Index of Refraction versus Oxygen Partial Pressure for Tantalum Oxide and Silicon Dioxide Films Produced by Ion Beam Deposition

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INDEX OF REFRACTION VERSUS OXYGEN PARTIAL PRESSURE FROM TANTALUM OXIDE AND SILICON DIOXIDE FILMS PRODUCED BY ION BEAM DEPOSITION

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KEY WORDS

Ion Beam Sputtering (IBS), SiO_x, TaOx, index of refraction

ABSTRACT

Tantalum oxide and silicon oxide films were made using an ion beam sputtering system. It was found that even though these films were deposited from oxide targets, additions of oxygen were necessary to achieve stoichiometry and hence index of refraction. It was observed that the tantalum oxide target changed color from white to gray, indicating that the oxygen was being depleted from the target. The addition of oxygen to the chamber during deposition replenished the target and improved film stoichiometry. The deposition rate decreased with increasing oxygen partial pressure. It was experimentally determined that by varying the oxygen partial pressure and keeping all other variables fixed, the index of refraction of the film changed in a predictable manner. That is, as the oxygen partial pressure was increased, the index decreased rapidly initially and then reached a saturation point where it stayed fixed with oxygen content. With this data a coating process can be set up using the minimum amount of oxygen (thus increasing filament lifetime) to produce a fully stoichiometric film that has a fixed index. This paper will present the details of these observations and results.

INTRODUCTION

Ion beam sputtering offers the technologist a method for making high quality films of practically any material (1) (2). In this work, dielectric films of silicon and tantalum were made by ion beam sputtering. Oxide films can be made using either a metal or an oxide target depending on how much control one needs over

film qualities, such as composition. Magnetron sputtering (MS) is also utilized to make dielectric films (3). It was found that IBS offered improved step coverage than MS. Filament failure greatly impeded the ability to make films by IBS. The more oxygen that was added, the more frequently the filament failed due to oxidation. A plasma bridge neutralizer (PBN), providing electrons for beam neutralization, was added to the gun in place of the neutralizer filaments. The PBN greatly enhanced the efficiency of the process (4). It was found that the index of refraction changed in a predictable manner by changing the oxygen partial pressure. Substrate heating did not affect index. The deposition rate of both materials did vary with oxygen content as well.

EXPERIMENTAL DETAILS

The IBS system that was used for this work has an 8 centimeter DC cathode type ion gun with elliptical grids. The targets are 4 inch diameter by 1/4 inch thick and are metallurgical bonded to a copper backing plate and closely coupled to a water recirculater to draw heat from the target surface. The target is at 45 degrees to the beam the elliptical grids formed a circular beam impinging onto the target. Deposition rate and film thickness are monitored by a quartz crystal that is mounted in close proximity to the beam. Oxygen was delivered directly to the substrate surface and flow was controlled with a leak valve. Chamber pressure was monitored with a hot filament Bayard-Alpert type ion gauge. Substrates were mounted to a water cooled aluminum block with high vacuum adhesive. A boron nitride heater was used for substrate heating. After completion of a coating cycle the substrates were removed from the chamber and measured for thickness with a stylus type profilometer. Index of refraction was

measured at a wavelength of 6328å with an ellipsometer.

Depositions were done at various oxygen partial pressures to determine the trend in index and deposition rate. The total chamber pressure was measured and kept constant for these set of experiments and is described as:

$$P_{(Tot)} = P_{(O2)} + P_{(Ar\ ion\ gun)} + P_{(Ar\ PBN)}$$

Where $P_{(tot)} = total$ chamber pressure

 $P_{(O2)}$ = pressure of oxygen

 $P_{(\text{Ar ion gun})} = \text{pressure of} \quad \text{argon for ion} \\ \text{gun plasma}$

 $P_{(\mbox{\scriptsize Ar PBN})} = \mbox{pressure of argon for plasma} \label{eq:plasma}$ bridge neutralizer

Some substrates were heated to various temperatures to see the effect on index. The chamber was pumped to a base pressure of 1×10^{-7} Torr with a cryo pump. Argon was fed into the back end of the ion gun through a mass flow controller. Oxygen directed to the substrate coated surface. The ion beam parameters were kept constant for each deposition and tabulated in table I. Figure 1 shows the system set up.

Table I Ion Beam Parameters

	TaO _x	SiO_x
beam (I/V)	190mA/1kv	172mA/1kv
accelerator	17mA/241V	7mA/220V
discharge	2.8A/40V	1.7A/39V
cathode	12A/	8.5A
neutralizer	300mA	174mA
current		

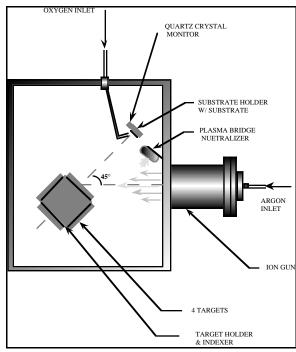


Figure 1 Schematic of Ion Beam Sputtering (IBS) System

RESULTS AND DISCUSSION

Figure 2 shows the index of refraction versus oxygen partial pressure for SiO_x. Figure 3 shows index for TaO_x versus oxygen partial pressure. Figure 4 shows the silicon deposition rate as a function of oxygen partial pressure. Figure 5 shows the deposition rate of tantalum oxide as a function of oxygen partial pressure. As the oxygen partial pressure is increased the index of refraction of SiO_x and TaO_x decreases in a manner shown in Figures 2 and 3. Heating of the substrate showed little effect on index although visual observation revealed that some film crystallization occurred. The deposition rate decreased with increased oxygen partial pressure with both materials as shown in Figures 4 and 5. The plasma bridge neutralizer (PBN) improved the performance of the system by increasing filament lifetime. No improvement in index was observed with the addition of the PBN.

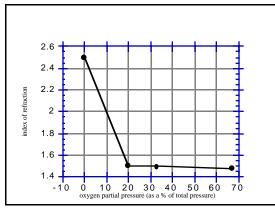


Figure 2 Index of refraction of SiOx vs. oxygen partial pressure. Total pressure is 1.5x10⁻⁴ Torr.

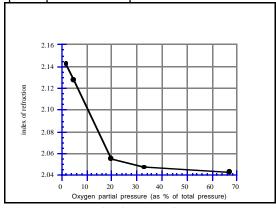


Figure 3 Index of refraction of TaO_x vs. oxygen partial pressure. Total pressure is 1.5×10^{-4} Torr.

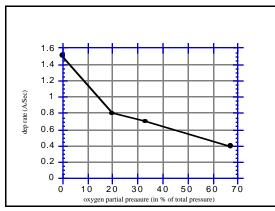


Figure 4 Deposition rate of SiOx vs. oxygen partial pressure. Total pressure is 1.5×10^{-4} Torr.

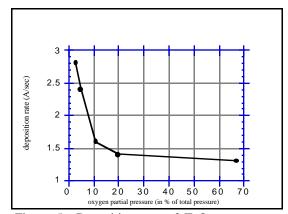


Figure 5 Deposition rate of TaO_x vs. oxygen partial pressure. Total pressure is $1.5x10^{-4}$ Torr.

CONCLUSION

It was found that with ion beam deposition, as the oxygen partial pressure is increased, the dielectric films of tantalum and silicon become more stoichiometric and therefore the index of refraction approached that of bulk material. Substrate heating had little effect on index for both materials. As the oxygen partial pressure increases, the deposition rate decreases as shown in the Figures 4 and 5. Future work will involve chemical characterization diffraction and electron photon spectroscopy), making dielectric wave guides and testing for optical loss. The plasma bridge neutralizer greatly improved the performance of the ion gun.

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REFERENCES

- M. Varasi, C. Misiano, L. Lasaponara, "Deposition Of Optical Thin Films By Ion Beam Sputtering. Thin Solid Films, 117 (1984), 163-172.
- D. Wei, "Ion Beam Interference Coating For Ultralow Optical Loss" Applied Optics, Vol. 28, No. 14, (15 July 1989).
- 3. D. Wang, Y. Nagahata, M. Masuda, Y. Hayashi,

"Effect Of Nonstoichiometry Upon Optical Properties Of Radio Frequency Sputtered AlN Thin Films Formed at Various Sputtering Pressures." J. Vac. Sci. Technol, A 14 (6), Nov/Dec1996.

4. Ibid., 1.

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