Sputtering Deposition of Metal and Dielectric Films

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Motivation

Recently, the SNF bought two new physical vapor deposition (PVD) systems from Intlvac. One system uses electron beam and thermal evaporation to deposit metal and dielectric films, and the other system uses sputtering for the same purpose. These systems have the SNF Coral names of intlvac_evap and intlvac_sputter, respectively. These systems were bought primarily to reduce usage stress on the Innotec and Metallica deposition systems. Additionally, these systems offer more versatility. Reactive deposition with a process gas (either O_2 or N_2) can yield a variety of oxides and nitrides. An integrated ion gun can either clean the substrate before deposition, or aid the deposition through IBAD processes. Figure 1 shows an image of the system.

Figure 1: The Intlvac vacuum deposition system, applicable for both sputtering and evaporation

The sputtering system has AC and RF power sources that can extend the range of materials deposition to dielectrics as well as metals. The substrate itself can be heated or biased in order to modulate the deposition parameters. In this project, three materials were deposited and characterized in order to test the capabilities of the sputtering system.

These materials were titanium, silicon oxide, and tungsten. Titanium was co-sputtered with two AC guns whose power was varied from 100 W to 2000 W. Silicon dioxide was co-sputtered with two AC guns while flowing a mixture of Ar and O_2 into the chamber. Additionally, the ion source was used to investigate any structural change in the deposited film. Tungsten was sputtered with an RF gun. In this way, several features of the sputtering system could be examined: AC deposition of metals and dielectrics, RF deposition, reactive deposition, and ion beam assisted deposition.

Experimental – Titanium

The first experiment using the sputtering system was to deposit titanium films so as to set a baseline point for the reactive sputtering of titanium nitride and titanium oxide films. The sputtering conditions are the following: the two AC sources are active and loaded with titanium targets, the argon flow rate was 45 sccm such that the chamber pressure was maintained at 6 mTorr during the sputtering process, the wafer carousel was rotated at a speed of 100 rpm, and the AC power was varied from 100 W to 2000 W. The deposition was performed on 4-inch silicon wafer substrates for a time of 10 minutes.

After sputtering, both the resistivity and step height were measured so as to establish a deposition rate of the material. The resistivity was measured by a 49-point map using the Prometrix system. The step height was measured using the Alphastep profiling tool. Since the Alphastep tool requires a section of bare silicon wafer, the substrate was selectively etched using an HF:H_2O wet etchant and a baked Shipley 3612 photoresist wet etch mask. The photoresist was dropped at various points in the wafer such that the step height could be profiled at the top, bottom, center, left, and right regions of the wafer. Table I shows the average resistivities and uniformity percentages of the sputtered titanium films, and Table II shows the measured thicknesses from the step height profiling measurement.

Table I: Resistivity Data on Sputtered Titanium Films

Power (W)	Average Resistivity (Ω/sq)	Uniformity (%)
100	20.118	10.802
250	7.103	9.120
500	3.186	7.895
750	2.123	7.810
1000	1.585	7.789
2000	0.8914	7.806

Table II: Step Height Data on Sputtered Titanium Films (in Angstroms)

Power (W)	Top	Center	Bottom	Left	Right
100	297	446	337	337	393
250	943	959	978	983	930
500	1746	1983	2110	2027	1860
750	2799	3271	3375	3193	2357
1000	3626	4142	4258	3912	3893
2000	7282	7533	7555	7128	7050

The data from the step heights can be used to calculate the deposition rates. These rates are expressed in Figure 2:

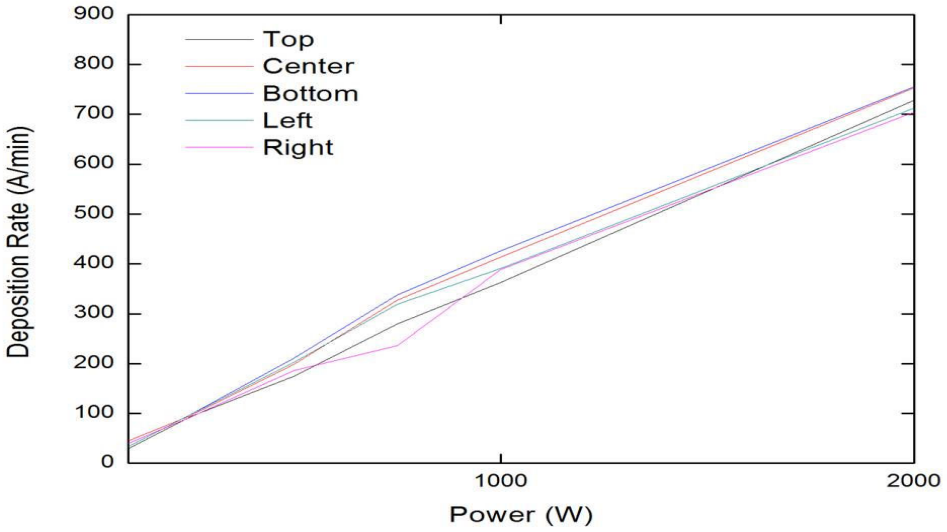

Figure 2: Titanium deposition rates on a 4-inch silicon wafer

Experimental – SiO_2

The second experiment using intlvac_sputter was to reactively deposit silicon dioxide films so as to examine the capabilities of the tool to do reactive sputtering of dielectric materials. The sputtering conditions are the following: the two AC sources are active and loaded with silicon, the argon flow rate was 33.8 sccm, the wafer carousel was rotated at a speed of 100 rpm, and the AC power was set at 550 W. Due to a problem with the cooling water flow rate, this was the maximum available AC power. The deposition was performed on 4-inch silicon wafer substrates for a time of 20 minutes.

Before the actual deposition, the O_2 flow rate was varied in order to investigate target contamination, which has been an effect studied within reactive sputtering of oxides [1]. As the O_2 flow rate was varied, the argon flow rate was kept constant at a value of 33.8 sccm and the sputtering guns were maintained at a power of 550 W. During this variation, the voltage of the sputtering guns was viewed. A transition from a high voltage to a low voltage signifies a transition of the target from metallic mode into poisoned mode, in which the target is covered with a layer of dielectric and sputters material at a reduced rate. The results of this experiment are shown in Figure 3, and show that the transition occurs when the O_2 flow rate is roughly 11 sccm.

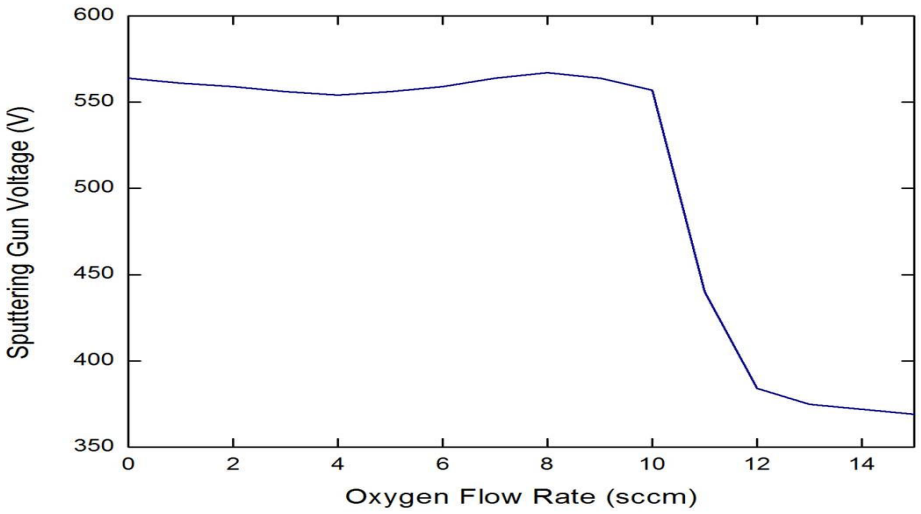

Figure 3: Sputtering gun voltage as a function of the O_2 flow rate

Films were deposited using three different values of the O_2 flow rate: 6 sccm, 10 sccm, and 12 sccm. 10 and 12 sccm were chosen as two points on the edge of the poisoned/metallic target transition so as to confirm target poisoning. 6 sccm was chosen as a point that would be far enough away from the transition so that unintentional target poisoning would not occur. Figure 4 shows an example of the XPS spectra obtained from all three samples. The oxygen 1s, silicon 1s, and silicon 2p peaks are all dominant, thus showing that the material is of the form Si_xO_y. Table III shows the elemental composition of the material from the XPS data for 6 sccm and 10 sccm O_2 flow rates. 12 sccm was not analyzed due to the target being in poisoned mode at that flow rate.

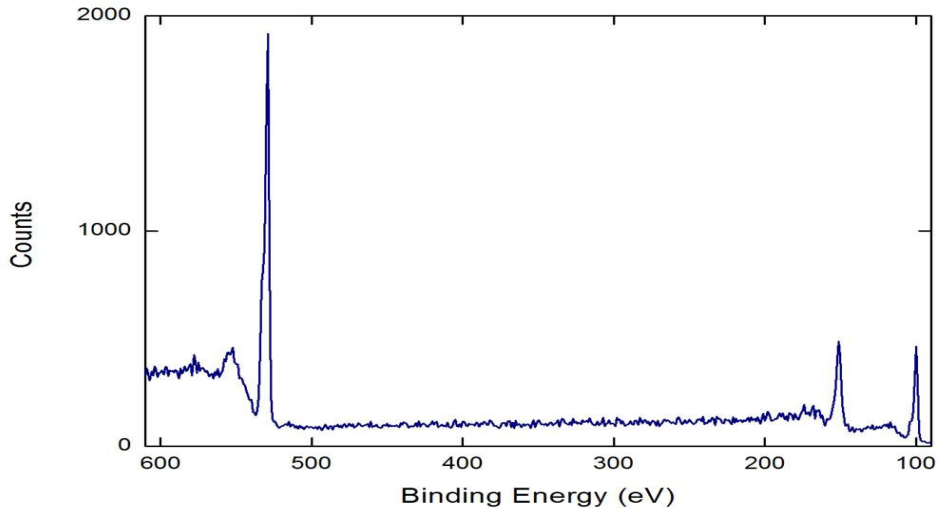

Figure 4: XPS spectrum of sputtered SiO_2

Table III: XPS Compositions of Sputtered Material

O_2 Flow Rate (sccm)	% Si	% O_2
6	45	55
10	36	64

These results show that the O_2 flow rate of 6 sccm does not incorporate enough oxygen within the sputtered silicon in order to make compositionally correct SiO_2. This assessment was confirmed as the Woollam ellipsometer could not fit the refractive index parameters for the sample with an O_2 flow rate of 6 sccm. However, the Woollam

ellipsometer could fit the refractive index parameters for SiO_2 with the flow rates set at 10 and 12 sccm. Table IV shows the extracted thickness parameters from the Woollam ellipsometer for these flow rates.

Table IV: Thickness Data of SiO_2

Flow Rate (sccm)	Minimum Thickness (A)	Maximum Thickness (A)	Minimum Deposition Rate (A/min)	Maximum Deposition Rate (A/min)	Uniformity (%)
10	6047	8096	302.3	404.8	8.49
12	3896	5267	194.8	263.4	8.64

The reduction in thickness as the flow rate is increased confirms the target poisoning mechanism which occurs at 11 sccm.

In addition to the reactive sputtering deposition, the ion beam was investigated for SiO_2 deposition. The motivation for this is that the ion beam will improve the quality of the deposited SiO_2 film [2]. SiO_2 was sputtered under the 10 sccm flow rate, except with the ion beam turned on at a value of 120 V with a 0.5 A discharge current and a 0.6 A emission current. In order to investigate the film quality, both samples (with and without the ion beam) were wet etched using a solution of 20 mL 20:1 buffered oxide etch and 400 mL water. Every 30 seconds, the thicknesses of each sample were checked using the Woollam ellipsometer. The results are shown in Figure 5:

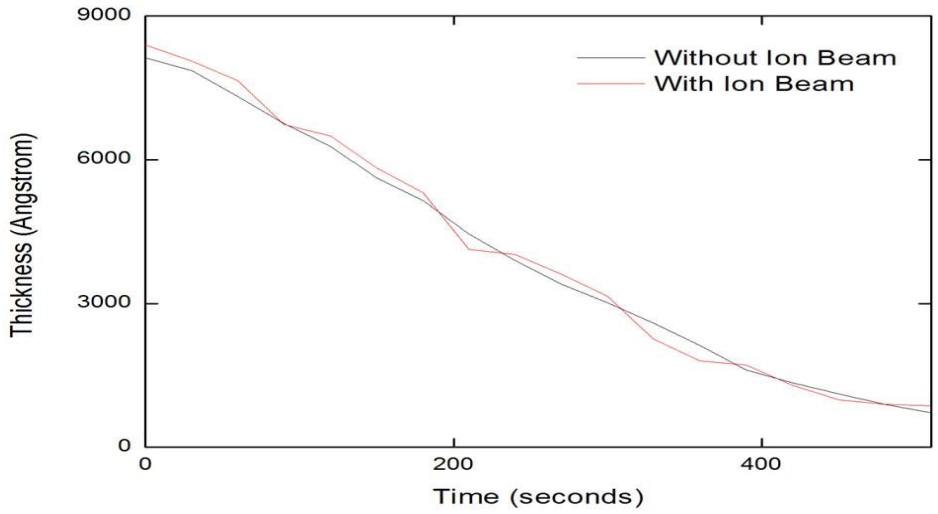

Figure 5: Etching over time of the sputtered SiO_2 films

The film quality appears to be unchanged even though the ion beam was used to sputter the material. Furthermore, the etching rate is much greater than the comparative etching rates of deposited silicon oxides by various methods [3]. This leads to the conclusion that the sputtered oxide film is less dense and of lower quality than deposition by other methods. Most likely, this material is not suitable for high-quality electronics applications and is more suited for structural and sacrificial layers within MEMS processing.

Experimental – W

The final experiment of this project was to use the RF power supply to deposit tungsten films. Although this power supply can go up to 600 W, only 150 W was possible within the framework of this experiment due to limited cooling water flow to the sputtering gun. The sputtering conditions are the following: the RF source is active and loaded with a tungsten target, the argon flow rate was 45 sccm such that the chamber pressure was maintained at 6 mTorr during the sputtering process, the wafer carousel was rotated at a speed of 100 rpm, and the RF power was 150 W. The deposition was performed on 4-inch silicon wafer substrates for a time of 20 minutes.

After sputtering, both the resistivity and step height were measured so as to establish a deposition rate of the material. The resistivity was measured by a 49-point map using the Prometrix system. The step height was measured using the Alphastep profiling tool. Since the Alphastep tool requires a section of bare silicon wafer, the substrate was selectively etched using an H_2O_2 wet etchant and a baked Shipley 3612 photoresist wet etch mask. The photoresist was dropped at various points in the wafer such that the step height could be profiled at the top, bottom, center, left, and right regions of the wafer. The post-etched wafers were then dipped in HF solution so as to remove any native oxide. Table V shows the results of this experiment:

Table V: Experimental Data for Tungsten Sputtering

Average Resistivity (Ω/sq)	Uniformity (%)	Average Deposition Rate (Angstrom/min)
1.799	8.847	44

Future Work

The goal of this project was to understand and develop the Intlvac sputtering tool for materials such that all of the features were explored. This was achieved in that AC and RF power was run, reactive sputtering was tested, and the ion gun was invoked as well. Additionally, the materials developed (titanium, silicon dioxide, tungsten) have benefit within the SNF user community as layers that can be deposited upon substrates and structures at low temperatures, thus making this tool ideal for processes with low thermal budgets.

The broader vision of this project is to eventually integrate both the Intlvac sputtering and evaporation tools to serve as complements to the Innotec and Metallica deposition machines with more functionality. Several materials can be integrated within the systems, and with reactive deposition, oxide and nitride materials can also be deposited. Over the course of time, these deposition tools can eventually be fully integrated within the SNF.

[1] I. Safi, "Recent aspects concerning DC reactive magnetron sputtering of thin films: a review," *Surface and Coatings Technology*, vol. 127, pp. 203-219, 2000.

[2] S. J. Rho, S. M. Jeong, H. K. Baik, and K. M. Song, "The structural, optical, and secondary electron emission properties of MgO and Mg-O-Cs thin films prepared by ion beam assisted deposition," *Thin Solid Films*, vol. 355-356, pp. 55-59, 1999.

[3] K. R. Williams, K. Gupta, and M. Wasilik, "Etch rates for micromachining processing – part II," *Journal of Microelectromechanical Systems*, vol. 12 , no. 6, 2003.