

Achieving Improved Optical Thin Film Control and Uniformity of Silicon Dioxide by Using Ion Assisted Deposition

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ABSTRACT

We have found that the deposition of critical optical thin film stacks with silicon dioxide from an electron beam gun is severely limited by the stability of the evaporation pattern of the material. We had not obtained satisfactory results in some of the more demanding applications with either solid or granular starting material. The amount of material deposited on a central monitor chip or control crystal in a box coater did not have a reproducible ratio to that received at other positions in the chamber. We solved the problem by evaporating silicon monoxide from a resistance source with simultaneous oxygen Ion Assisted Deposition (IAD) using a Denton CC-102R Cold Cathode Ion Source to produce silicon dioxide of high density and low absorption. The films are sufficiently uniform and repeatable over a full diameter calotte in a 760mm box coater. We describe here our observations of the operating characteristics of the ion source and the deposition scheme which we have found most satisfactory.

INTRODUCTION, THE PROBLEM

Figure 1 illustrates the variable distribution of silicon dioxide evaporant from an electron beam gun as is commonly experienced in physical vapor deposition. The "cloud" may be broad or narrow and not necessarily normal to the general surface of the material if there is "tunnelling". This is explained by the erratic melting/sublimation of silicon dioxide surfaces in both granular and solid disk forms. In our box coaters, we have experienced significant difficulty in achieving satisfactory repeatability of more complex coatings and sometimes even four layer antireflection coatings due to this problem. The properties of silicon dioxide such as its low index, clarity, weathering and abrasion resistance can otherwise be very desirable. We have therefore put forth extensive effort to retain these desirable properties while overcoming the limitations.

THE SOLUTION

We are aware of two solutions to getting reproducible silicon dioxide at high rates by physical vapor deposition in box coaters. One is to evaporate silicon which is in liquid form in an electron beam gun (E-gun) and therefore evaporates more uniformly and repeatably. It must be properly oxidized to silicon

dioxide as it is being deposited. We understand that this can be done by reactive ion plating and some plasma deposition systems. We were not in a position to acquire such a new and specialized chamber, and therefore we chose another approach. The second scheme is to evaporate silicon monoxide and complete its oxidation with ion assistance. This could be retrofit into our existing chambers and processes.

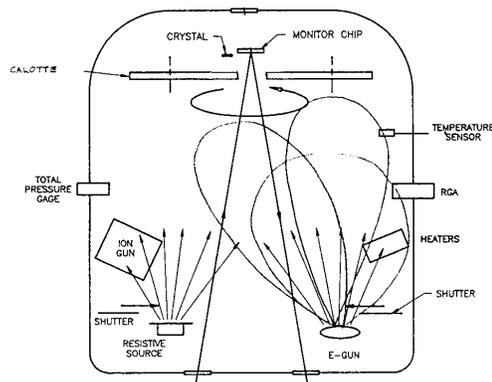


Fig. 1. Typical box coater chamber configuration illustrating the variable distribution directions and amounts found when depositing silicon dioxide from an electron beam gun.

Silicon monoxide sublimates and can be difficult to get satisfactory results using an E-gun, somewhat like silicon dioxide. However, it can be readily evaporated from resistance boats. These boats are typically a maze-like structure to limit the "spitting" or flying hot "ashes" that tend to occur from an open boat with this material. After an "outgassing" process which drives off the "sparks", the vapor deposition seems to be very uniform and stable. We found the R. D. Mathis SO-36 resistance boat to be satisfactory for our work which requires relatively large amounts of material. To evaporate at about 1nm/second at the center of a Balzers BAK760, we apply about 600 amps at 1.7-2.1 volts (depending on how well the electrical contacts have been made).

Our goal is to obtain satisfactory silicon dioxide at a rate of 1nm/second. This cannot normally be achieved by simply evaporating silicon monoxide in an oxygen background, even at an elevated temperature. If the pressure is high, the films will be porous and weak. If the pressure is low, the films will be absorbing and of high index. Energetic ions or neutrals

of oxygen are needed to obtain the rates and properties desired. Kaufman type ion sources can be used to a certain extent at lower rates by combinations of oxygen and argon, but the nature of the Kaufman gun's filament and grids makes its use with oxygen in a high power mode unfavorable. The Denton Vacuum CC-102R ion gun, on the other hand, works best in oxygen and less favorably in argon. We selected this gun for the project and have now operated it for hundreds of hours at maximum power in an industrial environment. We understand that the Commonwealth "End-Hall" type ion sources have closely related characteristics which we believe are discussed in another paper at this conference. We will describe our findings with the use of the Denton ion gun.

EXPERIMENTAL SETUP

The general configuration of the chamber is shown in Fig. 1. The cross section of the ion gun is seen in Fig. 2. The only papers of which we are aware on the gun are by Denton, Mussett, and Lee (1,2,3). They report some of their early work with this gun. A molybdenum anode is surrounded by an aluminum cavity which is in turn surrounded by a ring of permanent magnets which produce an axial magnetic field. The gas is admitted to the gun chamber through small orifices below the anode ring by a gas flow controller. The gun is water cooled. It can be biased to offset the voltage of the whole system with respect to ground, but we have no experience with this; we have only operated at zero bias volts. There is a tungsten neutralizer over the aperture which we typically operate at its maximum current of 20 amps. The neutralizer voltage will range from 12 volts upward with age. The filament will last more than 10 hours and is very easy and inexpensive to replace, so it is not as big a problem as in a Kaufman type source. The power supply has three controls: neutralizer amps, bias volts, and drive amps. There are also digital displays of drive

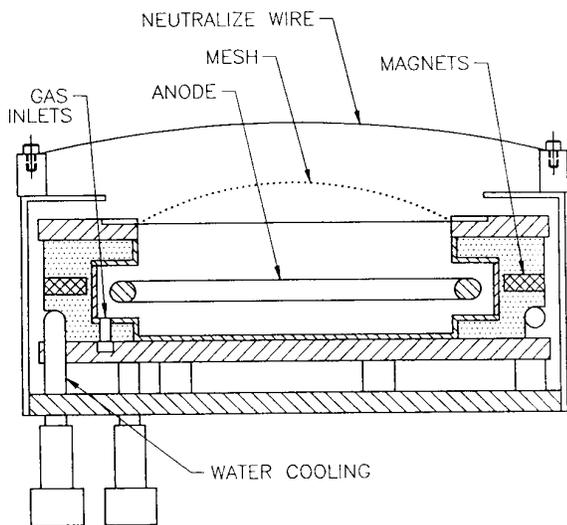


Fig. 2. General cross section of the Denton CC-102R Cold Cathode Ion Source used in this work.

volts and beam amps. We set the neutralizer to maximum current, the bias to zero (but not off), and we control only the drive volts through the drive current and the gas flow.

The ion source is aimed at the calotte for best uniformity which seems to be at about 13cm to the side of the center of rotation and at approximately 30 degrees to the surface normal of the calotte. The aim point is approximately midway between the points in the calotte which are directly over the silicon monoxide and titania sources. These sources have uniformity masks at about 3cm below the calotte and directly over the sources. The titania is evaporated from an E-gun diagonally opposite the silicon monoxide source and used as the high index material.

ION GUN CHARACTERISTICS

The behavior of the ion gun is a strong function of the gas pressure, flow conditions through the cold cathode cavity, the neutralizer emission, and the cleanliness of the gun cavity.

The gas to the gun passes through a gas flow controller before it enters the gun. The flow controller is calibrated for oxygen flow in standard cubic centimeters per minute (SCCM). When another gas is used the calibration would be different. Figure 3 shows the chamber pressure with indicated oxygen and argon flow in one of our Balzers BAK760's with no deposition or IAD in process. The dashed line in Fig. 3 shows how this changes with deposition and IAD with oxygen ions. The silicon monoxide getters the oxygen very readily and effectively increase the pumping speed of the overall system.

We assume that the drive voltage is somewhat linearly related to the actual output beam voltage and similarly that the drive amps are related to the beam amps. The drive voltage is primarily a function of the drive amps applied and the gas flow through the gun. Figure 4 shows this relationship. There is also a significant effect of gun cleanliness by which we mean

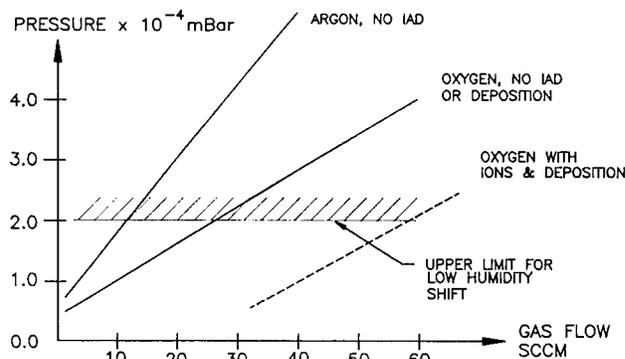


Fig. 3. Chamber pressure versus indicated gas flow in Balzers BAK760 with and without oxygen IAD and silicon monoxide deposition. Upper limit on chamber pressure to produce no humidity shift in coatings is shown from our experience.

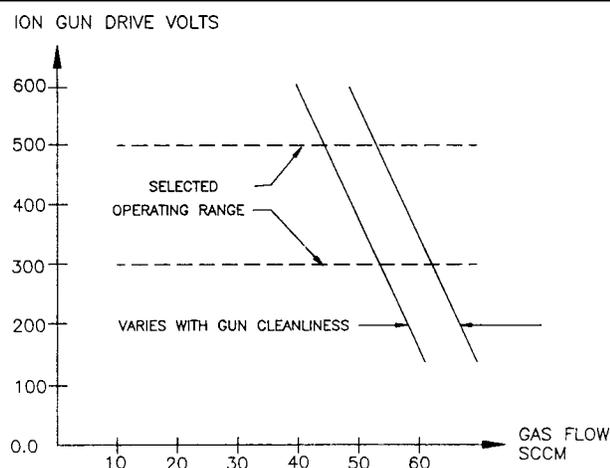


Fig. 4. Operating conditions of drive volts versus gas flow for CC-102R Source as used in our work.

the state of deposits which build up in the cavity with use. We assume that these are oxides of the aluminum and possibly molybdenum and the deposition materials. We have not had a chance to investigate this further. We find it necessary to clean the gun periodically as we will describe below.

When operating the source with argon, we found that the drive voltage decreased with neutralizer amps up to some current and then increased with additional current. When oxygen was used the same general behavior was seen, but the maximum available current of one amp was reached before an inflection was observed. We believe that the lowest drive voltage at a maximum drive current is desirable in our application. We therefore wonder if it would be beneficial to have an extended neutralizer capability to reach the drive voltage minimum with oxygen.

If the source is operated without enough neutralization, we observe sparks on the substrates and fixturing, and arcing indications on the gun power supply. We have seen no ill effects of excess neutralization, so we always apply the maximum available. We seem to observe that, if the pressure in the cavity of the gun drops too low, the drive voltage increases and at some point the gun will arc. This seems disrupt the steady operation of the ion beam conditions, and therefore we avoid this condition if at all possible.

Under certain conditions we have observed an instability in the process where the pressure drops (as indicated by two of the chambers gages) from approximately 2×10^{-4} mbar to 1×10^{-4} in about a second.

This may then cause arcing and the pressure returns to the original level. We hypothesize that under these circumstances the gettering action/reaction of the silicon monoxide coated surfaces in the chamber with the active oxygen ions reduces the chamber pressure such that the ions then produced are even more reactive and quickly gettered. Once this has reduced the pressure in the gun cavity to the point of arcing, the

favorable beam effects are disturbed and the pressure returns to the higher level. We have performed enough checks on the chamber and gas flow controller to give us reasonable confidence that the phenomenon is not due to them but is ion source related. Our explanation is only conjecture at this time, but we do know that we want to avoid this unstable mode of operation. It does not seem to occur when the drive volts are staying below about 400 and sometimes even up to 500.

Our early experiments pointed to the desirability of keeping the drive volts below 400 for minimum absorption in the films and above 300 for maximum densifying effect. We prefer to operate between 300 and 400, but can get reasonable results up to 500 drive volts. This is indicated in Fig. 4. We seldom approach the lower limit of 300 because the gas flow required for that with the typical gun conditions and the resulting chamber pressures would be too high to allow the production of the desired robust films.

The ion gun is water cooled. When operating at the high powers which we use, if the neutralizer is turned off, one can observe the molybdenum anode to be glowing a dull red. We believe that we accidentally operated the gun without water cooling at one time and that this partially demagnetized the magnets of the gun. The effect of the lower magnetic field is to increase the drive voltage under otherwise constant conditions. Since we find this very undesirable, we think that operation without water cooling is to be avoided. The magnets are expensive to replace.

ION GUN CLEANING

As mentioned above, the gun accumulates a buildup of coating within the cavity. This causes the drive volts to increase with otherwise constant conditions. When the volts get out of the selected operating range, it becomes necessary to clean the gun. The deposits observed range from yellow to brown and green, and they can be quite hard in some cases and somewhat chalky in others. We have also noticed that the molybdenum anode ring erodes in the region near the gas inlet holes to the cavity. This is most noticeable at the hole nearest the gas source where the pressure and gas flow are assumed to be the highest. The gas inlet holes in the aluminum base plate of the cavity also show significant erosion which is greatest nearest the gas source. When these effects become too severe, we replace the components.

We use two different techniques for cleaning. One is to disassemble the cavity and abrade away the deposits with Scotchbright and if necessary emery paper. We have been advised to strive for a smooth surface to avoid points for arcing, and therefore we eschew bead blasting. The second approach is to sputter clean the gun with argon before loading the chamber for a coating run. This avoids disassembly and can be effective between chamber cleanings. The sputter cleaning takes about 1/2 hour.

RESULTS

We have used this deposition arrangement and technique extensively for the production of stacks ranging from 40 to 90 layers of silicon dioxide and titania. Figure 5 is an actual spectral curve illustrating the insignificant spectral shift with changing humidity which can be achieved using this IAD system. The sample was immersed in water and placed in the spectrophotometer with just the visible water removed. The spectral transmittance was measured before and after the sample compartment was purged with dry nitrogen for 10 minutes. Without IAD, we typically may see a 15 to 20nm spectral shift. In the case of Fig. 5, the shift is no more than 1nm.

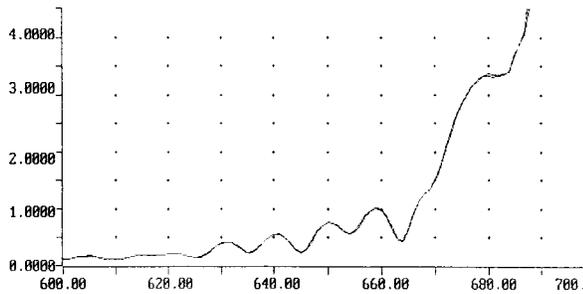


Fig. 5. Optical density versus wavelength of IAD coated sample showing negligible humidity shift.

The deposition conditions of this run were: 225 degrees Celsius, 1nm/second for both materials, 48 SCCM of oxygen through the ion gun, 450-500 drive volts, 1.0 drive amps, 20 amps for the neutralizer, and the chamber pressure was mostly in the range of 1.4 to 2.0×10^{-4} mbar. The silicon monoxide layers getter more than the titania and therefore the higher chamber pressures are associated with the titania. These films also pass the adhesion and severe abrasion tests of MIL-C-675. The resulting index of the silica is about 1.50. We have found that the conditions which give little or no humidity shift with this type of coating are not necessarily compatible with some laser damage requirements. Additional oxygen is required to reduce the laser damage and give an index of about 1.46, but the humidity shift will partially return.

We have generally been able to achieve good and repeatable uniformity over a full chamber diameter calotte by the use of masks. However, the ion gun parameters must be reasonably stable or the uniformity can be seen to have some changes. The ion beam has a distribution which is clearly more concentrated on the axis of the beam. It appears that if an adequate amount of ions reach the less bombarded areas, the excess ions in the central areas are not detrimental. We have had some indication that this is less true when the drive volts are in the high (500) region. The hypothesis is that some ion etching may be occurring at the higher drive volts and thereby the thicknesses of the layers are slightly reduced at the center of the beam impingement area.

CONCLUSIONS

We have found the oxygen ion assisted deposition of silicon monoxide to be a solution to the reproducibility problems of silicon dioxide films. We have been able to deposit films at acceptably high rates in production environments. The Denton CC-102R Cold Cathode Ion Source has been generally satisfactory in this environment. There are several areas of the operational behavior of the gun that we do not understand as well as we would like to at this time, but they have not prohibited us from obtaining practical results.

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