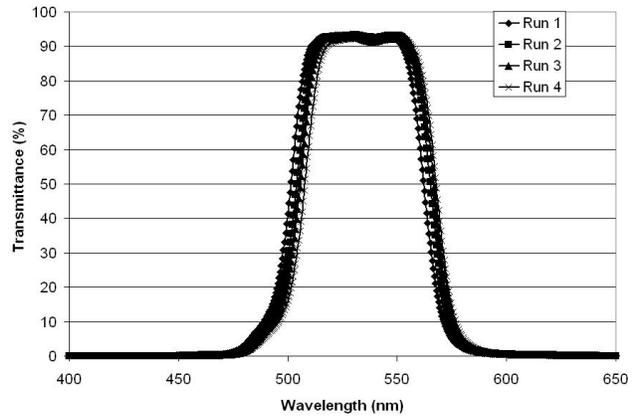


Figure 5. Measured performance of a green filter for 4 consecutive runs of a 38 layer SiO₂ - Nb₂O₅ interference filter.

height of the drum is 0.67 meters for a total coated surface area or more than 2 square meters. Figure 6 shows the uniformity over the drum height for a series of 5 consecutive runs of the green filter shown in Figure 5. The relative standard deviation of measurements at 5 cm intervals along the height of the drum is ± 0.53 .

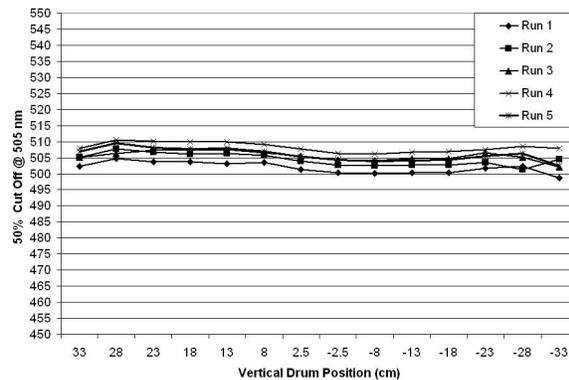


Figure 6. Uniformity of the 50% cut off at 505 nm for the green filter along the height of the drum for 5 consecutive runs.

Hot Mirror

Hot mirror coatings are used for heat management applications to reject heat from light engines and pass visual light. It is important that the coatings withstand the high temperature conditions without failure and with minimal spectral shift. Spectral scans from a 45 layer ZrO₂ - SiO₂ hot mirror is shown in Figure 7. ZrO₂ has been chosen for this particular application because of its

ability to withstand the extreme operating temperatures with almost no spectral shift [8].

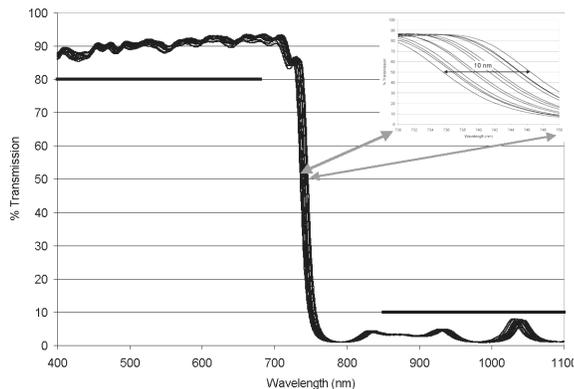


Figure 7. ZrO₂ - SiO₂ hot mirror coating. Inset shows the uniformity over 0.67 meter drum height.

Curved Surface Cold Mirror

One of the distinct advantages of the reactive sputtering process is the higher operating pressure than evaporative processes. The high operating pressures results in scattering of the deposited flux, which provides the ability to coat highly curved surfaces uniformly. One example is the concave surface of a high temperature light engine reflector. A cold mirror is applied that reflects the visual wavelengths and permits the infrared energy to pass through the reflector protecting the components downstream of the light source. Figure 8 shows the spectral performance of a 41 layer Nb₂O₅ - SiO₂ cold mirror deposited on the reflector shown in the inset. Thickness uniformity over this highly curved surface is achieved in part by the scatter of the sputter flux and by a proprietary masking technology. The gradient of the thin film thickness is tailored for both the SiO₂ and the Nb₂O₅ to match the angular spectral distribution of the light source centered in the reflector. The tailored thickness profile compensates for the shift of the thin film's reflectance to shorter wavelengths with angle of incidence.

CONCLUSION

Reactive sputtering with auxiliary microwave plasma results in superior deposition rate stability and thin film properties. The presence of the microwave plasma

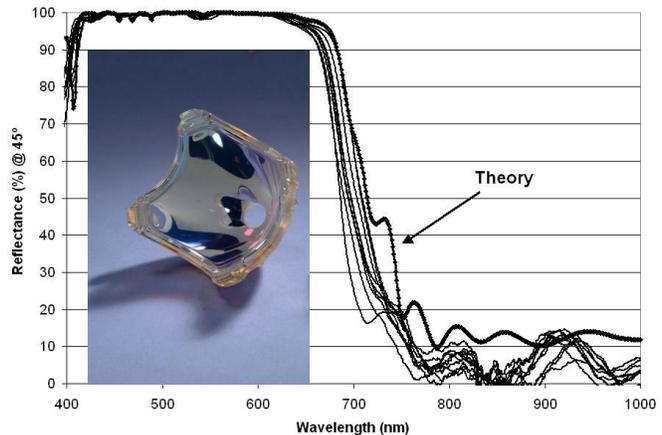


Figure 8. Cold Mirror reflector on glass ceramic curved reflector. Envelope of spectral measurements is along the height of the drum. Reflector surface shown in inset.

provides surface modification and surface energy not present in other reactive sputtering processes. The microwave plasma also permits operation in a more forgiving region of the sputtering hysteresis without sacrificing thin film stoichiometry. The versatile nature of the reactive sputtering drum coater permits the mixing and matching of processes on the faces of the chamber's octagon as needed for different products. Curved surfaces are routinely coated without the complex planetary motion required in lower pressure evaporative processes.

Acknowledgements

The authors would like to thank the hard work of Deposition Sciences technicians and process engineers who made this work possible. We thank Jeff Pool for the process development of the green filter and the hot mirror. We acknowledge the effort of Miles Rains for comparison of the hysteresis loops. We recognize the development by Quinn Gomes, Justin Mosier and Jim Sternbergh for the cold mirror reflector.

REFERENCES

1. N. Boling, B. Woods and P. Morand, "A High Rate Reactive Sputtering Process for Batch, In-Line or Roll Coaters," *38th Annual Technical Conference Proceedings, Society of Vacuum Coaters*, pg. 286, 1995.
2. M. Cornett, M. George, B. Fries, H. Walde, L. Casson and R. Pini, "Optical Emission Studies for the Characterization of Pulsed Magnetron Sputtering Systems," *45th Annual Technical Conference*

Proceedings, Society of Vacuum Coaters, pg. 335, 2002.

3. A. Belkind, A. Freilich, and R. Scholl, "Electrical Dynamics of Pulsed Plasmas", *39th Annual Technical Conference Proceedings, Society of Vacuum Coaters*, pg. 321, 1998.

4. D.A. Glocker, *J. Vac. Sci. Tech. A*, 11(6) pg. 2989, 1993.

5. M.A. George, E.A. Craves, R. Shehab and K. Knox, "*47th Annual Technical Conference Proceedings*", Society of Vacuum Coaters, To Be Published.

6. N. Boling, U.S. Pat. # 6,402,902 "Apparatus and method for a reliable return current path for sputtering processes", June 11, 2002.

7. N. Boling, U.S. Pat. # 5,616,224 "Apparatus for reducing the intensity and frequency of arcs which occur during a sputtering process", April 1, 1997.

8. B.A. Wood and W.H. Howard, U.S. Pat. # 5,923,471, "Optical interference coating capable of withstanding severe temperature environments", July 13, 1999.

© 2004 Society of Vacuum Coaters 505/856-7188 1

47th Annual Technical Conference Proceedings (2004) ISSN 0737-5921

Paper O-13

©2017 Lockheed Martin Corporation. All Rights Reserved.