ROCHESTER INSTITUTE OF TECHNOLOGY MICROELECTRONIC ENGINEERING

# Physical Vapor Deposition – Evaporation and Sputtering

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11-26-2013 PVD\_Recipes.ppt/

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### **EVAPORATION DATA**

Material Formula Melt pt.

Temp °C @ Vapor Pressure °C 1E-8	1E-6
-------------------------------------	------

Aluminum	Al	660	677	812
Alumina	Al2O3	2045	1045	1210
Antimony	Sb	630	279	345
Arsenic	As	814	107	152
Beryllium	Be	1278	710	878
Boron	B	2100	1278	1548
Cadmium	Cd	321	64	120
<b>Cadmium Sulfide</b>	CdS	1750		
Chromium	Cr	1890	837	977
Cobalt	Со	1495	850	990
Gallium	Ga	30	619	742
Germanium	Ge	937	812	957

MRC Co., "Evaporation and Sputtering Data Book," Orangeburg, NY http://www.epimbe.com/pages/vp

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### **EVAPORATION DATA**

Material Formula Melt pt.

Temp °C @ Vapor Pressure

	$^{\circ}\mathbf{C}$	<b>1E-8</b>	1E-6	1E-4
Au	1062	807	947	1132
HfO2	2812			2500
Ni	1453	927	<b>987</b>	1262
Pd	1550	842	<b>992</b>	1192
Pt	1769	1292	1492	1747
Se	217	<b>89</b>	125	170
Si	1410	<b>992</b>	1147	1337
SiO2	1800			1025
Si3N4				800
Ag	961	574	617	684
Ta	2966	1960	2240	2590
Ti	1668	1067	1235	1453
$\mathbf{W}$	3410	2117	2407	2757
Zr	1852	1477	1702	1987
	Au HfO2 Ni Pd Pt Se Si SiO2 Si3N4 Ag Ta Ti W Zr	Au       1062         HfO2       2812         Ni       1453         Pd       1550         Pt       1769         Se       217         Si       1410         SiO2       1800         Si3N4		

MRC Co., "Evaporation and Sputtering Data Book," Orangeburg, NY

http://www.epimbe.com/pages/vp

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## **EVAPORATION TECHNIQUES**

**Aluminum** - evaporate copper with tungsten wire basket. One pellet at 20 cm gives about 3000 A.

**Copper** - evaporate copper with tungsten wire basket. The basket needs to be crushed a little so the openings are small and the copper does not fall out of the basket once it is melted. One pellet at 20 cm gives about 3000 A. Dimpled Tungsten boats work great.

**Chromium** – use special Chromium coated tungsten wire filaments. Current through the filament heats the Cr which sublimes.

**Gold** - gold or gold/germanium can easily be evaporated from a basket with tightly spaced loops. The basket needs to be crushed a little so the openings are small and the gold does not fall out of the basket once it is melted. Dimpled Tantalum boats work great. Moly boats are good because gold does not wet the Moly thus less gold is lost.



### **CHROME**

Deposit chrome by evaporation (actually sublimation) from special chrome coated tungsten rods. Using the CVC evaporator. Heat rods to red hot by setting filament voltage to 190 on the dial. Then open the shutter for the desired time calculated from rate of 35 Å/sec. (at a distance of 40 cm from source to substrate)



R.D.Mathis P.O. Box 92916 Long Beach, CA 90809-2916 www.rdmathis.com

Part No. ?? Cost \$250/50 qty

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### **DEPOSITION OF SILICON MONOXIDE (SiO)**

Evaporate SiO with Ta boat and cover with hole. The material sublimes and a film will be deposited. It looks like glass and can be measured on the ellipsometer. The ellipsometer gave an index of refraction of 1.88

Using the CVC evaporator X mg at 40 cm gives about 300 Å. Set to 250 on the dial.

R.D.Mathis P.O. Box 92916 Long Beach, CA 90809-2916 <u>www.rdmathis.com</u>

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Part No. Cost



### **PE4400 SPUTTER / SPUTTER ETCH TOOL**



#### **PVD Recipes PE4400 – AL THICKNESS NON UNIFORMITY** FileName: C:\4P\Factory.prj\Al THK.rcp\0105I027.RsM CDE ResMap RunTitle CDE Demo otID, WaferID NyLot NyWafer 8728 8693 08:02 01/05/10 unDate 8848 8786 Factory Al THK ecip Name. per[Engr[Equp]: CDE[Customer [ResMap] 9896 9595 9680 9939 SinglePrbCnfg afer No. 10.4K 10.4K 150 Flat 'aferDia 10.81 10.61 10.4 10.6K10.6K10.8K 12.0 FollowMajorFlat dgeExclusn 10.9K 10.9K robePoints: 61 #Good: 61 Avg 11.169K Ohms/sq 11.3K11.4K11.4K11.4K11.5K 11.4 11.4 11.4 11.6 11.2K tdDev 971.858 8.701% 35qma=26.104% in 8693.4 Max 12.14K Range 3448.2 11.7K Hx-Mn)/(Mx+Mn) 16.55% (-)/2Av 15.44% min:22.17% Lmax:8.70% (-)/Av 30.87% 11.9 11.9 11.5 11.5 11.8K 11.9K11.9K11.9K11.9K `11.9K radients: R/2=5.420% ~R=7.823% erit: 10.9 50% 2.02 25.0 12.1K12.1K12.1K sns 9.584 IdvMx 0.455 VsnsMx 4.99m 12.0K 12.11 12.11 12.1K ataRejectSigma: 3.0 12.0K 12.0K 12.0K 12.0K .... CDE ResMap RunTitle FileName: C:\4P\Factory.pri\A1 THK.rcp\0105I027.RsM 11.5K 11.8 CDE Demo 11.1 LotID, WaferID MyLot MyWafer 11.0K 08:02 01/05/10 RunDate Factory Al THK Recip Name Oper[Engr[Equp]: CDE|Customer [ResMap] Ave = 11.17KSinglePrbCnfg Wafer No. WaferDia 150 Flat 12.0 FollowMajorFlat EdgeExclusn Min = 8.69KProbeFoints: 61 #Good: 61 w Avg 11.169K Ohms/sq StdDev 971.858 8.701% 3Sqma=26.104% Max = 12.1KMin 8693.4 Max 12.14K Range 3448.2 (Mx-Mn)/(Mx+Mn) 16.55% (-)/2Av 15.44% Lmin:22.17% Lmax:8.70% (-)/Av 30.87% Gradients: R/2=5.420% ~R=7.823% Non Uniformity = 16.55%Merit: 10.9 50% 2.02 25.0 Rsns 9.584 IdvMx 0.455 VsnsMx 4.99m DataRejectSigma: 3.0 #data=61 Rs Spacing = 1/3 Sigma 10.845K 10.521K 11.817K **Rochester Institute of Technology** 11.493K 10.1978 0.16503 9873.45 Microelectronic Engineering 9549.50 9225.55 8901.60 © November 26, 2013 Dr. Lynn Fuller Page 10

### **PE4400 SPUTTER ETCH RATE**

A	B	C	D	E	F	G	Н		J
	Original	Post Etch			Original	Post Etch		Original	Post Etch
1	1992	1506		Average	2241.279	1685.459		2242.28	1685.8
2	2046	1543		Std. Dev	115.8784	86.18035		116.699	86.524
3	2059	1545		Min	1981	1500		1981.3	1500
4	2030	1518		Max	2414	1815		2417.3	1815
5	1981	1500		Range	433	315		436.05	315
6	2111	1597							
7	2155	1624		Etch Rate	18.52732	A/min		18.54933	
8	2168	1629							1

The sputter etch rate was calculated from measured aluminum thickness before and after sputter etch. Measurements were made using 4point probe thickness technique on the CDE resistivity mapper. The sputter etch rate of aluminum was 18 Å per minute.

Power = 500 watts Pressure = 5 mTorrFlow = 20 sccmTable Rotation = Yes



~18Å/min

### CHA FLASH EVAPORATOR



### FLASH EVAPORATOR THICKNESS UNIFORMITY



### *CVC601*



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### **CVC601 THICKNESS UNIFORMITY**



### MEASUREMENT OF METAL THICKNESS USING FOUR POINT PROBE

The existing four point probe with a more sensitive voltmeter (20 mV full scale) can be used to measure the sheet resistance of a metal film. The film thickness (t) equals the bulk resistivity divided by the sheet resistance (Rhos). The bulk resistivity from tables of materials properties can be used as a starting value but thin films often have resistivities a high as two times the value of the published bulk resistivity.

Example: A sputtered Aluminum film was measured with the four point probe. The current and voltage were found to be I=102.2 mA, V=1.296 mV. Using the published bulk resistivity of 2.65E-6 ohm-cm find t.

Rhos = 4.532 V/I = 0.0572 ohms/sq

t = Rho/Rhos = 2.65E-6 ohm-cm / 0.0572 ohm = 4633 Å

The Alpha step measured thickness was 7800 Å so the corrected value of resistivity that should be used for sputtered aluminum/1%Si films is Rho=4.46E-6 ohm-cm, Another data point gave Rho of 6.3E-6 and a third gave Rho of 5.01E-6

t = Rho/Rhos = 5E-6 ohm-cm / 0.0572 ohm = 8741 Å



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Rho for Cu films=3.1E-6

### **4 PT PROBE WAFER THICKNESS MEASUREMENTS**





**EQUATIONS USE BY CDE RESISTIVITY MAPPER** 

Thickness =  $\frac{\text{Known Bulk Resistivity}}{\text{Measured Sheet Resistance}}$ 

Bulk Resistivity is assumed to be known

Measured Sheet Resistance =  $(\pi/\ln 2)(V/I)$ 

The CDE Resistivity Mapper can be programmed to automatically convert measured V/I to thickness

Uniformity = (Max-Min)/(Max+Min)



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### **MODELING OF BULK RESISTIVITY**

Bulk Resistivity is assumed to have a value =  $x Exp^{(y)}$ 

Where the pre exponential value may be different for different film deposition techniques (i.e. evaporation, RF sputtering, DC sputtering, etc.)

	X	У	Rho ohm- Å
CDE Manual	337.17	-0.92401	133.8
PE4400 (300watts)	412	-0.92401	163.5
CVC601	540	-0.92401	214.3
Flash Evaporator	490	-0.92401	194.5

Note: bulk Aluminum Rho = 270 ohm-Å

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### **VERIFICATION USING THE TENCORE P2**











### **Ti DEPOSITION USING CVC EVAPORATOR**



Pressure			are if ellet of if 21 ellets of if			
(Torr)	Mean (Å)	STD	Mean (Å)	STD	]	
3.4 × 10 <sup>-6</sup>	321.67	176.16	469.44	138.48		
1.6 × 10 <sup>-6</sup>	340.00	84.84	1366.67	163.58		
7	© Nove	mber 26, 2013 Dr. Lynn Fi	uller Page 2	26		



### CVC 601 SPUTTER TOOL

CVC 601 Sputter Tool Loading 6 inch wafers

Thickness 10,000Å Dep Rate ~300 A/min Pressure 5 mT Ar Flow 28 sccm Time ~ 33 min



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### **SPUTTERING**

**DC Sputtering** - Sputtering can be achieved by applying large (~2000) DC voltages to the target (cathode). A plasma discharge will be established and the Ar+ ions will be attracted to and impact the target sputtering off target atoms. In DC sputtering the target must be electrically conductive otherwise the target surface will charge up with the collection of Ar+ ions and repel other argon ions, halting the process.

**RF Sputtering** - Radio Frequency (RF) sputtering will allow the sputtering of targets that are electrical insulators (SiO2, etc). The target attracts Argon ions during one half of the cycle and electrons during the other half cycle. The electrons are more mobile and build up a negative charge called self bias that aids in attracting the Argon ions which does the sputtering.

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### **SPUTTERING PLATINUM**

Platinum is easy to sputter in a DC Argon plasma. Platinum is an expensive metal. We purchased a Platinum foil of 50mm by 50mm by 100 um for \$1,100. The foil was mounted in our Denton sputter coater which we normally used to gold coat samples for our Scanning Electron Microscope. Sputtering Pt is straight forward using the normal settings used for Gold. The Denton sputter coater provided a sputter rate of 100 to 200 Å/min. The coating is very non uniform and varies from center to edge of the wafer. The coating at the center of the wafer was twice as thick as at the edge of the wafer. The coating thickness is the main parameter that determines the sheet resistance. This problem can be solved with different equipment including, larger foil target, substrate rotation, etc.



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### **AVAILABLE SPUTTER TARGETS**

### 8" Bonded for CVC-601

Aluminum 100% Aluminum/1% Silicon Chrome Chrome Oxide Copper Molybdenum Tantalum Titanium Titanium10%/Tungsten 90% Silicon Dioxide Silicon Indium Tin Oxide

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**8"Unbonded for CVC-601** Molybdenum/Titanium Titanium/Al 1%/Silicon 2%

### 4" Unbonded for CVC 601

Chrome Indium 90%/Tin 10% Nickel Titanium Tantalum Tin Nickel-Chromium 80%/20% 108E-6 ohm cm, TCR 110 E-6/°C \$450- 4"x1/4" Mel Hollander, Research and PVD Materials Corp. (973) 575-4245 **2" Unbonded for Denton** Gold Palladium

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### **AVAILABLE SPUTTER TARGETS**

### **PE 2400/4400 Targets**

Au	Ta2O5
Zr	Cr
SiO2 Qty2	Ta
Si Qty2	Mg
TiO2	NiFe
Nb2O5	CrSiO
In2O5 Qty2	Nb
Permalloy	SnO2
Fe	A12O3
AlNi	MgF2
NiFeMg	MgO
Ni	Target Insulators 3
Co	Backing Plates6
	C

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### RIT CVC601 SPUTTERING DATA

Material	Head	Power (watts)	Rate
Aluminum	8"	2000	240 Å/min.
Nickel	4"	500	170
Chromium	8"	1350	350
InSn + O2	4"	100	80
Copper	8''	325	110
Gold*	2"	40 mA,50mTorr	250
Tantalum	4"	500	190
Titanium	8''	1350	220
Titanium	4"	500	100
Tungsten	4"	500	100
Tungsten	8"	1000	115
Palladium#	2"	10mA, 90 mTorr	100

This data is for the CVC 601 Sputter System at 5 mTorr Argon Pressure, Base Pressure Prior to Sputter <1E-5 \*Denton Sputter Machine, # Technics Hummer VI

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## **CVC 601 SPUTTERING RECIPES**

CVC601 Alpha Phase Tantalum -Place Kapton Dot on wafer surface for thickness

measurement

Tantalum 4" target, Alpha Phase Tantalum 30 uOhm-cm, Positive TCR ~ 825ppm/°C
Thin layer of reactively sputtered TaN followed by Ta only
Radiant heating, 200 C, entire time starting 5 min prior to pre sputter
Pre Sputter 4" Ta target at 500 watts, 5.5 mTorr, 43.6 sccm Ar, 16.3 sccm N2, 5 min.
Tantalum 4" target, 175 watts, 5.5 mTorr, 43.6 sccm Ar, 16.3 sccm N2, 2 min. ~100Å TaN
Tantalum 4" target, 200 watts, 5.5 mTorr, 43.6 sccm Ar, 30 min. Gives ~2500Å Ta

**CVC 601 Tantalum** 4" target, Beta Phase Tantalum 200 uOhm-cm, Negative TCR ~ -200ppm Tantalum 4" target, 500 watts, 5.5 mTorr, 43.6 sccm Ar, 15 min. Gives ~2800Å Ta

CVC 601 Aluminum at 2000 watts, 5 mTorr, 25 min gives ~7500 Å, 20% nonuniformity

**CVC 601 Copper,** 8" target, 6000 Å 5 min pre sputter, Power = 325 watts, 650 volts, 0.5 A, 5 mTorr Pressure, Time = 20 min. Note: Runs made at 25 and 50 min had problems with copper adhesion and stress in the copper film causing it to roll and peel up. 50 min very bad, recipe needs more work



### **CVC-601 SUBSTRATE ROTATION**

Continuous rotation of the substrate holder in the CVC601 is achieved with the Superior Electric Indexer Unit ON and the left-most knob set to EXT. Then, activating the rotostrate toggle switch results in continuous rotation. If you rotate the knob to - or + for clockwise/counterclockwise rotation, then you disable the rotostrate toggle switch and need to depress the execute Function switch on the superior electric Indexer Unit to achieve rotation. The rotation distance is controlled by the settings of five numbered dials on the Indexer Unit.

00209 setting will give a 90° rotation each time the execute function switch is pressed



### **STRESS IN SPUTTERED FILMS**

Compressively stressed films would like to expand parallel to the substrate surface, and in the extreme, films in compressive stress will buckle up on the substrate. Films in tensile stress, on the other hand, would like to contract parallel to the substrate, and may crack if their elastic limits are exceeded. In general stresses can be negative or positive or near zero depending on many parameters.



### **STRESS IN SPUTTERED TUNGSTEN FILMS**

Tungsten

CVC 601 4" Target 500 Watts 50 minutes 5 mTorr Argon Thickness ~ 0.8 μm

Blisters - Compressive Stress Cracking – Tensile Stress

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Picture from scanner in gowning

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### **REACTIVE SPUTTERING**

**Reactive Sputtering -** introducing gases such as oxygen and nitrogen during sputtering can result in the deposition of films such as indium tin oxide (ITO) or titanium nitride TiN (other examples include AlN,  $Al_2O_3$ , AnO  $Ta_2O_5$ )

**Unwanted Background Gases in Sputtering -** Most Films are very reactive when deposited. Water and oxygen cause rougher films, poorer step coverage, discoloration (brown aluminum), poorer electrical properties, etc.



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### FRIT GLASS SPUTTER TARGETS

Frit Glass Powder is mixed With DI water and coated On wafer then fired at 500C And cooled slowly to form a Sputter target.



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